# San Jacinto River Waste Pits Superfund Site

Comments of International Paper Company and McGinnes Industrial Maintenance Corporation on Environmental Protection Agency Region 6 Proposed Remedial Action Plan

# Appendix H

2016 Data Summary Report

January 12, 2017



## DATA SUMMARY REPORT: 2016 STUDIES SAN JACINTO RIVER WASTE PITS SUPERFUND SITE

**Prepared for** U.S. Environmental Protection Agency, Region 6

**On behalf of** McGinnes Industrial Maintenance Corporation International Paper Company

**Prepared by** Integral Consulting Inc. 719 2nd Avenue, Suite 700 Seattle, Washington 98104

September 2016

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### LIST OF ACRONYMS AND ABBREVIATIONS

Ancho	or QEA	Anchor QEA, LLC		
AOC		Administrative Order on Consent		
DOC		dissolved organic carbon		
EM		end member		
I-10		Interstate Highway 10		
Integr	al	Integral Consulting Inc.		
IPC		International Paper Company		
MIMC		McGinnes Industrial Maintenance Corporation		
ng/kg		nanograms per kilogram		
OCDE	)	octachlorodibenzo-p-dioxin		
OCDF	ŗ	octachlorodibenzofuran		
PDMS	5	polydimethylsiloxane		
PeCDI	D	1,2,3,7,8-pentachlorodibenzo-p-dioxin		
PeCDI	F	2,3,4,7,8-pentachlorodibenzofuran		
pg/L		picograms per liter		
PCL		protective concentration level		
PRC		performance reference compound		
RI/FS		Remedial Investigation/Feasibility Study		
SAP		Sampling and Analysis Plan		
Site		San Jacinto River Waste Pits Superfund Site		
SJRF		San Jacinto River Fleet, L.L.C.		
SJRW	Р	San Jacinto River Waste Pits		
SPME		solid phase microextraction		
TDS		total dissolved solids		
TCDD	)	2,3,7,8-tetrachlorodibenzo- <i>p</i> -dioxin		
TCDF		2,3,7,8-tetrachlorodibenzofuran		
TCEQ	-	Texas Commission on Environmental Quality		
TCRA	-	time-critical removal action		
TEQ		toxicity equivalent		
TEQdef	7	TEQ concentration calculated using only dioxin and furan congeners		
TMDL	_	total maximum daily load		

TOC	total organic carbon
TSS	total suspended solids
TSWQS	Texas surface water quality standards
UAO	Unilateral Administrative Order
USEPA	U.S. Environmental Protection Agency

### **1** INTRODUCTION

This Data Summary Report (Report) presents a summary of the results of five studies conducted in 2016 as part of the Remedial Investigation/Feasibility Study (RI/FS) for the San Jacinto River Waste Pits (SJRWP) Superfund Site (Site) in Harris County, Texas (Figure 1-1; CERCLA Docket No. 06-10-03). It was prepared on behalf of International Paper Company (IPC) and McGinnes Industrial Maintenance Corporation (MIMC) (collectively referred to as the Respondents) in response to direction from the U.S. Environmental Protection Agency (USEPA) received on August 6, 2015, and in subsequent meetings (Appendix A).

#### 1.1 Purpose

This Report was prepared by Respondents to provide USEPA with additional current Site data from the 2016 studies for USEPA's use in remedial decision making. USEPA requested that Respondents perform the 2016 studies to "[c]onfirm that the [time-critical removal action (TCRA)] cap continues to prevent dioxin/furan migration from the waste pits to the San Jacinto River..." (Appendix A). USEPA required Respondents to collect new data for the following environmental media:

- Surface sediments surrounding the impoundments north of Interstate Highway 10 (I-10)
- Porewater of the TCRA armored cap
- Groundwater beneath the impoundments north of I-10 and south of I-10
- Surface water
- Tissue of Gulf killifish (*Fundulus grandis*)

These 2016 studies were undertaken to provide multiple lines of evidence to address USEPA's request for additional information regarding the effectiveness of the TCRA cap and conditions south of I-10 in containing dioxins and furans (Appendix A).

A summary of the results of these studies is as follows:

• Sediments. 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) and 2,3,7,8tetrachlorodibenzofuran (TCDF) concentrations in surface sediments surrounding the impoundments north of I-10 are statistically significantly lower in 2016 than in 2010. The 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) toxicity equivalent concentrations calculated for dioxins and furans using toxicity equivalent factors for mammals (TEQ<sub>DF,M</sub>) concentrations in 2016 sediments are also lower than in 2010 sediments, and are well below the sediment protective concentration level (PCL) of 220 ng/kg.

- **TCRA Armored Cap Porewater.** TCDD, TCDF, and 2,3,4,7,8pentachlorodibenzofuran (2,3,4,7,8-PeCDF), the target compounds for the TCRA armored cap porewater study, were not detected in any of the samples of porewater of the TCRA armored cap.
- **Groundwater north of I-10.** In groundwater beneath the impoundments north of I-10, TCDD, TCDF, and 2,3,4,7,8-PeCDF, the target dioxin and furan compounds for the groundwater study, were not detected.
  - Groundwater beneath the impoundments north of I-10 meets the Texas surface water quality standards (TSWQS) for the TCDD toxicity equivalent (TEQ) of 0.0797 pg/L.
  - Groundwater results indicate that there is no evidence of subsurface transport of dioxins and furans from the northern impoundments into groundwater or into surface water.
  - The groundwater results for the northern impoundments are consistent with the results of the previous groundwater study north of I-10 (Integral and Anchor QEA 2013).
- **Groundwater south of I-10.** In groundwater beneath the impoundment south of I-10, TCDD, TCDF, and 2,3,4,7,8-PeCDF were not detected in 8 out of 10 wells. The only wells where these compounds were detected were those drilled directly into the waste.
  - None of the target compounds were detected in the deep groundwater well south of I-10.
  - None of the target compounds were detected in wells along the shoreline of the peninsula south of I-10.
  - There is no evidence of subsurface transport of dioxins and furans from the impoundment to surface water.
  - Groundwater beneath the impoundment south of I-10 meets the TSWQS for TEQ except where detected, in the two shallow wells drilled directly into the wastes.

- Results of 2016 groundwater sampling south of I-10 are consistent with results of prior sampling in 2012 and 2013 (Anchor QEA and Integral 2013).
- Surface Water. The average TEQ concentrations in surface water above the submerged portion of the impoundments north of I-10 in 2016 were 92 percent lower than their 2009 pre-TCRA average TEQ<sub>DF,M</sub> values.
- **Gulf killifish tissue**. Concentrations of TEQ<sub>DF,M</sub> in Gulf killifish composite samples collected adjacent to the TCRA armored cap in 2016 are comparable to most fish samples from other stations sampled in 2016.
  - Concentrations of TEQ<sub>DF,M</sub> in Gulf killifish adjacent to the TCRA armored cap have decreased relative to concentrations in 2010, and the decrease is driven by a corresponding decrease in TCDD at that location.
- Source evaluation. Noticeable changes in dioxin and furan congener concentrations in surface water, sediment, and fish tissue, particularly in a tissue sample collected adjacent to the upland sand separation area, suggest that dioxin and furan concentrations in these media within USEPA's preliminary Site perimeter in 2016 are influenced by a new source or sources of dioxins and furans. The spatial and temporal patterns in congeners in tissue, sediment, and water indicate that the source of dioxins and furans in tissue collected in 2016 is likely not the wastes from the impoundments.

Because the armored cap constructed for the TCRA is the primary subject of the studies reported in this document, it is described briefly below.

#### 1.2 Time-critical Removal Action

Concurrent with the RI/FS, a TCRA was implemented by IPC and MIMC pursuant to an Administrative Order on Consent (AOC) with USEPA (Docket No. 06-12-10, April 2010; USEPA 2010). The TCRA involved capping and isolation of the wastes in the impoundments north of I-10, with related construction completed in July 2011. The purpose of the TCRA was to stabilize the entire area within the original 1966 perimeter of the impoundments north of I-10 (the TCRA Site) (Figure 1-1), until a final remedy could be selected and implemented (USEPA 2010).

As required by the AOC, the Respondents prepared a TCRA alternatives analysis of potential design options for the TCRA. Upon review of the TCRA alternatives analysis, USEPA selected a granular cover designed to withstand a storm event with a return period of 100 years. The selected design included the following construction elements:

- A security fence on the uplands to prevent unauthorized access to the TCRA Site
- Placement of "Danger" signs indicating that this location is a Superfund site, and providing a phone number for more information
- Installation of a stabilizing geotextile barrier over the eastern cell
- Installation of a low-permeability geomembrane and geotextile barrier in the western cell
- Installation of granular (e.g., rock) cover
- Design and implementation of an operations, monitoring, and maintenance plan for the TCRA.

TCRA operations, monitoring, and maintenance are ongoing.

Implementation of the TCRA, including construction of the armored cap, was undertaken as an interim remedial measure to contain and isolate the wastes during the RI/FS. The success of the armored cap in achieving this objective was the focus of the studies conducted in 2016 (Appendix A).

### 1.3 Report Organization

This Report consists of brief descriptions of each study and summary tables and figures to describe results and how results address the sampling objectives. This Report includes the following appendices:

- Appendix A. USEPA Communications Directing Respondents to Conduct Studies of the TCRA Armored Cap and Southern Impoundment Area
- Appendix B. Validation Reports for 2016 Studies
- Appendix C. PRC Fibers for the 2016 Assessment of Porewater within the Armored Cap.

Respondents may prepare an addendum to the RI (Integral and Anchor QEA 2013) to provide a more extensive and in-depth analysis of data collected in 2016, and an update to the conceptual site model. This Report is not intended to be a substitute for that addendum.

### 2 SURFACE SEDIMENT DATA COLLECTED IN 2016

In 2016, surface sediments (0 to 6 inches) were collected from 17 locations that were also sampled in 2010. Surface sediments were also collected in 2016 at 11 stations not previously sampled (Figure 2-1).

At several stations sampled in both 2010 and 2016, TCDD TEQ<sub>DF,M</sub> concentrations are lower in 2016 than in 2010. In 2016, concentrations of both TCDD and TCDF, which are indicators of the waste within the impoundments, are statistically significantly lower than in 2010. Many of the most marked changes in sediment quality have taken place along the west and northwest perimeter of the impoundments north of I-10. Results of unmixing analysis indicate that several 2016 samples do not contain any dioxins and furans from the waste in the impoundments. A majority of 2016 sediments from locations at which dioxins and furans were associated with waste from the impoundments that were detected in 2010 are currently less affected by wastes, or are not affected by the wastes at all.

## 2.1 Summary of Sampling Activities and Sampling Schedule

Sediment sampling was conducted from May 6 through 10, 2016, and on July 17, 2016 (Table 2-1); surface sediment samples were collected using a stainless-steel grab sampler. Samples were analyzed for dioxins and furans, total organic carbon (TOC), and grain size distribution. Detailed information on the sediment sampling program is presented in Integral (2016a).

## 2.2 Results

Results of 2016 surface sediment sampling are summarized in Table 2-2. The general spatial distribution of TEQ<sub>DF,M</sub> concentrations in all sediments collected in 2016 is shown in Figure 2-2. Concentrations of TEQ<sub>DF,M</sub> in all sediments collected in 2016 are below the PCL for sediments of 220 ng/kg dw.

## 2.2.1 Concentrations of Dioxins and Furans in Surface Sediments in 2016 vs. 2010

Results of 2016 surface sediment sampling demonstrate improvement to sediment quality, and an overall reduction of the influence of the wastes from the northern impoundments in surrounding sediments (Figure 2-3). In statistical comparisons of the populations of samples

for stations sampled in both 2010 and 2016, concentrations of TCDD and TCDF in sediments are significantly lower in 2016 than in 2010 (Wilcoxon Rank Sum, p <0.10). As a result,  $TEQ_{DF,M}$  is lower, though not significantly.

Some congeners are higher in 2016 sediments than in 2010 sediments (Table 2-2). The minimum, maximum, and average octachlorodibenzo-p-dioxin (OCDD) concentrations are higher in 2016. This is also true for 1,2,3,7,8-pentachlorodibenzo-p-dioxin (PeCDD) and octachlorodibenzofuran (OCDF). As discussed later in this Report, these results may suggest the presence of a new source or sources of dioxins and furans to these sediments (e.g., OCDD and OCDF are typically associated with diesel exhaust; USEPA 2006). These congeners are not indicators of wastes from within the northern impoundments (see RI Report, Section 5.4; Integral and Anchor QEA 2013).

To evaluate the extent to which wastes from the impoundments could be contributing to the dioxins and furans in sediments collected in 2016, Integral ran the unmixing model that was developed in 2012 and described in detail in the RI Report (Section 5.4) with the full RI sediment data set, including data collected in 2016 (Table 2-3; Figure 2-2). Results of this model are expressed as the proportion of each sample attributable to each of two source types or "end members" (EMs). In this model, EM1 reflects the characteristics of dioxin and furan mixtures originating from a broad range of sources in urban areas, or "urban background," and EM2 reflects the characteristics of the dioxin and furan mixture in wastes within the impoundments. The term "residuals" refers to a measure of uncertainty in model results—in this case, the proportion of the sample dioxin and furan mixture that is not explained by the model.

Table 2-3 reports the proportion of each sample collected in 2016 that conforms to each of the two EMs, and the unmixing results for these samples in 2010. In most samples collected in 2016, there is no discernable contribution to the mixture from wastes originating within the impoundments. In those locations where samples were collected in both 2010 and 2016, a reduction in the proportion of EM2 is evident.

#### 2.3 Conclusions from 2016 Surface Sediment Sampling

Sediment data collected in 2016 demonstrate an improvement in the quality of sediments surrounding the impoundments north of I-10, with lower TEQ<sub>DF,M</sub>, TCDD, and TCDF concentrations than in 2010. All sediments sampled in 2016 were below the PCL for sediments at this site (220 ng/kg dw). Reductions in TCDD and TCDF, as well as the results of the unmixing model, clearly demonstrate that the influence of the wastes from the impoundments on the quality of sediments surrounding the impoundments is less than in 2010, before construction of the TCRA armored cap. These lines of evidence show that the TCRA armored cap is effective in containing the dioxins and furans associated with wastes in the northern impoundments.

A distinct increase in several congeners, including PeCDD, OCDD, and OCDF, in sediments from locations sampled in both 2010 and 2016 is evidence that there may be one or more new and distinctive sources of dioxins and furans affecting sediment quality in the 2016 samples. These congeners are not associated with paper mill wastes, and therefore suggest the presence of another source or sources. Additional discussion of this topic is presented in Section 7.

### **3** TCRA CAP POREWATER DATA COLLECTED IN 2016

Porewater sampling within the TCRA armored cap was conducted using solid phase microextraction (SPME) fibers, and following a study design very similar to the porewater sampling performed in 2012 (Integral and Anchor QEA 2012). In 2016, target dioxin and furan congeners were identified as described in the TCRA Cap Porewater Assessment Sampling and Analysis Plan (SAP) Addendum 1 (Integral and Anchor QEA 2016a, pp. 5-6): TCDD, TCDF, and 2,3,4,7,8-PeCDF. The additional congener (2,3,4,7,8-PeCDF) was included in the 2016 study upon USEPA request. Together, these three congeners account for more than 90 percent of risk due to exposure to TEQ<sub>DF,M</sub> in wastes from within the original impoundment perimeter.

Target dioxin and furan congeners were not detected in any of the SPME porewater samples collected in 2016 (Table 3-1). There is no evidence of vertical gradients in concentrations of any of the target dioxin and furan congeners in the porewater of the armored cap. SPME fibers that extended into the surface water were deployed in two locations. Target dioxin and furan congeners were not detected in one of the surface water SPME samples; the other surface water SPME sample was lost during the deployment period, likely due to disturbance due to storms that occurred in May. Results of the 2016 TCRA cap porewater assessment study confirm that the armored cap continues to effectively contain dioxins and furans from the waste impoundments, consistent with the results of the TCRA cap porewater assessment conducted in 2012 (Integral and Anchor QEA 2013).

## 3.1 Summary of Sampling Activities and Sampling Schedule

A summary of field activities undertaken in support of the TCRA cap porewater assessment in 2016 is provided in Anchor QEA (2016a). Dates of sampler deployment and retrieval are presented in Table 2-1. Porewater sampling locations are shown in Figure 3-1.

As in 2012, selected fibers were impregnated with performance reference compounds (PRCs) (Figure 3-1) to provide an indication of the degree of equilibrium that the fibers had achieved after deployment. Pre-deployment and post-deployment PRC concentrations are presented in Table 3-2. Some of the samplers and PRC fibers were displaced during deployment due to storm events and could not be retrieved, as described in Anchor QEA

(2016a), and some of the fibers containing PRCs could not be used (Appendix C). However, a sufficient number of both samples and PRC-impregnated fibers were successfully deployed, retrieved, and analyzed for this study.

#### 3.2 Results

Target analytes in all retrieved samplers were below detection limits (Table 3-1).

### 3.3 Conclusions from 2016 TCRA Cap Porewater Sampling

None of the target analytes were detected in any porewater or surface water SPME sample. According to USEPA and Texas Commission on Environmental Quality (TCEQ) requirements, the study was designed to be highly sensitive and employed very low detection limits for all target compounds. No target congeners were detected, which indicates the absence of a measureable vertical concentration gradient within the armored rock of the TCRA armored cap. These results coupled with the results observed in 2012 confirm the TCRA armored cap continues to effectively contain dioxins and furans from the impoundments north of I-10.

#### 4 GROUNDWATER DATA COLLECTED NORTH OF I-10 IN 2016

Sampling of groundwater using SPME fibers was conducted in 2016 at four wells beneath the impoundments north of I-10. Target congeners were the same as those in the 2016 porewater study: TCDD, TCDF, and 2,3,4,7,8-PeCDF. Target compounds were not detected in any of the wells beneath the impoundments north of I-10, and all of these wells were in compliance with TCEQ's TSWQS of 0.0797 pg/L TEQ<sub>DF,M</sub> (Table 4-1). These results verify that dioxin and furan congeners in the wastes beneath the TCRA armored cap on the northern impoundments are not transported to groundwater, and do not contribute to degradation of surface water quality.

#### 4.1 Summary of Sampling Activities and Sampling Schedule North of I-10

Four new monitoring wells were installed directly beneath the impoundments north of I-10 (Figure 4-1) according to the schedule shown in Table 2-1, and described by Anchor QEA (2016b). Two of these wells were angled from outside the cap to monitor the shallowest permeable zone beneath the waste (Station IDs: SJMW011 and SJMW013). The two other wells were installed vertically (one west of the impoundments and one at the north end of the central berm) to monitor the shallowest permeable zone (Station IDs: SJMW010 and SJMW012; Figure 4-1). These four new wells were sampled for TCDD, TCDF, and 2,3,4,7,8-PeCDF using two 2.5-foot-long SPME fibers combined in series to result in a 5-foot-long sample fiber (Anchor QEA and Integral 2016). A detailed summary of field activities undertaken in 2016 in support of SPME sampling of the groundwater beneath the TCRA armored cap is provided in Anchor QEA (2016b).

Dates of SPME fiber deployment and retrieval times for the 2016 groundwater study are presented in Table 2-1; fiber length and PRC concentrations present in the groundwater SPME fibers before and after deployment are presented in Table 4-2. These PRC concentrations were used to measure the fraction of equilibrium achieved in each groundwater SPME fiber for each target analyte.

#### 4.2 Results

All target analytes were below detection limits in groundwater SPME samples north of I-10 (Table 4-3). In addition, the equilibrium-corrected groundwater concentrations of all of these samples were below the state water quality criteria of 0.0797 pg/L TEQ<sub>DF,M</sub> (Table 4-1).

As described in the Groundwater SAP Addendum 3 (Anchor QEA and Integral 2016) and at the request of TCEQ, results of the groundwater SPME samples were analyzed to determine an estimated TEQ<sub>DF,M</sub> concentration in groundwater for comparison to the TSWQS of 0.0797 pg TEQ/L. To do this requires three steps:

- 1. The degree of equilibrium (f<sub>e</sub>) achieved by each SPME fiber is calculated using the results from PRC fibers (Table 4-2).
- 2. The mass of each congener in each fiber sample is divided by the volume of the polydimethylsiloxane (PDMS) coating on the fiber (as a function of the length of the fiber and the thickness of the PDMS coating) to determine the concentration of the target congener in the PDMS (C<sub>f</sub>).
- 3. Information on the fraction of equilibrium achieved in each groundwater well is then combined with the fiber-water partition coefficient (K<sub>fw</sub>) to estimate equilibrium-corrected groundwater concentrations for each target analyte using the equation below:

$$C_w = \frac{C_f}{K_{fw} \times f_e}$$

Where:

- C<sub>w</sub> = estimated concentration of target compound in groundwater (pg/L)
- $C_{\rm f}$  = concentration of target compound in fiber in the PDMS (pg/L)
- $K_{fw}$  = fiber-water partition coefficient
- $f_e = fraction of equilibrium (unitless)$

C<sub>w</sub> was calculated for each of the target congeners, and summed for each fiber to determine the TEQ<sub>DF,M</sub> concentration for groundwater in the well. This analysis method was described in the Groundwater SAP Addendum 3 (Anchor QEA and Integral 2016).

In groundwater well location SJMW013, deployment of the PRC-impregnated fiber was delayed because that well location required unanticipated access improvements prior to well installation (Anchor QEA 2016b). Because of the delay, PRC data from this sampler could not be used to reliably address equilibrium status for the sampler in well location SJMW013. To estimate the value of fe for this sampler in performing the above calculation, fe was approximated as the average fraction of equilibrium achieved by the remaining fibers deployed in groundwater wells.

The analysis of the groundwater sample results demonstrates that the samples were below the TSWQS of 0.0797 pg TEQ/L.

### 4.2.1 Conclusions from 2016 Groundwater Sampling North of I-10

The absence of detectable target analytes in groundwater samples north of I-10 confirms that dioxin and furan congeners in the wastes beneath the TCRA armored cap on the northern impoundments are not transported to groundwater. Concentrations of target analytes in groundwater estimated from SPME results confirm that groundwater beneath the impoundments north of I-10 does not contribute to degradation of surface water quality.

### 5 GROUNDWATER DATA COLLECTED SOUTH OF I-10 IN 2016

Sampling of groundwater using SPME fibers was conducted in 10 wells in and adjacent to the impoundments south of I-10 (Figure 5-1). Target congeners were the same as for the porewater study: TCDD, TCDF, and 2,3,4,7,8-PeCDF. Results of groundwater sampling south of I-10 confirm that conditions there are effective in containing dioxins and furans, and specifically, that there is no potential for transport of dioxins and furans via shallow groundwater to surface water and to deep groundwater.

In eight of the ten wells, none of the target compounds were detected. These include the four wells installed along the western shoreline of the peninsula south of I-10. In the eight groundwater wells in which target compounds were not detected, estimated concentrations of TEQ<sub>DF,M</sub> in groundwater were all below the TSWQS of 0.0797 pg/L (Table 5-2).

Two of the wells were drilled into the waste material (SJMW003 and SJMW004S). In these wells, TCDF and TCDD were detected in SPME samples. Estimated  $TEQ_{DF,M}$  concentrations in these two are greater than the TSWQS.

## 5.1 Summary of Sampling Activities and Sampling Schedule South of I-10

Four vertical monitoring wells were installed along the western edge of the peninsula south of I-10 to monitor the shallowest permeable zone (Figure 5-1). In addition, six wells previously sampled in 2012 and 2013 (five shallow wells and one deep well) were also sampled, for a total of 10 groundwater samples in 2016. Dates of deployment and retrieval are presented in Table 2-1. As for the groundwater sampling north of I-10, SPME passive samplers were deployed in each of these wells, and analyzed for target compounds TCDD, TCDF, and 2,3,4,7,8-PeCDF. A detailed summary of field activities undertaken in support of SPME sampling of the groundwater south of I-10 in 2016 is provided by Anchor QEA (2016b).

## 5.2 Results

Groundwater concentrations of each target congener and of TEQ<sub>DF,M</sub> were estimated from SPME concentrations using fiber-water partition coefficients and corrected for equilibrium as described in Section 4.1.2, and using information on the PRC fibers for these samples

(Table 5-1) and on the reported mass of each target compound in each SPME sample (Table 5-3).

In eight of ten groundwater wells south of I-10, target analytes were not detected (Table 5-3). The equilibrium-corrected groundwater concentrations of these samples were below the state water quality criteria of 0.0797 pg/L TEQ (Table 5-2).

In two wells drilled directly into the wastes (SJMW003 and SJMW004), the estimated  $TEQ_{DF,M}$  concentrations in groundwater were greater than the TSWQS of 0.0797 pg/L.

#### 5.3 Conclusions from 2016 Groundwater Sampling South of I-10

The absence of target analytes in groundwater samples in the four wells along the western shoreline of the peninsula south of I-10, and the resulting estimated groundwater concentrations, confirm that these wells are in compliance with state surface water quality standards. The absence of dioxins and furans in the samples from these wells also confirms that there is no subsurface transport of dissolved dioxins and furans by groundwater to the surface water. Therefore, current conditions effectively contain dioxins and furans within the waste impoundment south of I-10, and are not contributing to degradation of surface water quality.

#### 6 SURFACE WATER DATA COLLECTED IN 2016

Surface water samples were collected at seven locations (Figure 6-1) once per week during each of three consecutive weeks in July 2016. Sampling stations were at five locations previously sampled by the TCEQ's dioxin total maximum daily load (TMDL) program from 2002 to 2004, and two new stations. The same methods used by the TMDL program were used in 2016 to enable direct comparisons of current and past conditions. The study was designed to allow this comparison, and to provide information on trends across a large area, including the presence of dioxins and furans in surface waters upstream and downstream of USEPA's preliminary Site perimeter.

Results of the surface water quality study show that the greatest change in TEQDF,M between past and current conditions occurred at the station located directly above the waste impoundments north of I-10, and that the second greatest change was at the station just downstream of the impoundments north of I-10, under the I-10 bridge. These results show that the TCRA armored cap is effective in containing dioxins and furans in the wastes of the impoundments. Results also show increases in TEQDF,M and TCDD in surface water upstream of USEPA's preliminary Site perimeter.

### 6.1 Summary of Surface Water Sampling Activities and Sampling Schedule

Surface water samples were collected from July 5 to 22, 2016 (Table 2-1). Surface water sampling was conducted using high volume pump systems designed to collect information on hundreds of liters of water per sample, and to provide data for each dioxin and furan congener concentration in both the dissolved phase and in suspended solids. Water quality parameters measured at each station included TOC, dissolved organic carbon (DOC), total suspended solids (TSS), and total dissolved solids (TDS). A detailed account of the field sampling program is provided by Integral (2016b).

### 6.2 Results

Table 6-1 lists the results for each dioxin and furan congener and  $TEQ_{DF,M}$  in each surface water sample collected in 2016.

#### 6.3 Concentrations of Dioxins and Furans in Surface Water in 2016 vs. 2010

Tables 6-2 and 6-3 list average TEQ<sub>DF,M</sub> and TCDD concentrations, respectively, by year at each re-sampled location. The percent difference between the highest of past concentrations and the average of 2016 concentrations is also shown for each station. Concentrations of both TEQ<sub>DF,M</sub> and TCDD in surface water above the impoundments north of I-10 are lower in 2016 than in 2009 by more than 90 percent.

Changes in TEQ<sub>DF,M</sub> concentrations over time are also illustrated by Figure 6-2 which shows results for all stations over time. The tables and figure demonstrate that the largest improvement in water quality at all stations sampled was at the station directly above the impoundments north of I-10, and the second largest improvement was immediately downstream at the location under the I-10 bridge.

These tables and figures also show increasing TEQ<sub>DF,M</sub> and TCDD concentrations at the station upstream of the Site, station SJMW001 (TMDL station 11197).

#### 6.4 Conclusions from 2016 Surface Water Sampling

Surface water data collected in 2016 demonstrate that, following construction of the TCRA armored cap, there has been a substantial improvement in dioxin and furan concentrations in surface water in the vicinity of the impoundments north of I-10. Improvements are attributable to some extent to reductions in the concentrations of TCDD. However, as described in the next section, the presence of PeCDD in surface water at concentrations higher than in the past both upstream and downstream of USEPA's preliminary Site perimeter is not attributable to the wastes in the impoundments, but could impact the overall rate of reductions in TEQ<sub>DF,M</sub> in surface water over time.

### 7 FISH TISSUE DATA COLLECTED IN 2016

Composite samples of Gulf killifish were collected in 2016 from locations within USEPA's preliminary Site perimeter that were also sampled in 2010 (Figure 7-1). Results include the following:

- Concentrations of TEQ<sub>DF,M</sub> in Gulf killifish collected from SJTTR3 (adjacent to the TCRA armored cap) in 2016 were much lower than TEQ<sub>DF,M</sub> concentrations in 2010. This reduction was driven by a marked decrease in the concentration of TCDD in killifish at this location.
- Concentrations of TEQ<sub>DF,M</sub> in Gulf killifish from the other transect locations sampled in 2016, those not located directly adjacent to the TCRA cap, were higher than in 2010.
- The highest TEQ<sub>DF,M</sub> concentration occurred in one of two Gulf killifish composite samples collected from SJTTR5, which is adjacent to the upland sand separation area and operations of the San Jacinto River Fleet (SJRF). The TEQ<sub>DF,M</sub> concentration was largely (67 percent) the result of an elevated concentration of PeCDD in that sample. The proportion of PeCDD in this fish sample is very high relative to the proportions of TCDD and TCDF. In contrast, in the wastes within the northern impoundments, PeCDD contributes only a very small fraction of the total mass of dioxins and furans. This congener was often not detected in samples from within the perimeter of the northern impoundments during the RI (Appendix F of the RI Report).
- Observed congener patterns in the 2016 Gulf killifish samples indicate that they are likely affected by dioxin sources other than the wastes within the impoundments.

## 7.1 Summary of Fish Tissue Sampling Activities and Sampling Schedule

Tissue samples were collected using minnow traps and following the specifications of Addendum 2 to the Tissue SAP (Integral and Anchor QEA 2016b), and methods described by the 2010 Tissue Field Sampling Plan (Integral 2010). A detailed summary of field activities undertaken in support of fish tissue sampling in 2016 is provided by Integral (2016a).

Fish were collected along four nearshore transects within USEPA's preliminary Site perimeter (Figure 7-1) from May 8 to 10 and from July 16 to 22, 2016 (Table 2-1). Gulf

killifish were collected as composite samples of fish of specified lengths (Integral 2016a). Two composite samples of Gulf killifish were collected from each transect, except SJTTR3, where only a single composite sample of this species could be obtained, likely because the armored cap does not provide attractive habitat for this species. To achieve the requirements of the SAP for this study and with USEPA's approval, one composite of five inland silversides (*Menidia beryllina*) captured at SJTTR3 was prepared and analyzed (Integral 2016a). The five individuals ranged in length from 58 to 80 mm and were collected on July 18, 2016.

All 17 2,3,7,8-substituted dioxin and furan congeners and percent lipids were analyzed in the seven Gulf killifish and the inland silverside composites.

### 7.2 Results

Concentrations of each congener and of TEQ<sub>DF,M</sub> in each Gulf killifish sample are presented in Table 7-1; results for the composite of inland silversides are presented in Table 7-2. For the purposes of evaluating spatial and temporal patterns in fish tissue concentrations, TEQ<sub>DF,M</sub> concentrations were calculated assuming that concentrations of congeners not detected were zero.

### 7.3 Concentrations of Dioxins and Furans in Fish Tissue in 2016 vs. 2010

The Gulf killifish composite collected from SJTTR3 (the transect adjacent to the TCRA armored cap) in 2016 showed a large decrease in TEQ<sub>DF,M</sub> concentration relative to 2010 (Figure 7-2). This reduction was driven by a marked decrease in the concentration of TCDD (Figure 7-2).

At other transects, TEQ<sub>DF,M</sub> concentrations in 2016 Gulf killifish were higher than those in killifish sampled in 2010 (Figure 7-2). TEQ<sub>DF,M</sub> concentrations in one of the two 2016 Gulf killifish composites from SJTTR5 (adjacent to the upland sand separation area; Figure 7-1), were much greater than the others in either 2010 or 2016, at 14.5 ng/kg ww (SJTTR5-F4). In this composite sample, PeCDD accounted for the largest fraction of the TEQ<sub>DF,M</sub>, and the overall mixture of congeners was unusual among the other Gulf killifish captured this year (Figure 7-3a). Changes in both the TEQ<sub>DF,M</sub> concentration and in the congener pattern from those of killifish in 2010 were at least partly the result of higher concentrations of PeCDD

and OCDD in sample SJTTR5-F4 than at other stations (Figure 7-2). The concentration of OCDD was also much higher in this fish composite in 2016 than in other fish collected this year, and higher than in any fish collected in 2010 (Figure 7-2). Neither PeCDD nor OCDD are typically found in large proportions by mass in waste materials from within the impoundments (see Appendix F of the RI Report).

The unusual congener patterns in fish tissue and the spatial patterns of key congeners, TCDD, PeCDD, and OCDD in tissue and sediment, strongly suggest that concentrations of dioxins and furans in 2016 Gulf killifish tissue are due to a source or sources of dioxins and furans other than the wastes within the impoundments. Conditions within USEPA's preliminary Site perimeter have changed substantially since fish were sampled in 2010, and now include the operations of the SJRF. A brief data analysis to address the potential reasons for the unexpected patterns of dioxins and furans in fish tissue is presented below.

#### 7.4 Evaluation of Potential Sources of Dioxins and Furans in 2016 Gulf killifish

The observed congener patterns in Gulf killifish tissue show an important role for at least two dioxin congeners not associated with wastes from the impoundments: PeCDD and OCDD. The data generated for this RI in 2016 strongly support a conclusion that another source or sources of dioxins are affecting fish tissue chemistry. Some key observations to support this conclusion are:

- The congener accounting for the largest fraction of total dioxin and furan mass in 2016 fish tissue is OCDD (Figure 7-3a; Table 7-1), a congener associated with dioxins and furans in background sources such as diesel exhaust (see RI Report, Section 5.4, Figure 5-25). This was not true in 2010 samples collected adjacent to the northern impoundments (see RI Report, Table 5-18), when the concentration and the proportion of OCDD were less than those of TCDD and TCDF in Gulf killifish (Figure 7-3b). In 2010 Gulf killifish from SJTTR3, collected adjacent to the pre-TCRA waste impoundments, the dominant congeners were TCDD and TCDF (Figure 7-3b).
- 2016 Gulf killifish samples collected proximal to the cap showed a large decrease in concentrations of TCDD and TEQ<sub>DF,M</sub> relative to 2010. These fish also showed an increase in OCDD concentrations, as did tissue from the other transects sampled (Figure 7-2). The divergence between decreasing TCDD and increasing OCDD in fish

from SJTTR3 is a strong indication that the source of dioxin in the fish collected in 2016 is not the wastes in the impoundments.

- Surface sediments show a significant reduction in TCDD (Figure 7-4) and TCDF concentrations (Section 2) and an increase in OCDD and to a lesser extent PeCDD concentrations over the same time period (Figures 7-5 and 7-6). In 2016 samples, the increasing concentrations of these two congeners in sediments were observed on the western side of the northern impoundments, near the upland sand separation area and SJRF operations, particularly for OCDD.
- The dissolved fraction of surface water samples collected in 2016 show an overall decrease in TCDD and TEQ<sub>DF.M</sub> concentrations relative to historical samples within and downstream of USEPA's preliminary Site perimeter, but an increase upstream of the preliminary Site perimeter (Section 6, above; Figure 7-7). This might explain the consistent TCDD signal in Gulf killifish collected across the area sampled in 2016. If the waste from the impoundments was the source of the TCDD observed in 2016 fish, it would not be expected to be uniform across all transects, but would show a more marked spatial pattern, as in 2010 Gulf killifish. Thus, the spatial pattern of TCDD in Gulf killifish in 2016 and the presence of higher concentrations of TCDD in water from upstream suggests a more diffuse source or sources for this congener, potentially upstream of USEPA's preliminary Site perimeter.
- Consistent with the pattern observed in tissue and sediment, the concentration of dissolved PeCDD in surface water has increased over time (Figure 7-7), both upstream and downstream of USEPA's preliminary Site perimeter, and under the I-10 bridge.

### 7.5 Conclusions from 2016 Fish Tissue Sampling

Concentrations of TEQ<sub>DF,M</sub> in Gulf killifish adjacent to the TCRA armored cap have decreased relative to concentrations in 2010, and the decrease is driven by a corresponding decrease in TCDD at that location. While an increase in TEQ<sub>DF,M</sub> relative to 2010 was observed at other transects within USEPA's preliminary Site perimeter, the spatial and temporal patterns in congeners in tissue, sediment, and water indicate that the source of dioxins and furans to tissue collected in 2016 is likely not the wastes from the impoundments. The changes in concentrations of TCDD, PeCDD, and OCDD in fish, sediment, and water over time seem to indicate a new source or sources of these dioxins to fish.

Because the TCDD and overall TEQ<sub>DF,M</sub> concentrations in Gulf killifish collected directly adjacent to the TCRA armored cap have dropped substantially since 2010, and because of the results described above for the other lines of evidence in this study, results of tissue sampling indicate that the TCRA armored cap is effective in preventing exposure of fish to dioxins and furans in wastes in the impoundments.

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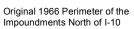
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# FIGURES



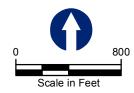


USEPA's Preliminary Site Perimeter



Approximate TCRA Footprint

Soil Investigation Area 4

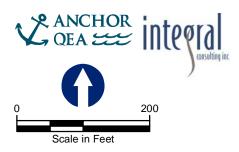


<sup>a</sup> Designation of the sand separation area is intended to be a general reference to areas in which such activities are believed to have taken place based on visual observations of aerial photography from 1998 through 2002.

FEATURE SOURCES: Aerial Imagery: 0.5-meter. Photo Date: 01/14/2009 Texas Strategic Mapping Program (StratMap), TNRIS

Figure 1-1 Overview of Area within USEPA's Preliminary Site Perimeter Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC





- Samples Collected to Address Study Element 1. Nature and Extent Evaluation
- TCRA Sediment Stations Reoccupied to Address Study Element 3. Physical CSM and Fate and Transport Evaluation
- Nature and Extent Stations Reoccupied to Address Study Element 3. Physical CSM and Fate and Transport Evaluation
- Original 1966 Perimeter of the Impoundments North of I-10



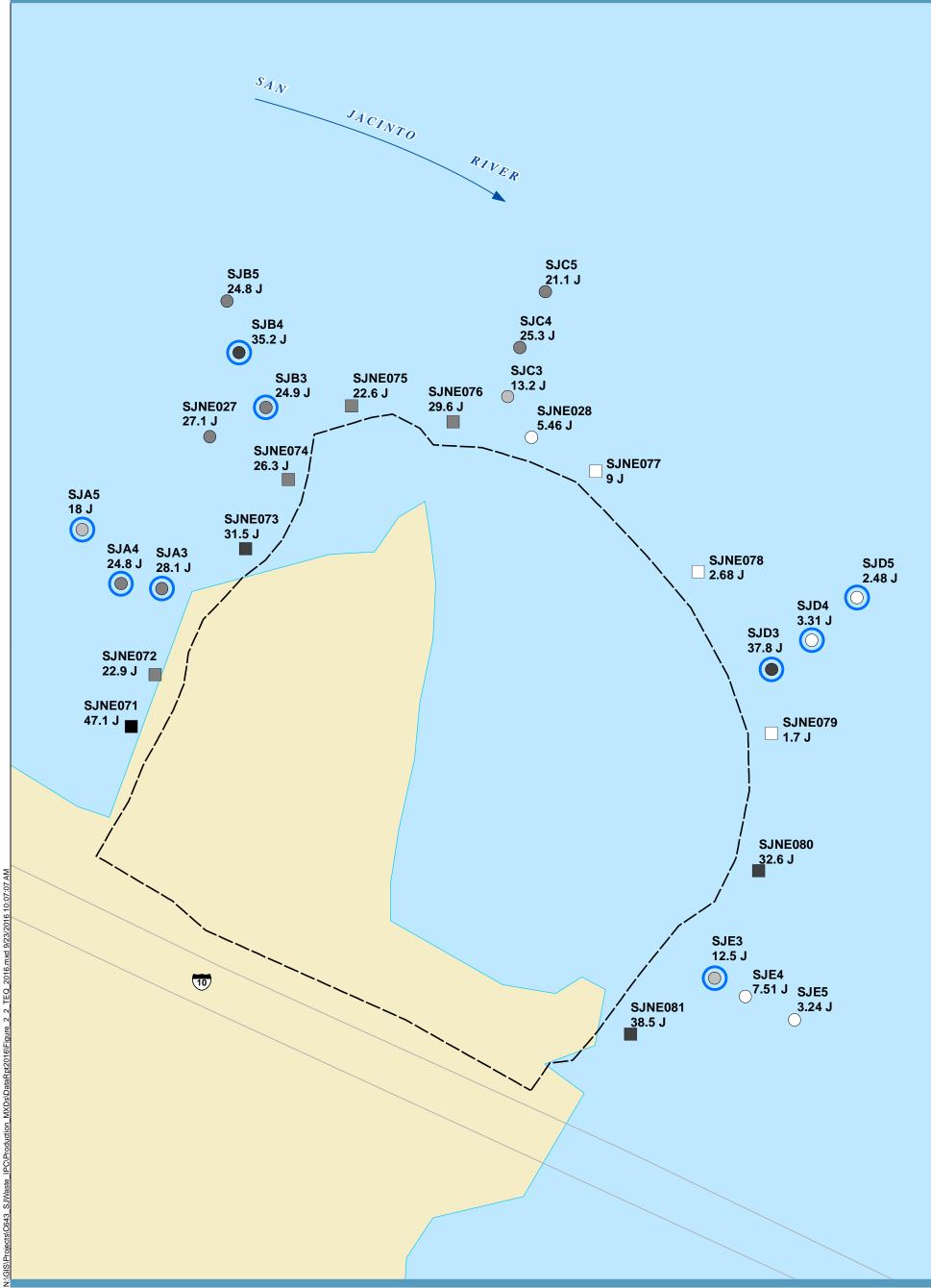
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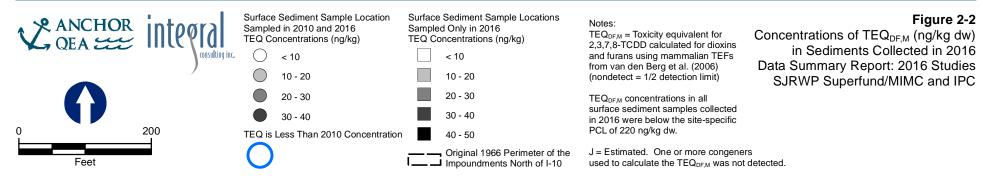
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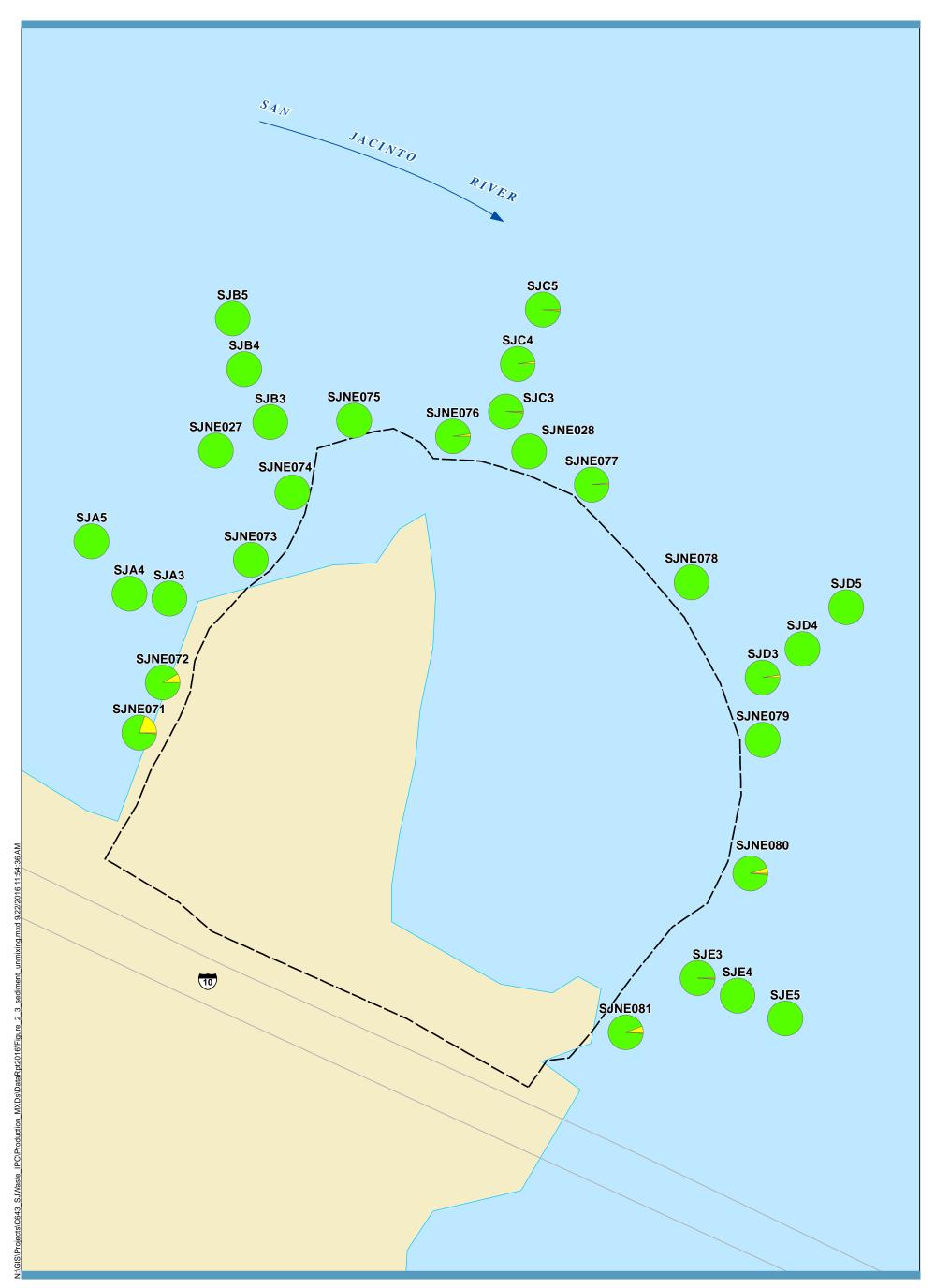
Approximate TCRA Footprint

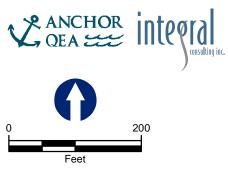
FEATURE SOURCES: Aerial Imagery: 1-Foot USGS EROS

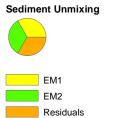
Figure 2-1 Sediment Stations Sampled for Surface Sediments in 2016 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC





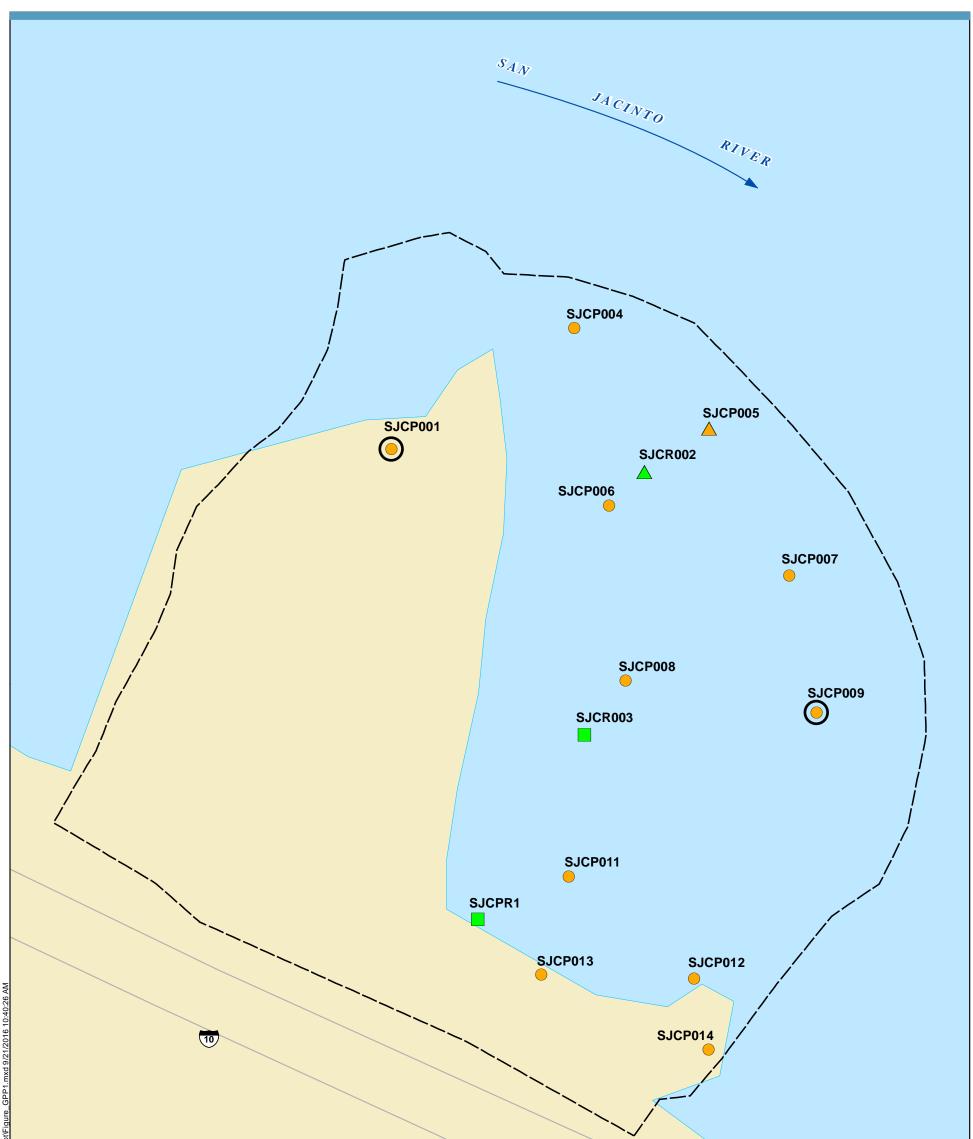






Original 1966 Perimeter of the

Figure 2-3 Results of Sediment Unmixing Analysis Using 2016 Sediment Data Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC



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Porewater Sampling Station

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Porewater Sampling Station with Surface Water

Performance Reference Compound Station

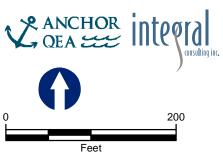
Performance Reference Compound Station With Surface Water

Location With Replicate Sampler

Original (1966) Perimeter of the Northern Impoundments

Figure 3-1 2016 TCRA Cap Porewater Sample Locations Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC





Groundwater Wells Sampled in 2016

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Groundwater Wells Sampled in 2011

Original (1966) Perimete of the Northern Impoundments

Figure 4-1 2016 Groundwater Sampling Locations, North of I-10 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC





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Groundwater Wells Sampled in 2012, 2013 and 2016

Groundwater Wells Sampled in 2013 and 2016

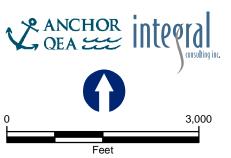
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The smaller of two approximate impoundment boundaries proposed by EPA on the basis of a 1964 aerial photograph.

The larger of two approximate impoundment boundaries proposed by EPA on the basis of historical drawings by the TSDH.

Figure 5-1 2016 Groundwater Sampling Locations, South of I-10 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC





USEPA's Preliminary Site Perimeter

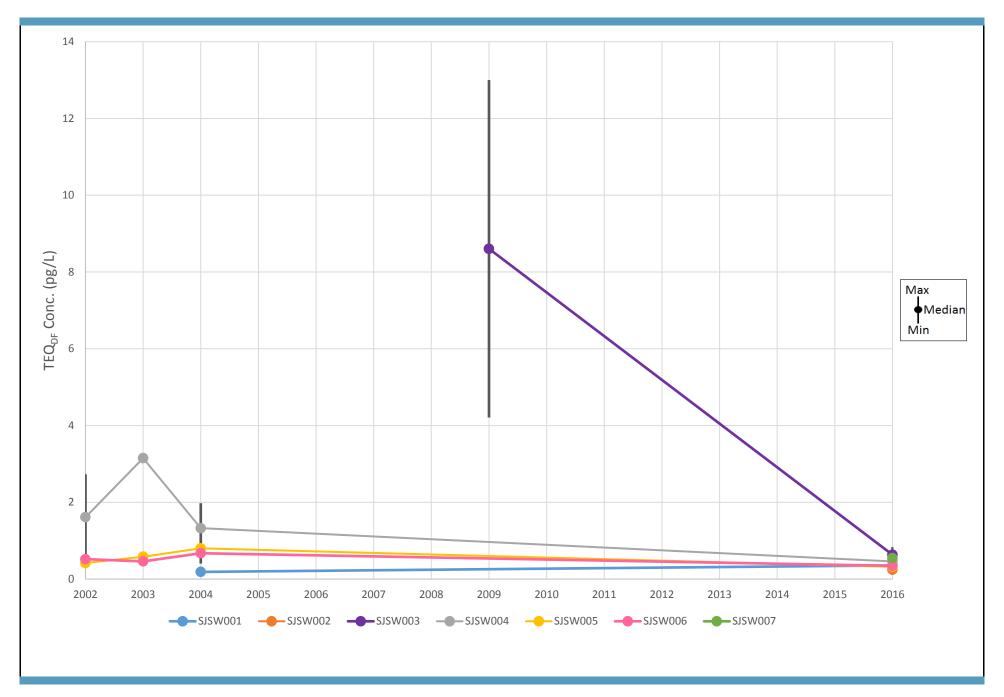
Original 1966 Perimeter of the

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Impoundments North of I-10

Surface Water Actual Sampling Location TMDL Station ID shown in ()

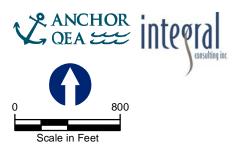
Figure 6-1 Locations of Surface Water Samples Collected in 2016 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC



**Figure 6-2** Trends in TEQ<sub>DF,M</sub> in Surface Water Over Time Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC

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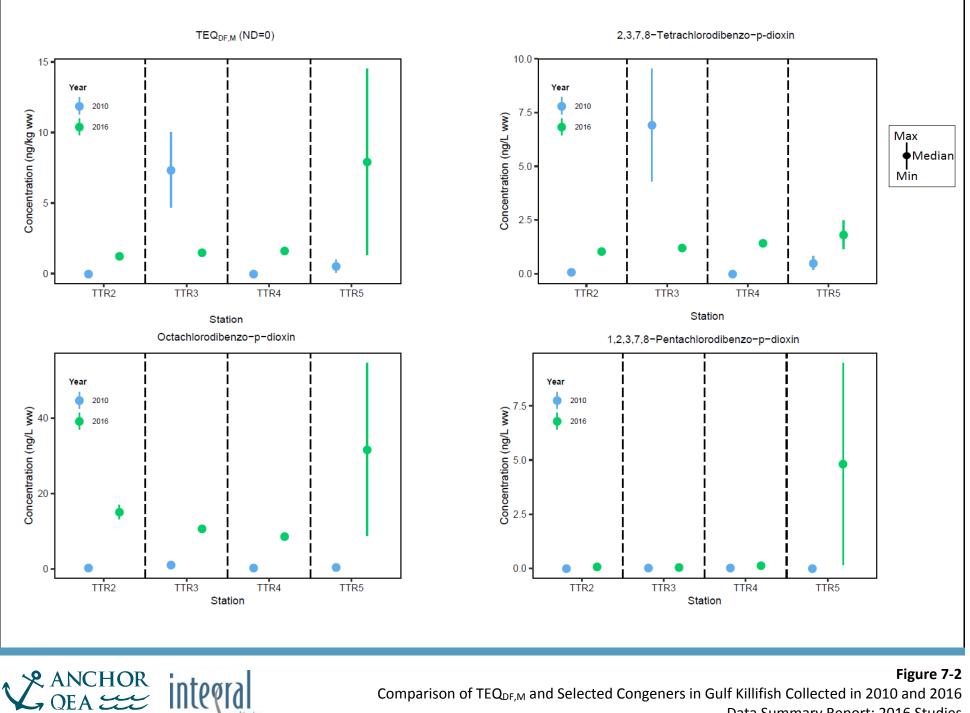


Preliminary Site Perimeter Small Fish

Original (1966) Perimeter of the Impoundments Figure 7-1 Locations of Gulf Killifish Samples Collected in 2016 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC

<sup>a</sup> Designation of the sand separation area is intended to be a general reference to areas in which such activities are believed to have taken place based on visual observations of aerial photography from 1998 through 2002.

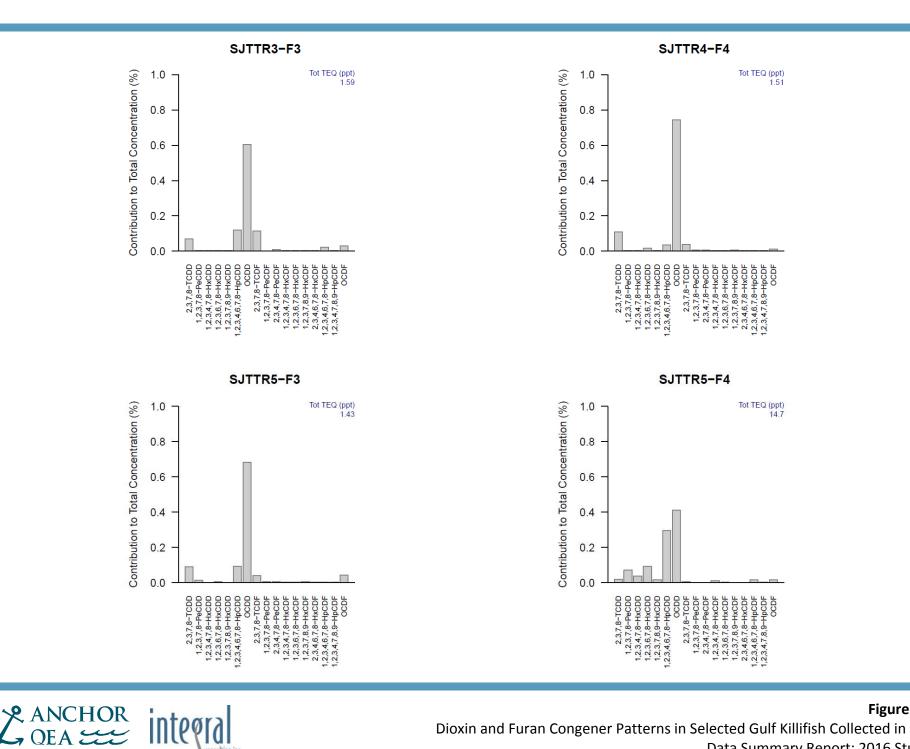
FEATURE SOURCES: Aerial Imagery: USGS



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## Figure 7-2

Comparison of TEQ<sub>DF,M</sub> and Selected Congeners in Gulf Killifish Collected in 2010 and 2016 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC

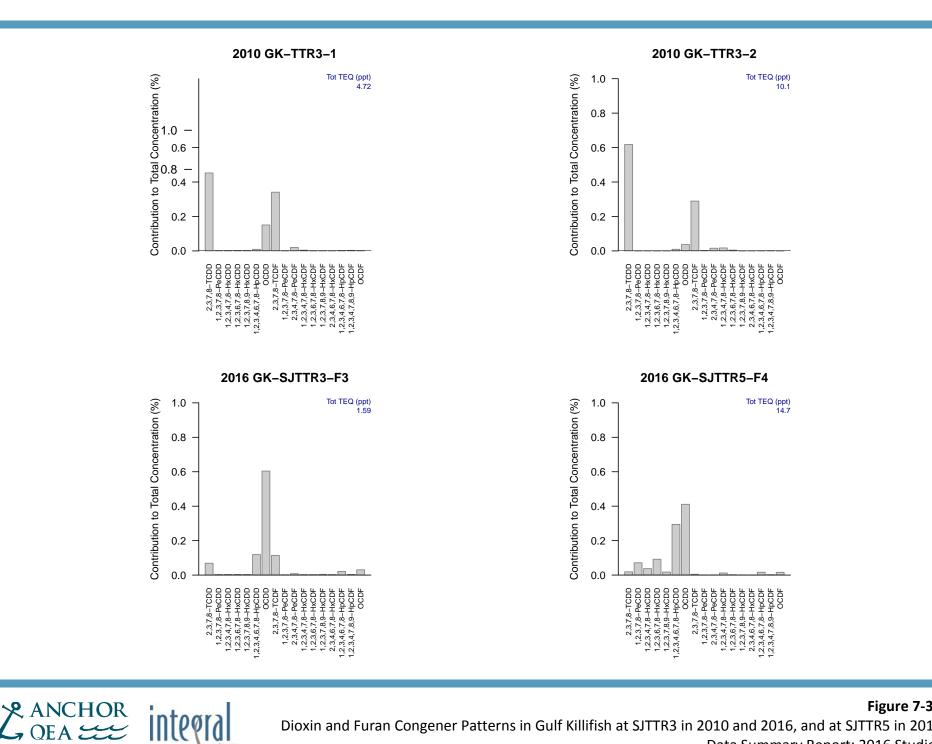


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## Figure 7-3a

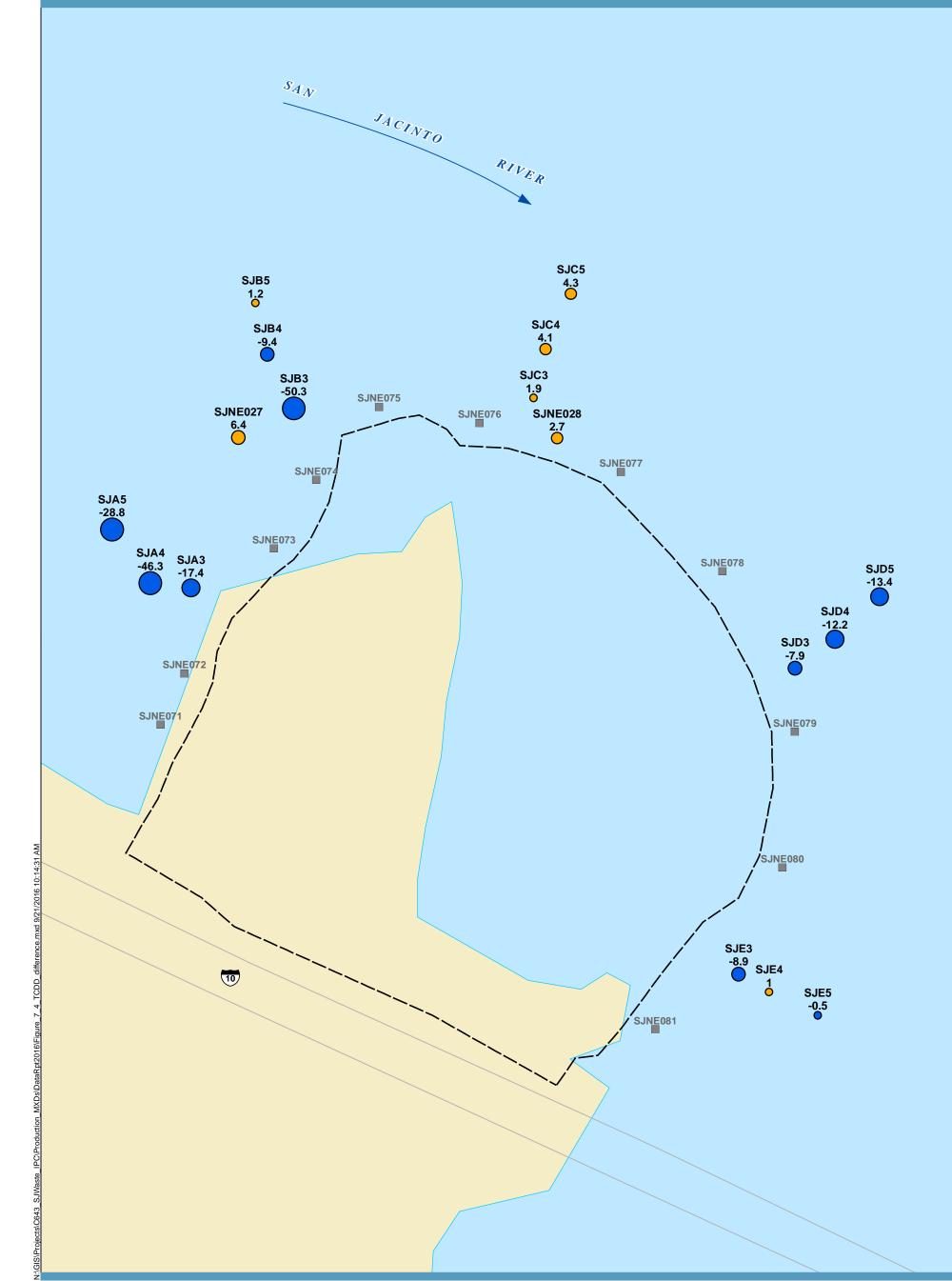
Dioxin and Furan Congener Patterns in Selected Gulf Killifish Collected in 2016 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC



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## Figure 7-3b

Dioxin and Furan Congener Patterns in Gulf Killifish at SJTTR3 in 2010 and 2016, and at SJTTR5 in 2016 Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC



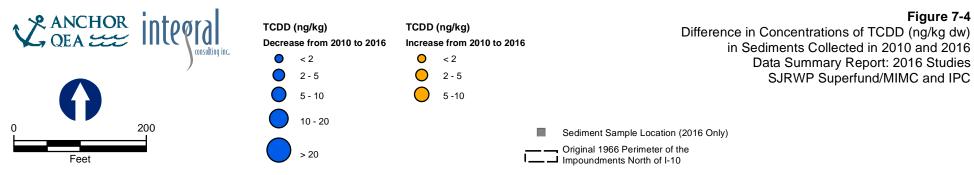
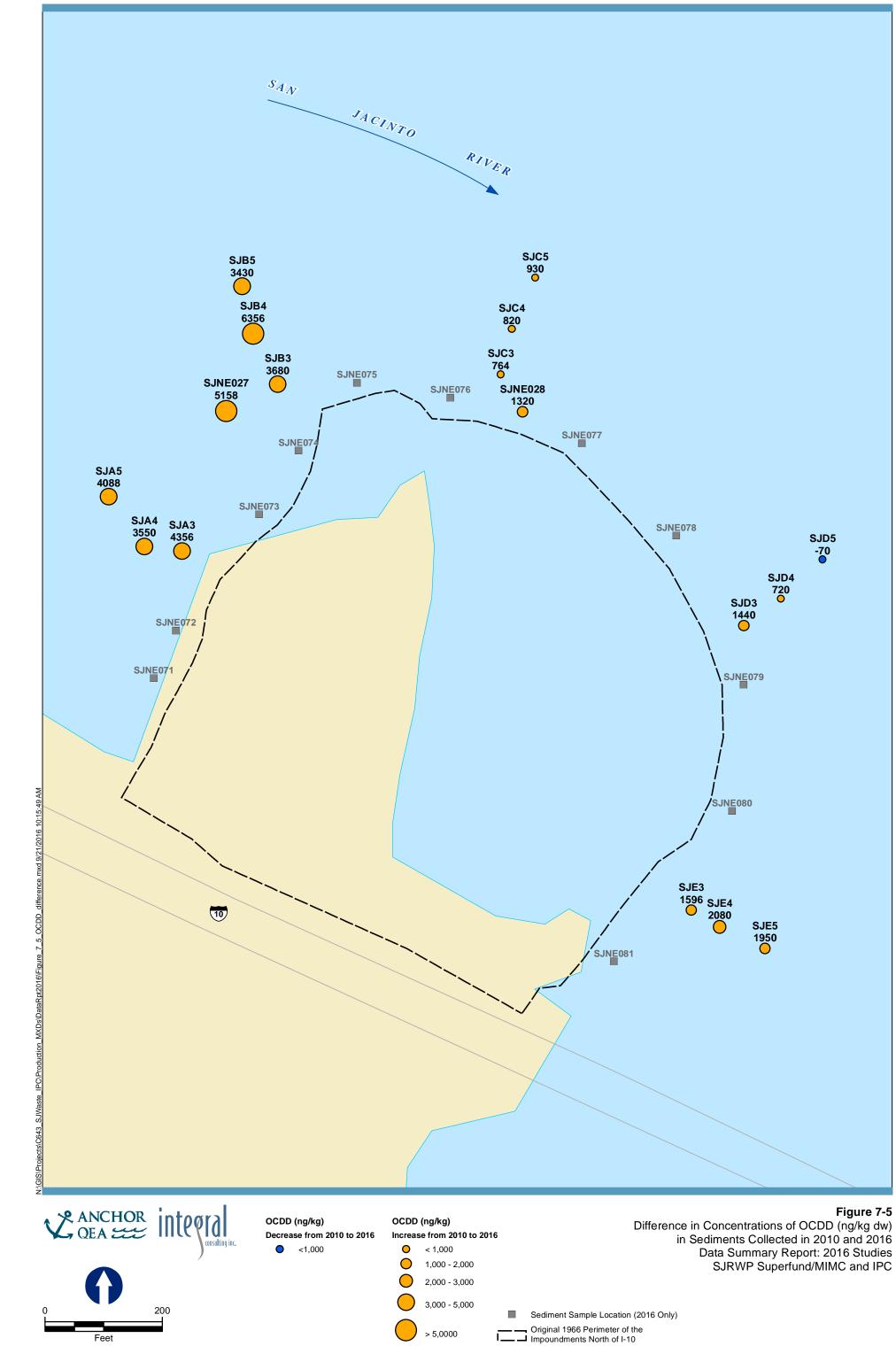
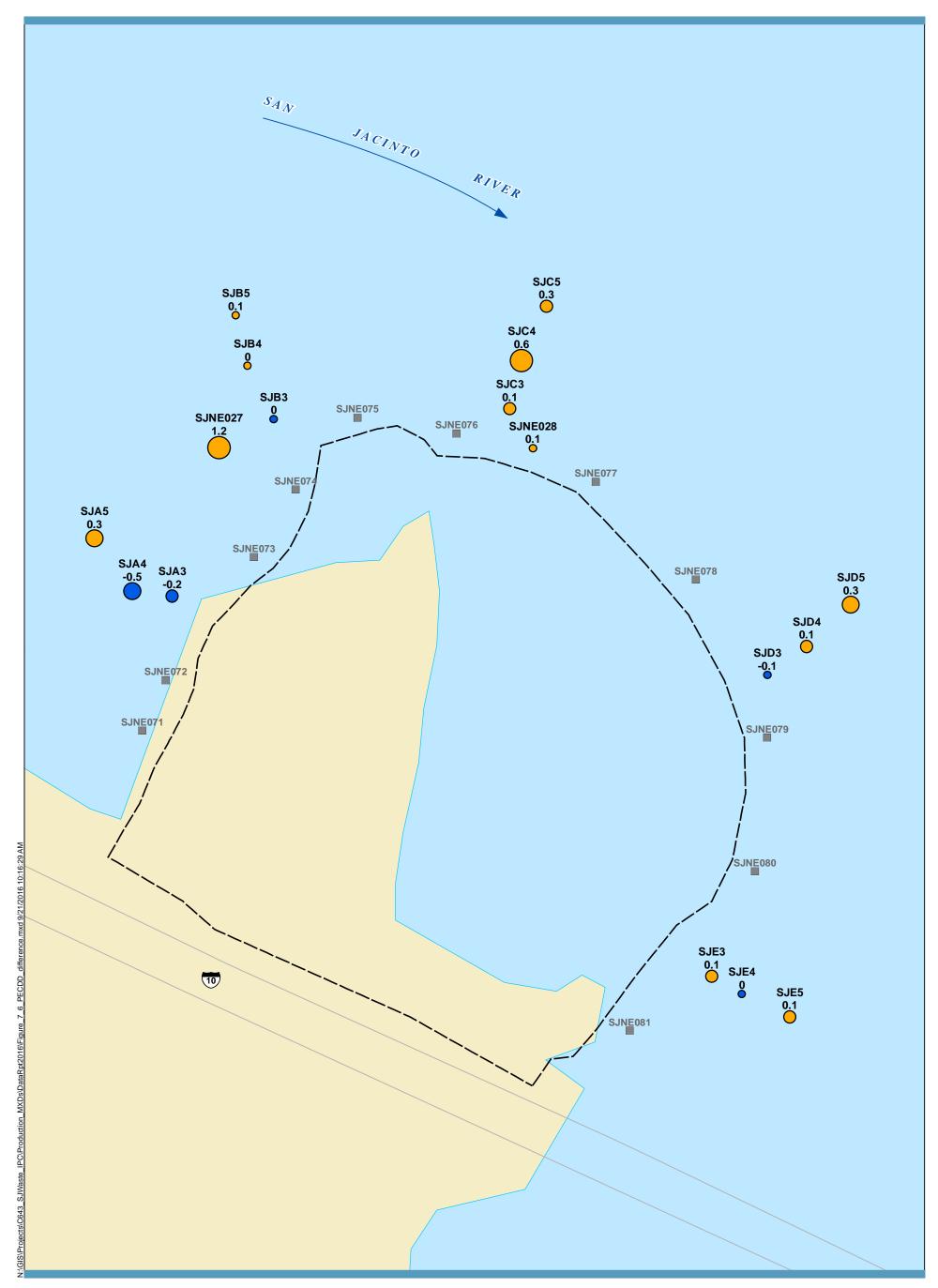
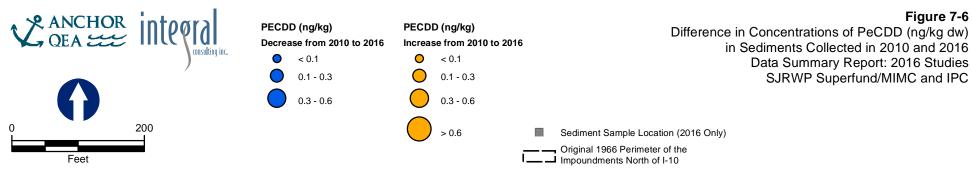
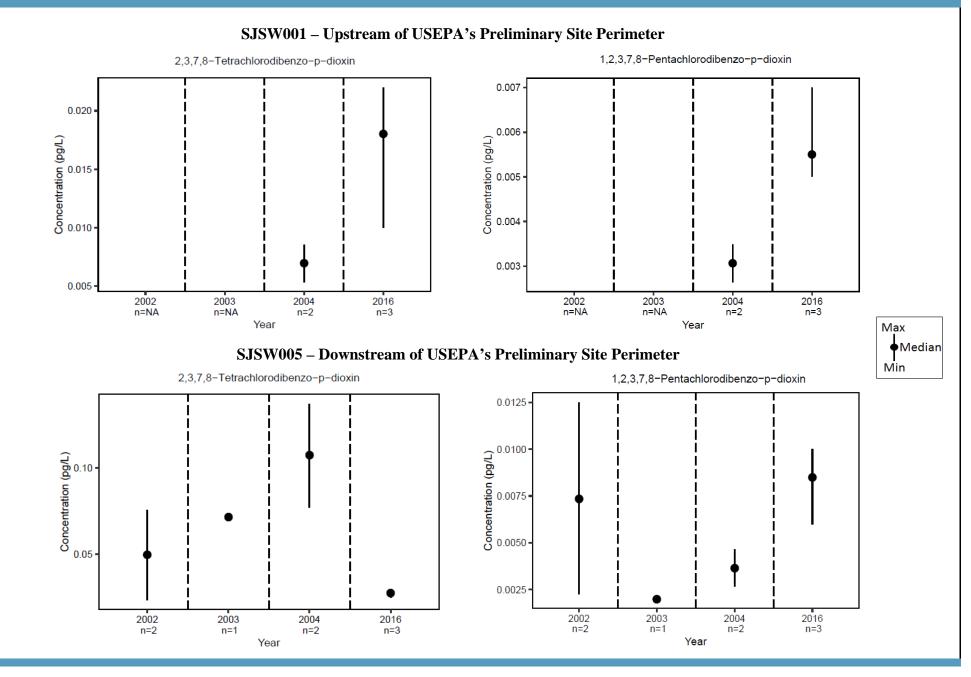


Figure 7-4









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## Figure 7-7

Dissolved Concentrations of TCDD and PeCDD in Surface Water by Sampling Year Data Summary Report: 2016 Studies SJRWP Superfund/MIMC and IPC

## TABLES

Table 2-1
Schedule of Sampling Events Conducted in 2016

Sampled Media	Field Activities Conducted by	<b>Mobilization Dates</b>	Description of Field Activities
Sediment	· · ·		
	Integral	May 6 to May 10, 2016	Collection of surface sediment from all stations except SJNE028, SJD4, and SJD5
		July 17, 2016	Collection of surface sediment from stations SJNE028, SJD4, and SJD5
Tissue			
	Integral	May 8 to May 10, 2016	Collection of Gulf Killifish from all transects
		July 16 to July 22, 2016	Collection of Gulf Killifish from transects SJTTR2 and SJTTR3 and Inland Silversides from
			transect SJTTR3
Surface Water			
	Integral	April 17 to April 18, 2016	No sampling conducted due to storm event
		May 24 to May 26, 2016	Collection of surface water at stations SJSW001, SJSW002, SJSW004, and SJSW005
			Sampling was suspended due to a storm event
		July 5 to July 22, 2016	Collection of surface water at all stations
Groundwater			
	Anchor	April 12 to April 13, 2016	Deployment of samplers at all wells except SJMW013
		May 14, 2016	Deployment of sampler at well SJMW013
		June 14 to June 15, 2016	Retrieval of samplers from all wells except SJMW013
		July 19, 2016	Retrieval of sampler from well SJMW013
Porewater	· · ·		
	USEPA	May 16 to May 18, 2016	Deployment of samplers
		May 15, 2016	Retrieval of performance reference fiber at SJCPR1
		July 19 to July 21, 2016	Retrieval of remaining samplers

Anchor = Anchor QEA

Integral = Integral Consulting Inc.

USEPA = U.S. Environmental Protection Agency

 Table 2-2

 Summary Statistics for Dioxins and Furans in Surface Sediments: 2010 vs. 2016

			20	)10			20	)16	
	Sample	Minimum	Maximum	Average	Median	Minimum	Maximum	Average	Median
Analyte	Number	(ng/kg ww)							
2,3,7,8-Tetrachlorodibenzo-p-dioxin	17	0.817	65.3	21.40	13.9	0.343	23.9	11.0	13.6
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	17	0.0342	0.936	0.27	0.176	0.1	1.2	0.4	0.298
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	17	0.0373	0.607	0.26	0.266	0.24	1.7	0.7	0.66
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	17	0.185	1.85	0.91	0.927	0.409	3.16	1.8	1.62
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	17	0.0685	2.08	0.84	0.725	0.408	4.35	1.7	1.55
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	17	6.83	58.2	32.66	31.7	40.4	181	95.0	101
Octachlorodibenzo-p-dioxin	17	242	2250	1145	1110	1450	7030	3609	3900
2,3,7,8-Tetrachlorodibenzofuran	17	2.09	220	70.81	45.7	0.8	91	37.9	45.3
1,2,3,7,8-Pentachlorodibenzofuran	17	0.0655	5.16	1.64	1.27	0.126	2.41	1.075	0.974
2,3,4,7,8-Pentachlorodibenzofuran	17	0.056	3.91	1.31	0.98	0.128	2.8	1.14	1.03
1,2,3,4,7,8-Hexachlorodibenzofuran	17	0.101	9.89	2.82	1.23	0.0645	4.15	2.10	2.03
1,2,3,6,7,8-Hexachlorodibenzofuran	17	0.0422	2.54	0.74	0.605	0.061	1.85	0.75	0.605
1,2,3,7,8,9-Hexachlorodibenzofuran	17	0.0246	0.243	0.07	0.0555	0.075	0.727	0.27	0.204
2,3,4,6,7,8-Hexachlorodibenzofuran	17	0.0235	0.798	0.20	0.172	0.0645	1.78	0.66	0.486
1,2,3,4,6,7,8-Heptachlorodibenzofuran	17	0.468	8	3.00	2.83	0.605	17.9	8.09	7.79
1,2,3,4,7,8,9-Heptachlorodibenzofuran	17	0.036	1.64	0.47	0.53	0.08	1.67	0.77	0.665
Octachlorodibenzofuran	17	0.147	67.2	27.28	21.1	4.97	228	79.08	73.8
TEQ <sub>DF,M</sub> (ND=0)	17	2.45	92.4	30.40	20.1	0.959	37.4	17.52	21.1
TEQ <sub>DF,M</sub> (ND=1/2DL)	17	2.65	92.4	30.50	20.2	2.48	37.8	18.52	21.1
TEQ <sub>DF,M</sub> (ND=DL)	17	2.85	92.4	30.59	20.2	4.01	38.2	19.50	25.1

Field duplicates were averaged for this analysis.

DL = detection limit

ND = non-detect

TEQ<sub>DF,M</sub> = TEQ calculated using Van den Berg et al. (2006) toxicity equivalency factors for mammals

ww = wet weight

Sample Location ID (depth)		2010			2016	
	EM1	EM2	Residual	EM1	EM2	Residual
SJA3(0-15 cm)	0.77	0.229	0.002	0.99	0	0.01
SJA4(0-10 cm)	0.804	0.195	0.001	0.991	0	0.009
SJA5(0-10 cm)	0.859	0.14	0.002	0.99	0	0.01
SJB3(0-15 cm)	0.857	0.141	0.002	0.992	0	0.008
SJB4(0-15 cm)	0.83	0.169	0.002	0.991	0	0.009
SJB5(0-15 cm)	0.944	0.054	0.002	0.99	0	0.01
SJC3(0-15 cm)	0.962	0.036	0.002	0.987	0	0.013
SJC4(0-15 cm)	0.957	0.04	0.003	0.969	0.023	0.008
SJC5(0-15 cm)	0.972	0.025	0.003	0.987	0	0.013
SJD3(0-15 cm)	0.946	0.053	0.001	0.966	0.025	0.009
SJD4(0-15 cm)	0.952	0.045	0.003	0.993	0	0.007
SJD5(0-15 cm)	0.967	0.031	0.002	0.994	0	0.006
SJE3(0-15 cm)	0.845	0.151	0.004	0.989	0	0.011
SJE4(0-15 cm)	0.995	0	0.005	0.995	0	0.005
SJE5(0-15 cm)	0.998	0	0.002	0.993	0	0.007
SJNE027 (0-15.24 cm)	0.833	0.165	0.003	0.992	0	0.008
SJNE028 (0-15.24 cm)	0.963	0.035	0.002	0.993	0	0.007
SJNE071(0-15 cm)				0.786	0.201	0.012
SJNE072(0-15 cm)				0.909	0.082	0.009
SJNE073(0-15 cm)				0.991	0	0.009
SJNE074(0-15 cm)				0.99	0	0.01
SJNE075(0-15 cm)				0.992	0	0.008
SJNE076(0-15 cm)				0.97	0.022	0.008
SJNE077(0-15 cm)				0.977	0.015	0.008
SJNE078(0-15 cm)				0.992	0	0.008
SJNE079(0-15 cm)				0.993	0	0.007
SJNE080(0-15 cm)				0.934	0.055	0.011
SJNE081(0-15 cm)				0.932	0.057	0.01

Table 2-3Results of Unmixing Analysis: 2010 vs. 2016

Residuals = proportion of the data that is not explained by the unmixing model

--- There is no data for 2010 at this location

EM = end member

 Table 3-1

 Mass of Each Target Compound in Each Porewater SPME Sample

Sampling Location	Sample Code	Sampler Type	Deployment Date	<b>Retrieval Date</b>	Depth Interval (inches)	2,3,7,8 TCDD	) (pg)	2,3,7,8 TCDF	(pg)	2,3,4,7,8-PeC	DF (pg)
SJCP001	SJCP-001-SP-1-A-DUP	SPME with duplicate	5/16/2016	7/19/2016	1-3	1.49	UJ	2.69	UJ	1.09	UJ
SJCP001	SJCP-001-SP-1-B-DUP	SPME with duplicate	5/16/2016	7/19/2016	4-6	1.08	UJ	0.970	UJ	0.478	UJ
SJCP001	SJCP-001-SP-1-C-DUP	SPME with duplicate	5/16/2016	7/19/2016	7-9	2.10	UJ	2.46	UJ	0.630	UJ
SJCP001	SJCP-001-SP-1-A	SPME	5/16/2016	7/19/2016	1-3	2.36	UJ	4.64	UJ	1.36	UJ
SJCP001	SJCP-001-SP-1-B	SPME	5/16/2016	7/19/2016	4-6	1.98	UJ	4.97	UJ	1.86	UJ
SJCP001	SJCP-001-SP-1-C	SPME	5/16/2016	7/19/2016	7-9	0.660	UJ	2.06	UJ	0.785	UJ
SJCP002	SJCP-002-SP-1-A	SPME	Not provided	Abandoned <sup>a</sup>	N/A	N/A		N/A		N/A	
SJCP002	SJCP-002-SP-1-B	SPME	Not provided	Abandoned <sup>a</sup>	N/A	N/A		N/A		N/A	
SJCP002	SJCP-002-SP-1-C	SPME	Not provided	Abandoned <sup>a</sup>	N/A	N/A		N/A		N/A	
SJCP003	SJCP-003-SP-1-A	SPME	Not provided	Abandoned <sup>a</sup>	N/A	N/A		N/A		N/A	
SJCP003	SJCP-003-SP-1-B	SPME	Not provided	Abandoned <sup>a</sup>	N/A	N/A		N/A		N/A	
SJCP003	SJCP-003-SP-1-C	SPME	Not provided	Abandoned <sup>a</sup>	N/A	N/A		N/A		N/A	
SJCP004	SJCP-004-SP-1-A	SPME	5/17/2016	7/21/2016	12-14	2.26	UJ	3.76	UJ	1.08	IJ
SJCP004	SJCP-004-SP-1-B	SPME	5/17/2016	7/21/2016	16-18	1.72	UJ	2.42	UJ	1.18	UJ
SJCP004	SJCP-004-SP-1-C	SPME	5/17/2016	7/21/2016	20-22	1.26	UJ	1.25	UJ	1.13	UJ
SJCP005	SJCP-005-SP-1-A-W	SPME with surface water	N/A	Abandoned <sup>b</sup>	N/A	N/A		N/A		N/A	
SJCP005	SJCP-005-SP-1-A	SPME	5/17/2016	7/21/2016	2-4	1.46	UJ	0.900	UJ	1.05	IJ
SJCP005	SJCP-005-SP-1-B	SPME	5/17/2016	7/21/2016	5-7	1.24	UJ	1.70	UJ	0.935	UJ
SJCP005	SJCP-005-SP-1-C	SPME	5/17/2016	7/21/2016	8-10	1.08	UJ	1.44	UJ	1.02	UJ
SJCP006	SJCP-006-SP-1-A	SPME	5/17/2016	7/21/2016	0-2	1.83	UJ	1.98	UJ	0.955	UJ
SJCP006	SJCP-006-SP-1-B	SPME	5/17/2016	7/21/2016	2-4	1.46	UJ	1.66	UJ	0.715	UJ
SJCP006	SJCP-006-SP-1-C	SPME	5/17/2016	7/21/2016	4-6	2.00	UJ	2.40	UJ	0.850	UJ
SJCP007	SJCP-007-SP-1-A	SPME	5/17/2016	7/21/2016	3-5	1.36	UJ	1.14	UJ	0.535	UJ
SJCP007	SJCP-007-SP-1-B	SPME	5/17/2016	7/21/2016	10-12	1.56	UJ	1.83	UJ	0.675	UJ
SJCP007	SJCP-007-SP-1-C	SPME	5/17/2016	7/21/2016	18-20	1.54	UJ	2.07	UJ	0.920	UJ
SJCP008	SJCP-008-SP-1-A-W	SPME with surface water	5/18/2016	7/20/2016	Surface water (6-8 inch)	1.55	UJ	1.73	UJ	0.980	UJ
SJCP008	SJCP-008-SP-1-A	SPME	5/18/2016	7/20/2016	1-3	4.91	UJ	RS		1.08	UJ
SJCP008	SJCP-008-SP-1-B	SPME	5/18/2016	7/20/2016	4-6	1.58	UJ	0.88	UJ	1.12	UJ
SJCP008	SJCP-008-SP-1-C	SPME	5/18/2016	7/20/2016	7-9	1.78	UJ	2.10	UJ	0.760	UJ
SJCP009	SJCP-009-SP-1-A-DUP	SPME with duplicate	5/18/2016	7/21/2016	3-5	1.76	UJ	2.08	UJ	0.94	UJ
SJCP009	SJCP-009-SP-1-B-DUP	SPME with duplicate	5/18/2016	7/21/2016 7/21/2016	10-12 17-19	1.84 2.52	LU LU	2.10 2.86	UJ	1.07 1.30	LU LU
SJCP009 SJCP009	SJCP-009-SP-1-C-DUP SJCP-009-SP-1-A	SPME with duplicate SPME	5/18/2016 5/18/2016	, ,	3-5	1.65	UJ	1.53	UJ	0.765	UJ
SJCP009	SJCP-009-SP-1-A	SPINE	5/18/2016	7/21/2016 7/21/2016	10-12	1.05	UJ	1.60	UJ	0.760	UJ
SJCP009	SJCP-009-SP-1-C	SPME	5/18/2016	7/21/2010	17-19	1.30	UJ	1.39	101	0.700	10
	SJCP-010-SP-1-A	SPME	5/18/2016	7/20/2016	1-3	N/A <sup>c</sup>	0,	N/A <sup>c</sup>	03	N/A <sup>c</sup>	0,
SJCP010		SPME	5/18/2016	7/20/2016	4-6	N/A <sup>c</sup>		N/A <sup>c</sup>		N/A <sup>c</sup>	
SJCP010	SJCP-010-SP-1-B	SPIME	5/18/2016	7/20/2016	4-6 7-9	N/A <sup>c</sup>		N/A N/A C		N/A <sup>c</sup>	
SJCP010	SJCP-010-SP-1-C	-			-	,		,			
SJCP011 SJCP011	SJCP-011-SP-1-A	SPME SPME	5/18/2016 5/18/2016	7/20/2016	1-3 4.5-6.5	1.14 1.34	LU LU	2.36	UJ	0.84	LU LU
SJCP011 SJCP011	SJCP-011-SP-1-B SJCP-011-SP-1-C	SPME	5/18/2016	7/20/2016	4.5-6.5 8-10	0.955	UJ	2.06	UJ	0.755	UJ
SJCP011 SJCP012	SJCP-011-SP-1-C SJCP-012-SP-1-A	SPINE	5/18/2016	7/20/2016	8-10 1-3	1.80	UJ	1.98	UJ	0.755	UJ

Table 3-1 Mass of Each Target Compound in Each Porewater SPME Sample

Sampling Location	Sample Code	Sampler Type	Deployment Date	<b>Retrieval Date</b>	Depth Interval (inches)	2,3,7,8 TCDD	(pg)	2,3,7,8 TCDF	(pg)	2,3,4,7,8-PeCD	DF (pg)
SJCP012	SJCP-012-SP-1-B	SPME	5/18/2016	7/21/2016	4-6	0.805	UJ	0.975	UJ	0.62	UJ
SJCP012	SJCP-012-SP-1-C	SPME	5/18/2016	7/21/2016	7-9	1.70	UJ	1.51	UJ	0.87	UJ
SJCP013	SJCP-013-SP-1-A	SPME	5/16/2016	7/19/2016	1-3	0.620	UJ	2.15	UJ	1.13	UJ
SJCP013	SJCP-013-SP-1-B	SPME	5/16/2016	7/19/2016	4-6	4.00	IJ	4.82	UJ	0.555	UJ
SJCP013	SJCP-013-SP-1-C	SPME	5/16/2016	7/19/2016	7-9	4.80	IJ	4.16	UJ	1.64	UJ
SJCP014	SJCP-014-SP-1-A	SPME	5/18/2016	7/20/2016	1-3	4.37	UJ	2.94	UJ	0.620	UJ
SJCP014	SJCP-014-SP-1-B	SPME	5/18/2016	7/20/2016	8.5-10.5	0.895	UJ	1.20	UJ	1.24	UJ
SJCP014	SJCP-014-SP-1-C	SPME	5/18/2016	7/20/2016	14-16	0.715	UJ	1.51	UJ	0.585	UJ

N/A = not applicable

RS = rejected due to very low surrogate recoveries (<10%)

SPME = solid phase microextraction

U = Compound analyzed, but not detected above detection limit

UJ = Compound analyzed, but not detected above estimated detection limit

<sup>a</sup> Locations were abandoned per instruction from USEPA. A storm event deposited 6-16 inches of sand over the sampling locations, and samplers could not be found.

<sup>b</sup> Location was abandoned per instruction from USEPA. A storm event separated the surface water sampler from the porewater sampler, and the surface water sampler could not be found. <sup>c</sup> Sampler not analyzed; sampler was found on top of cap.

2,3,7,8-TCDD = 2,3,7,8-tetrachlorodibenzo-*p*-dioxin

2,3,7,8-TCDF = 2,3,7,8-tetrachlorodibenzofuran

2,3,4,7,8-PeCDF= 2,3,4,7,8-pentachlorodibenzofuran

 Table 3-2

 Mass of Each Performance Reference Compound at the Beginning and End of Deployment Period

Sampling					Depth Interval								
Location	Sample Code	Sampler Type	Deployment Date	<b>Retrieval Date</b>	(inches)	<sup>13</sup> C-2,3,7,8-TCDD (	pg)	<sup>13</sup> C-2,3,7,8-TCDF (p	og)	<sup>13</sup> C-2,3,4,7,8-PeCD	)F (pg)		
N/A	05152016-SJPW201	Initial PRC	N/A	N/A	N/A	52.0		58.1		87.2			
N/A	05152016-SJPW202	Initial PRC	N/A	N/A	N/A	59.0		69.4		93.8			
N/A	05152016-SJPW203	Initial PRC	N/A	N/A	N/A	57.5		67.3		85.7			
N/A	05152016-SJPW204	Initial PRC	N/A	N/A	N/A	61.9		69.9		85.0			
N/A	05152016-SJPW205	Initial PRC	N/A	N/A	N/A	46.3		50.9		74.4			
N/A	05152016-SJPW206	Initial PRC	N/A	N/A	N/A	42.2		41.6		79.8			
SJCPR1	SJCPR1-PW-2-A	PRC	Not Provided	6/15/2016	3-5	Not Usable <sup>a</sup>		Not Usable <sup>a</sup>		28.6	J		
SJCPR1	SJCPR1-PW-2-B	PRC	Not Provided	6/15/2016	6-8	Not Usable <sup>a</sup>		Not Usable <sup>a</sup>		59.1			
SJCPR1	SJCPR1-PW-2-C	PRC	Not Provided	6/15/2016	9-11	Not Usable <sup>a</sup>		Not Usable <sup>a</sup>		60.9			
SJCPR2	SJCPR2-SP-2-A	PRC	5/16/2016	7/20/2016	3-5	Not Usable <sup>b</sup>		Not Usable <sup>b</sup>		Not Usable <sup>b</sup>			
SJCPR2	SJCPR2-SP-2-B	PRC	5/16/2016	7/20/2016	6-8	Not Usable <sup>b</sup>		Not Usable <sup>b</sup>		Not Usable <sup>b</sup>		Not Usable <sup>b</sup>	
SJCPR2	SJCPR2-SP-2-C	PRC	5/16/2016	7/20/2016	9-11	Not Usable <sup>b</sup>		Not Usable <sup>b</sup>		Not Usable <sup>b</sup>			
SJCR001	SJCR-001-SP-2-A	PRC	Not Provided	Abandoned <sup>b</sup>	N/A	N/A		N/A		N/A			
SJCR001	SJCR-001-SP-2-B	PRC	Not Provided	Abandoned <sup>b</sup>	N/A	N/A		N/A		N/A			
SJCR001	SJCR-001-SP-2-C	PRC	Not Provided	Abandoned <sup>b</sup>	N/A	N/A		N/A		N/A			
SJCR002	SJCR-002-SP-2-A-W	PRC with surface water	5/17/2016	7/21/2016	5-7	1.84	U	1.37	U	5.84	J		
SJCR002	SJCR-002-SP-2-A	PRC	5/17/2016	7/21/2016	5-7	27.9		22.1		31.4	J		
SJCR002	SJCR-002-SP-2-B	PRC	5/17/2016	7/21/2016	11-13	13.8	U	29.7		44.1	J		
SJCR002	SJCR-002-SP-2-C	PRC	5/17/2016	7/21/2016	18-20	25.6		24.2		39.6	J		
SJCR003	SJCR-003-SP-2-A	PRC	5/18/2016	7/20/2016	11-13	2.35	U	2.98	U	11.4	J		
SJCR003	SJCR-003-SP-2-B	PRC	5/18/2016	7/20/2016	14-16	2.78	U	6.54		11.5	J		
SJCR003	SJCR-003-SP-2-C	PRC	5/18/2016	7/20/2016	17-19	4.45	U	5.98		13.2	J		
SJCR004	SJCR-004-SP-2-A	PRC	5/18/2016	7/20/2016	3-5	Not Usable <sup>b</sup>		Not Usable <sup>b</sup>		Not Usable <sup>b</sup>			
SJCR004	SJCR-004-SP-2-B	PRC	5/18/2016	7/20/2016	6-8	Not Usable <sup>b</sup>		Not Usable <sup>b</sup>		Not Usable <sup>b</sup>			
SJCR004	SJCR-004-SP-2-C	PRC	5/18/2016	7/20/2016	9-11	Not Usable <sup>b</sup>		Not Usable <sup>b</sup>		Not Usable <sup>b</sup>			

J = estimated value

N/A = not applicable

PRC = performance reference compound

U = compound analyzed, but not detected

<sup>a</sup> The fiber at station SJCR001 could not be located for retrieval. The fibers at Stations SJCPR2 and SJCR004 were not usable due to a communication error with the laboratory. Refer to Appendix C for details.

<sup>b</sup> The fiber at station SJCR001 could not be located for retrieval. The fibers at Stations SJCPR2 and SJCR004 were not usable due to a communication error with the laboratory. Refer to Appendix C for details.

 $^{13}$ C-2,3,7,8-TCDD =  $^{13}$ C-2,3,7,8-tetrachlorodibenzo-*p* -dioxin

 $^{13}$ C-2,3,7,8-TCDF =  $^{13}$ C-2,3,7,8-tetrachlorodibenzofuran

<sup>13</sup>C-2,3,4,7,8-PeCDF= <sup>13</sup>C-2,3,4,7,8-pentachlorodibenzofuran

 Table 4-1

 Estimated Concentrations of Target Compounds and TEQ<sub>DF.M</sub> in Groundwater North of I-10

					Concentrations in Groundwater (pg/L)															
Sampling Location	Sample Code	Deployment Date	<b>Retrieval Date</b>	Fiber Length (mm)	2,3,7,8-TC	2,3,7,8-TCDD		2,3,7,8-TCDD		2,3,7,8-TCDD		2,3,7,8-TCDD		2,3,7,8-TCDD 2,3,7,8-TCDF		DF	2,3,4,7,8-PeCDF		DF TEQ <sub>DF,M</sub> (ND=	
SJMW010	SJMW010GW0510	4/13/2016	6/15/2016	1,419.82	0.003	U	0.013	U	0.002	U	0.005	U								
SJMW011	SJMW011GW50.555.5	4/13/2016	6/15/2016	1,415.08	0.015	U	0.033	U	0.007	U	0.021	U								
SJMW012	SJMW012GW09.814.8	4/13/2016	6/15/2016	1,431.94	0.005	U	0.053 <sup>a</sup>	U	0.004	U	0.011 <sup>a</sup>	U								
SJMW012 - Dup	SJMW1012GW09.814.8	4/13/2016	6/15/2016	1,422.70	0.003	U	0.013	U	0.002	U	0.005	U								
SJMW013 <sup>b</sup>	SJMW013GW50.355.3	5/14/2016	7/19/2016	1,032.77	0.010	UJ	0.044	UJ	0.003	UJ	0.015	UJ								

DL = detection limit

I-10 = Interstate Highway 10

J = estimated value

ND = non-detect

PRC = performance reference compounds

SPME = solid phase microextraction

 $TEQ_{DF,M}$  = TEQ calculated using Van den Berg et al. (2006) for mammals

U = compound analyzed, but not detected above detection limit

UJ = compound analyzed, but not detected above estimated detection limit

2,3,7,8-TCDD = 2,3,7,8-tetrachlorodibenzo-*p*-dioxin

2,3,7,8-TCDF = 2,3,7,8-tetrachlorodibenzofuran

2,3,4,7,8-PeCDF = 2,3,4,7,8-Pentachlorodibenzofuran

<sup>a</sup> Correction for equilibrium was greater than 90 percent, which imparts greater uncertainty to the calculation.

<sup>b</sup> Due to unreliable PRC data for this sampler, equilibrium corrections are based on the average equilibrium conditions of all other groundwater SPME samplers as described in the report.

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Table 4-2 Mass of Each Performance Reference Compound at the Beginning and End of Deployment Period—Northern Impoundments

Sampling Location	Sample Function	Sample Code	Deployment Date	Retrieval Date	Fiber Length (mm)	<sup>37</sup> Cl-2,3,7,8-TCDD (pg)	<sup>13</sup> C-1,2,3,4-TCDF (pg)	<sup>13</sup> C-2,3,4,7,8-PeCDF (pg)
N/A	Initial PRC	04072016SJGW11 <sup>a</sup>	N/A	N/A	1,512.00	39.6	89.9	67.0
N/A	Initial PRC	04072016SJGW12 <sup>a</sup>	N/A	N/A	1,500.00	44.7	108	72.9
N/A	Initial PRC	04072016SJGW13 <sup>a</sup>	N/A	N/A	1,500.00	36.1	99.4	65.9
N/A	Initial PRC	04072016SJGW14 <sup>a</sup>	N/A	N/A	1,508.00	40.4	104	73.4
N/A	Initial PRC	04072016SJGW15 <sup>a</sup>	N/A	N/A	1,501.00	40.3	91.1	63.2
N/A	Initial PRC	05132016SJGW300 <sup>b</sup>	N/A	N/A	814.16	19.1	41.7	31.2
N/A	Initial PRC	05132016SJGW301 <sup>b</sup>	N/A	N/A	721.12	15.3	30.5	27.6
N/A	Initial PRC	05132016SJGW302 <sup>b</sup>	N/A	N/A	866.82	15.1	30.0	29.3
N/A	Initial PRC	05132016SJGW303 <sup>b</sup>	N/A	N/A	846.13	17.3	35.7	31.2
SJMW010	Final PRC	SJMW010GW0510	4/13/2016	6/15/2016	1,419.82	24.5	73.1	45.4
SJMW011	Final PRC	SJMW011GW50.555.5	4/13/2016	6/15/2016	1,415.08	21.7	65.9	33.5
SJMW012	Final PRC	SJMW012GW09.814.8	4/13/2016	6/15/2016	1,431.94	32.1	88.4	56.3
SJMW012 - Dup	Final PRC	SJMW1012GW09.814.8	4/13/2016	6/15/2016	1,422.70	30.5	77.8	44.3
SJMW013 <sup>a</sup>	Final PRC	SJMW013GW50.355.3	5/14/2016	7/19/2016	1,032.77	20.9	53.0	34.1

N/A = not applicable

PRC = performance reference compound

 $^{37}$ Cl-2,3,7,8-TCDD =  $^{37}$ Cl-2,3,7,8-tetrachlorodibenzo-*p* -dioxin

<sup>13</sup>C-1,2,3,4-TCDF = <sup>13</sup>C-1,2,3,4-tetrachlorodibenzofuran

<sup>13</sup>C-2,3,4,7,8-PeCDF = <sup>13</sup>C-2,3,4,7,8-pentachlorodibenzofuran

<sup>a</sup> Samples 04072016SJGW11 to 04072016SJGW15 were used to determine the initial PRC concentrations and were sent directly to the analytical laboratory. These samples were not deployed in the field.

<sup>b</sup> Samples 05132016SJGW300 to 05132016SJGW303 were intended to determine the initial PRC concentrations for sampling location SJMW013 that was deployed later than other samplers. However, recoveries in SJMW013 upon retrieval were close to the initial concentrations. See report for details of calculations for SJMW013.

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 Table 4-3

 Mass of Each Target Compound in Each Groundwater SPME Sample North of I-10

					Concentrations in Fibers (pg)							
Sampling Location	Sample Code	Deployment Date	<b>Retrieval Date</b>	Fiber Length (mm)	er Length (mm) 2,3,7,8-TCDD 2,3,7,8-TCDF		2,3,7,8-TCDF		2,3,4,7,8-Pe	CDF		
SJMW010	SJMW010GW0510	4/13/2016	6/15/2016	1,419.82	0.780	U	0.640	U	0.665	U		
SJMW011	SJMW011GW50.555.5	4/13/2016	6/15/2016	1,415.08	4.94	U	2.23	U	3.83	U		
SJMW012	SJMW012GW09.814.8	4/13/2016	6/15/2016	1,431.94	0.585	U	0.720	U	0.640	U		
SJMW012 - Dup	SJMW1012GW09.814.8	4/13/2016	6/15/2016	1,422.70	0.432	U	0.520	U	0.845	U		
SJMW013	SJMW013GW50.355.3	5/14/2016	7/19/2016	1,032.77	1.94	UJ	1.94	UJ	0.935	UJ		

I-10 = Interstate Highway 10

SPME = solid phase microextraction

U = compound analyzed, but not detected above detection limit

UJ = compound analyzed, but not detected above estimated detection limit

2,3,7,8-TCDD = 2,3,7,8-tetrachlorodibenzo-*p* -dioxin

2,3,7,8-TCDF = 2,3,7,8-tetrachlorodibenzofuran

2,3,4,7,8-PeCDF = 2,3,4,7,8-pentachlorodibenzofuran

Sampling Location	Sample Function	Sample Code	Deployment Date	Retrieval Date	Fiber Length (mm)	<sup>37</sup> Cl-2,3,7,8-TCDD (pg)	<sup>13</sup> C-1,2,3,4-TCDF (pg)	<sup>13</sup> C-2,3,4,7,8-Pe	CDF (pg)
N/A	Initial PRC	04072016SJGW11 <sup>a</sup>	N/A	N/A	1,512.00	39.6	89.9	67.0	
N/A	Initial PRC	04072016SJGW12 <sup>a</sup>	N/A	N/A	1,500.00	44.7	108	72.9	
N/A	Initial PRC	04072016SJGW13 <sup>a</sup>	N/A	N/A	1,500.00	36.1	99.4	65.9	
N/A	Initial PRC	04072016SJGW14 <sup>a</sup>	N/A	N/A	1,508.00	40.4	104	73.4	
N/A	Initial PRC	04072016SJGW15 <sup>a</sup>	N/A	N/A	1,501.00	40.3	91.1	63.2	
SJMW001	Final PRC	SJMW001GW09.514.5	4/13/2016	6/15/2016	1,438.39	18.8	69.2	19.7	U
SJMW002	Final PRC	SJMW002GW07.512.5	4/13/2016	6/15/2016	1,434.27	21.6	63.0	17	U
SJMW003	Final PRC	SJMW003GW1015	4/13/2016	6/15/2016	1,438.08	26.4	66.5	51.5	
SJMW004S	Final PRC	SJMW004GW1217	4/12/2016	6/14/2016	1,435.17	21.4	54.1	35.7	
SJMW004D	Final PRC	SJMW004DGW77.582.5	4/13/2016	6/15/2016	1,508.07	26.6	77.1	50.8	
SJMW004D - Dup	Final PRC	SJMW1004DGW77.582.6	4/13/2016	6/15/2016	1,433.44	27.8	80.0	41.5	
SJMW005	Final PRC	SJMW005GW1318	4/12/2016	6/14/2016	1,508.74	23.8	62.9	40.3	
SJMW006	Final PRC	SJMW006GW1015	4/12/2016	6/14/2016	1,429.34	31.5	78.4	44.5	
SJMW007	Final PRC	SJMW007GW0712	4/12/2016	6/14/2016	1,435.32	24.9	74.3	47.8	
SJMW008	Final PRC	SJMW008GW2025	4/12/2016	6/14/2016	1,512.42	21.1	59.7	45.0	
SJMW009	Final PRC	SJMW009GW19.524.5	4/13/2016	6/15/2016	1,431.97	22.2	66.6	40.8	

 Table 5-1

 Mass of Each Performance Reference Compound at the Beginning and End of Deployment Period—Southern Impoundments

N/A = not applicable

PRC = performance reference compound

U = compound analyzed, but not detected above detection limit

 $^{37}$ Cl-2,3,7,8-TCDD =  $^{37}$ Cl-2,3,7,8-tetrachlorodibenzo-*p*-dioxin

 $^{13}$ C-1,2,3,4-TCDF =  $^{13}$ C-1,2,3,4-tetrachlorodibenzofuran

<sup>13</sup>C-2,3,4,7,8-PeCDF = <sup>13</sup>C-2,3,4,7,8-pentachlorodibenzofuran

<sup>a</sup> Samples 04072016SJGW11 to 04072016SJGW15 were used to determine the initial PRC concentrations and were sent directly to the analytical laboratory. These samples were not deployed in the field.

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Table 5-2Estimated Concentrations of Target Compounds and TEQ<sub>DF,M</sub> in Groundwater South of I-10

							Concer	ntrations in	Groundwater (p	g/L)		
Sampling Location	Sample Code	Deployment Date	<b>Retrieval Date</b>	Fiber Length (mm)	2,3,7,8-TC	DD	2,3,7,8-T	CDF	2,3,4,7,8-PeCDF		TEO	۲ ۲
SJMW001	SJMW001GW09.514.5	4/13/2016	6/15/2016	1,438.39	0.008	U	0.081	U	0.004	U	0.018	U
SJMW002	SJMW002GW07.512.5	4/13/2016	6/15/2016	1,434.27	0.023	U	0.058	U	0.003	U	0.030	U
SJMW003	SJMW003GW1015	4/13/2016	6/15/2016	1,438.08	0.053	J	0.970	J	0.004	U	0.151	J
SJMW004S	SJMW004GW1217	4/12/2016	6/14/2016	1,435.17	0.137	J	2.167		0.005	U	0.355	J
SJMW004D	SJMW004DGW77.582.5	4/13/2016	6/15/2016	1,508.07	0.004	U	0.014	U	0.001	U	0.005	U
SJMW004D - Dup	SJMW1004DGW77.582.6	4/13/2016	6/15/2016	1,433.44	0.004	U	0.021	U	0.001	U	0.006	U
SJMW005	SJMW005GW1318	4/12/2016	6/14/2016	1,508.74	0.003	U	0.014	U	0.001	U	0.005	U
SJMW006	SJMW006GW1015	4/12/2016	6/14/2016	1,429.34	0.041	U	0.118	U	0.012	U	0.056	U
SJMW007	SJMW007GW0712	4/12/2016	6/14/2016	1,435.32	0.004	U	0.015	U	0.002	U	0.006	U
SJMW008	SJMW008GW2025	4/12/2016	6/14/2016	1,512.42	0.003	U	0.012	U	0.001	U	0.004	U
SJMW009	SJMW009GW19.524.5	4/13/2016	6/15/2016	1,431.97	0.005	U	0.022	U	0.004	U	0.008	U

I-10 = Interstate Highway 10

J = estimated value

U = compound analyzed, but not detected above detection limit

2,3,7,8-TCDD = 2,3,7,8-tetrachlorodibenzo-*p* -dioxin

2,3,7,8-TCDF = 2,3,7,8-tetrachlorodibenzofuran

2,3,4,7,8-PeCDF = 2,3,4,7,8-pentachlorodibenzofuran

 Table 5-3

 Mass of Each Target Compound in Each Groundwater SPME Sample South of I-10

						C	oncentrations in <b>F</b>	ibers (pg)		
Sampling Location	Sample Code	Deployment Date	<b>Retrieval Date</b>	Fiber Length (mm)	2,3,7,8-TCD	D	2,3,7,8-TCI	DF	2,3,4,7,8-Pe	CDF
SJMW001	SJMW001GW09.514.5	4/13/2016	6/15/2016	1,438.39	3.38	U	5.10	U	2.96	U
SJMW002	SJMW002GW07.512.5	4/13/2016	6/15/2016	1,434.27	7.75	U	4.55	U	2.78	U
SJMW003	SJMW003GW1015	4/13/2016	6/15/2016	1,438.08	13.0	J	68.0	J	0.980	U
SJMW004S	SJMW004GW1217	4/12/2016	6/14/2016	1,435.17	47.2	J	219		2.62	U
SJMW004D	SJMW004DGW77.582.5	4/13/2016	6/15/2016	1,508.07	0.980	U	0.760	U	0.428	U
SJMW004D - Dup	SJMW1004DGW77.582.6	4/13/2016	6/15/2016	1,433.44	0.800	U	0.745	U	0.585	U
SJMW005	SJMW005GW1318	4/12/2016	6/14/2016	1,508.74	1.03	U	1.28	U	0.715	U
SJMW006	SJMW006GW1015	4/12/2016	6/14/2016	1,429.34	5.60	U	4.54	U	4.13	U
SJMW007	SJMW007GW0712	4/12/2016	6/14/2016	1,435.32	0.970	U	0.730	U	0.540	U
SJMW008	SJMW008GW2025	4/12/2016	6/14/2016	1,512.42	1.16	U	1.15	U	0.505	U
SJMW009	SJMW009GW19.524.5	4/13/2016	6/15/2016	1,431.97	1.67	U	1.53	U	1.46	U

I-10 = Interstate Highway 10

J = estimated value

SPME = solid phase microextraction

U = compound analyzed, but not detected above detection limit

2,3,7,8-TCDD = 2,3,7,8-tetrachlorodibenzo-p-dioxin

2,3,7,8-TCDF = 2,3,7,8-tetrachlorodibenzofuran

2,3,4,7,8-PeCDF = 2,3,4,7,8-pentachlorodibenzofuran

TMDL Station ID		11197			SJSW002			TCEQ2009_03		11193			
2016 Station ID	SJSW001-1	SJSW001-2	SJSW001-3	SJSW002-1	SJSW002-2	SJSW002-3	SJSW003-1	SJSW003-2	SJSW003-3	SJSW004-1	SJSW004-2	SJSW004-3	
Analyte	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L	pg/L	
2,3,7,8-tetrachlorodibenzo-p -dioxin	0.0668	0.156	0.180	0.0434	0.174	0.118	0.298	0.349	0.511	0.183	0.226	0.177	
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.0117	0.0180	0.0185	0.0105	0.0205	0.0125	0.0157	0.0285	0.0236	0.0195	0.0220	0.0255	
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.0169	0.0295	0.0435	0.0114	0.0280	0.0355	0.0330	0.0650	0.0465	0.0370	0.0815	0.0385	
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.0679	0.0990	0.0871	0.0385	0.0915	0.0745	0.0706	0.246	0.0946	0.0770	0.133	0.0365	
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.0973	0.208	0.110	0.0368	0.175	0.0615	0.137	0.189	0.133	0.131	0.218	0.0970	
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	2.74	4.00	3.14	1.84	3.15	2.59	3.69	4.23	3.46	3.71	5.23	2.29	
Octachlorodibenzo-p-dioxin	116	175	123	68.9	143	90.9	123	111	131	121	102	86.5	
2,3,7,8-tetrachlorodibenzofuran	0.252	0.359	0.534	0.176	0.442	0.277	1.18	0.887	1.44	0.573	0.563	2.37	
1,2,3,7,8-pentachlorodibenzofuran	0.0210	0.0170	0.0295	0.0179	0.0240	0.0230	0.0528	0.0495	0.0626	0.0385	0.0355	0.0320	
2,3,4,7,8-pentachlorodibenzofuran	0.0190	0.0175	0.0275	0.0104	0.0245	0.0230	0.0399	0.0495	0.0472	0.0155	0.0355	0.0310	
1,2,3,4,7,8-hexachlorodibenzofuran	0.0285	0.0499	0.0511	0.0325	0.0605	0.0405	0.176	0.0786	0.119	0.0860	0.0544	0.0496	
1,2,3,6,7,8-hexachlorodibenzofuran	0.0240	0.0161	0.0282	0.015 <sup>a</sup>	0.0370	0.0223	0.0454	0.0366	0.0450	0.0199	0.0381	0.0255	
1,2,3,7,8,9-hexachlorodibenzofuran	0.0185	0.0210	0.0135	0.121	0.0195	0.0135	0.0195	0.0225	0.0115	0.0150	0.0120	0.0135	
2,3,4,6,7,8-hexachlorodibenzofuran	0.0165	0.0195	0.0255	0.0325	0.0185	0.0125	0.0240	0.0295	0.0260	0.0350	0.0325	0.0215	
1,2,3,4,6,7,8-heptachlorodibenzofuran	0.197	0.260	0.263	0.137	0.274	0.201	0.355	0.278	0.326	0.360	0.335	0.238	
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.0400	0.0250	0.0340	0.0388	0.0355	0.0305	0.0840	0.0565	0.0405	0.0587	0.0690	0.0315	
Octachlorodibenzofuran	1.09	1.81	2.51	0.803	2.27	1.79	2.70	2.50	3.41	3.06	2.63	2.69	
TEQ <sub>DF,M</sub> (ND=0)	0.169	0.322	0.338	0.0893 <sup>a</sup>	0.308	0.207	0.527	0.572	0.781	0.349	0.422	0.474	
TEQ <sub>DF,M</sub> (ND=½DL)	0.202	0.356	0.369	0.106 <sup>a</sup>	0.367	0.247	0.576	0.634	0.832	0.384	0.460	0.530	
TEQ <sub>DF,M</sub> (ND=DL)	0.236	0.389	0.400	0.123 <sup>a</sup>	0.427	0.287	0.626	0.696	0.884	0.418	0.498	0.587	

 Table 6-1

 Concentration of Dioxins and Furans in Each 2016 Surface Water Sample

Field duplicates were averaged for this analysis.

DL = detection limit

ND = non-detect

TEQ<sub>DF,M</sub> = TEQ calculated using Van den Berg et al. (2006) toxicity equivalency factors for mammals

<sup>a</sup> The dissolved result was rejected during validation, so value represents the suspended fraction only.

TMDL Station ID		11261			11264		
2016 Station ID	SJSW005-1	SJSW005-2	SJSW005-3	SJSW006-1	SJSW006-2	SJSW006-3	SJSW007-1
Analyte	pg/L						
2,3,7,8-tetrachlorodibenzo- <i>p</i> -dioxin	0.152	0.139	0.147	0.184	0.157	0.151	0.262
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.0241	0.0135	0.0213	0.0235	0.0135	0.0216	0.0162
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.0457	0.0315	0.0435	0.0205	0.0510	0.0391	0.0590
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.0825	0.0624	0.0810	0.0795	0.0895	0.0806	0.118
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.143	0.0930	0.104	0.119	0.151	0.0913	0.204
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	3.74	2.08	2.40	3.27	3.22	2.15	5.62
Octachlorodibenzo-p-dioxin	112	67.3	77.6	94.4	72.7	75.3	174
2,3,7,8-tetrachlorodibenzofuran	0.481	0.422	0.498	0.682	0.575	0.498	0.706
1,2,3,7,8-pentachlorodibenzofuran	0.0380	0.0304	0.0371	0.0425	0.0335	0.0381	0.0270
2,3,4,7,8-pentachlorodibenzofuran	0.0300	0.0305	0.0390	0.0425	0.0365	0.0378	0.0449
1,2,3,4,7,8-hexachlorodibenzofuran	0.0970	0.0526	0.0694	0.122	0.0872	0.0774	0.131
1,2,3,6,7,8-hexachlorodibenzofuran	0.0455	0.0294	0.0362	0.047	0.0376	0.0381	0.0250
1,2,3,7,8,9-hexachlorodibenzofuran	0.0145	0.0135	0.0145	0.0145	0.0145	0.0145	0.0295
2,3,4,6,7,8-hexachlorodibenzofuran	0.0380	0.0125	0.0260	0.0175	0.0290	0.0310	0.0985
1,2,3,4,6,7,8-heptachlorodibenzofuran	0.465	0.288	0.392	0.562	0.410	0.389	0.581
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.0750	0.0385	0.0400	0.0860	0.0920	0.0485	0.126
Octachlorodibenzofuran	3.89	3.33	3.91	5.37	4.11	4.24	3.89
TEQ <sub>DF,M</sub> (ND=0)	0.322	0.228	0.272	0.354	0.296	0.271	0.497
TEQ <sub>DF,M</sub> (ND=½DL)	0.358	0.279	0.320	0.401	0.346	0.322	0.546
TEQ <sub>DF,M</sub> (ND=DL)	0.396	0.330	0.370	0.447	0.396	0.373	0.596

 Table 6-1

 Concentration of Dioxins and Furans in Each 2016 Surface Water Sample

SJSW007	
SJSW007-2	SJSW007-3
pg/L	pg/L
0.303	0.248
0.0310	0.0200
0.0775	0.0415
0.317	0.0845
0.224	0.126
5.14	2.70
136	103
0.694	0.634
0.0455	0.0325
0.0505	0.0331
0.0868	0.0565
0.0431	0.0313
0.0220	0.0115
0.0385	0.0240
0.408	0.302
0.0710	0.0355
3.45	3.21
0.561	0.400
0.599	0.442
0.637	0.483

September 2016

 Table 6-2

 Comparison of Average Surface Water TEQ Concentrations 2002–2016

			2002		2003		2004		2009		2016	
TMDL Station ID	2016 Station ID	N	Average TEQ <sub>DF,M</sub> (pg/L)	Percent Change in TEQ <sub>DF,M</sub> Concentration								
11197	SJSW001					2	0.187			3	0.309	65
TCEQ2009_03 <sup>a</sup>	SJSW003							2	8.61	3	0.681	-92
11193	SJSW004	2	1.61	1	3.15	4	1.42			3	0.458	-85
11261	SJSW005	2	0.418	1	0.584	2	0.802			3	0.319	-60
11264	SJSW006	1	0.519	1	0.462	2	0.674			3	0.356	-47

Field duplicates were averaged for this analysis.

TEQ<sub>DF,M</sub> = TEQ calculated using Van den Berg et al. (2006) toxicity equivalency factors for mammals

TEQ calculated with non-detects set to  $\frac{1}{2}$  the detection limit.

Percent change calculated as follows: [ (2016 concentration - maximum past concentration) / (maximum past concentration)] \* 100

A positive result represents a percentage increase; a negative result represents a percentage decrease.

TEQ = toxicity equivalent

TMDL = total maximum daily load

<sup>a</sup> Includes results from Location TCEQ2009\_01 (sample Point#1&2), which was collected in close proximity.

#### Table 6-3

			2002		2003		2004		2009		2016	
TMDL Station ID	2016 Station ID	N	Average TCDD (pg/L)	Percent Change in TCDD Concentration								
11197	SJSW001					2	0.0653			3	0.134	106
TCEQ2009_03 <sup>a</sup>	SJSW003							2	4.58	3	0.386	-92
11193	SJSW004	2	1.11	1	2.16	4	0.929			3	0.195	-91
11261	SJSW005	2	0.214	1	0.328	2	0.488			3	0.146	-70
11264	SJSW006	1	0.270	1	0.241	2	0.395			3	0.164	-59

### Comparison of Average Surface Water TCDD Concentrations 2002–2016

#### Notes

Field duplicates were averaged for this analysis.

TCDD = 2,3,7,8-tetrachlorodibenzo-p -dioxin

Percent change calculated as follows: [(2016 concentration - maximum past concentration)/(maximum past concentration)]\*100

A positive result represents a percentage increase; a negative result represents a percentage decrease.

TMDL = total maximum daily load

<sup>a</sup> Includes results from Location TCEQ2009\_01 (sample Point#1&2), which was collected in close proximity.

Transect:	SJT	TR2	SJTTR3	SJT	TR4	SJT	TR5
Sample ID:	SJTTR2-F3	SJTTR2-F4	SJTTR3-F3	SJTTR4-F3	SJTTR4-F4	SJTTR5-F3	SJTTR5-F4
Analyte	ng/kg ww						
2,3,7,8-tetrachlorodibenzo-p -dioxin	1.16	0.95	1.21	1.56	1.32	1.15	2.48
1,2,3,7,8-pentachlorodibenzo-p-dioxin	0.0705	0.0675	0.062	0.217	0.06	0.161	9.47
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin	0.106	0.092	0.066	0.0326	0.035	0.0208	4.95
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin	0.136	0.252	0.069	0.087	0.193	0.0645	12.2
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin	0.101	0.0895	0.0635	0.033	0.0317	0.0192	2.32
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin	2.55	2.61	2.09	1.04	0.436	1.19	39.1
Octachlorodibenzo-p-dioxin	13.2	17	10.6	8.24	9.02	8.79	54.6
2,3,7,8-tetrachlorodibenzofuran	1.81	1.1	2	0.99	0.468	0.515	0.605
1,2,3,7,8-pentachlorodibenzofuran	0.0466	0.052	0.0336	0.0535	0.068	0.058	0.151
2,3,4,7,8-pentachlorodibenzofuran	0.0525	0.0535	0.14	0.053	0.068	0.0585	0.152
1,2,3,4,7,8-hexachlorodibenzofuran	0.0625	0.0615	0.055	0.0505	0.0515	0.0495	1.62
1,2,3,6,7,8-hexachlorodibenzofuran	0.057	0.042	0.051	0.0423	0.0468	0.0439	0.286
1,2,3,7,8,9-hexachlorodibenzofuran	0.088	0.06	0.073	0.0705	0.072	0.0695	0.144
2,3,4,6,7,8-hexachlorodibenzofuran	0.062	0.0471	0.0565	0.0476	0.0505	0.0474	0.09
1,2,3,4,6,7,8-heptachlorodibenzofuran	0.184	0.59	0.376	0.0695	0.034	0.0498	2.19
1,2,3,4,7,8,9-heptachlorodibenzofuran	0.0925	0.0775	0.0715	0.066	0.0376	0.0245	0.346
Octachlorodibenzofuran	1.38	1.72	0.535	0.134	0.126	0.545	2.14
TEQ <sub>DF,M</sub> (ND=0)	1.37	1.12	1.48	1.89	1.34	1.33	14.5
TEQ <sub>DF,M</sub> (ND=½DL)	1.52	1.25	1.59	1.94	1.51	1.43	14.7
TEQ <sub>DF,M</sub> (ND=DL)	1.67	1.37	1.69	2.00	1.67	1.53	14.8

Table 7-12016 Dioxin and Furan Concentrations and TEQ $P_{DF,M}$  in Gulf Killifish from Transects 2 Through 5

DL = detection limit

ND = non-detect

TEQ<sub>DF,M</sub> = TEQ calculated using Van den Berg et al. (2006) toxicity equivalency factors for mammals

ww = wet weight

Silversides from	Sample ID:	SJTTR3-F4
Analyte	Sumple ID.	ng/kg ww
2,3,7,8-tetrachlorodibenzo- <i>p</i> -dioxin		4.28
1,2,3,7,8-pentachlorodibenzo-p-dioxin		0.11
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin		0.123
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin		0.198
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin		0.118
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin		4.51
Octachlorodibenzo-p-dioxin		16.5
2,3,7,8-tetrachlorodibenzofuran		13
1,2,3,7,8-pentachlorodibenzofuran		0.0655
2,3,4,7,8-pentachlorodibenzofuran		0.122
1,2,3,4,7,8-hexachlorodibenzofuran		0.124
1,2,3,6,7,8-hexachlorodibenzofuran		0.112
1,2,3,7,8,9-hexachlorodibenzofuran		0.161
2,3,4,6,7,8-hexachlorodibenzofuran		0.123
1,2,3,4,6,7,8-heptachlorodibenzofuran		0.345
1,2,3,4,7,8,9-heptachlorodibenzofuran		0.18
Octachlorodibenzofuran		2.94
TEQ <sub>DF,M</sub> (ND=0)		5.63
TEQ <sub>DF,M</sub> (ND=1/2DL)		5.88
TEQ <sub>DF,M</sub> (ND=DL)		6.13

Table 7-22016 Dioxin and Furan Congener Concentrations and TEQSilversides from Transect 3

DL = detection limit

ND = non-detect

 $TEQ_{DF,M}$  = TEQ calculated using Van den Berg et al. (2006) toxicity equivalency factors for mammals ww = wet weight

# APPENDIX A USEPA COMMUNICATIONS DIRECTING RESPONDENTS TO CONDUCT STUDIES OF THE TCRA ARMORED CAP

From:	Miller, Garyg
To:	David Keith
Cc:	Foster, Anne; Sanchez, Carlos; Walters, Donn; Werner, Robert
Subject:	San Jacinto River Waste Pits Sampling
Date:	Thursday, August 06, 2015 6:02:56 AM
Attachments:	SJRWP Sampling Approach 8-6-2015.docx

David,

The purpose of this email is to notify you that the Environmental Protection Agency (EPA) has determined that additional sampling is required for the Remedial Investigation and Feasibility Study (RI/FS) being conducted by Respondents under the Unilateral Administrative Order (UAO), CERCLA Docket No. 06-03-10, for the San Jacinto River Waste Pits Superfund Site (Site). In keeping with Paragraph 53 of the UAO, the purpose of the sampling is to determine the nature and extent of contamination, including whether contamination from the waste pits has spread to the surrounding areas. Additional detail on the purpose of the sampling is contained in the attached sampling approach. In accordance with Paragraphs 54 and 71 of the above referenced UAO, the EPA is directing Respondents to revise the Final RI/FS Work Plan, dated November 2010, or prepare an addendum to the Work Plan, to provide the necessary plans and schedule to accomplish the sampling described in the attached sampling approach. The revised RI/FS Work Plan or Work Plan addendum shall be submitted to EPA in accordance with the UAO within thirty calendar days of receipt of this email.

Please let me know if you have any questions on this.

Regards,

Gary Miller EPA Remedial Project Manager 214-665-8318 <u>miller.garyg@epa.gov</u>

## San Jacinto River Waste Pits Superfund Site

## Purpose of Sampling:

- 1. Confirm that the cap continues to prevent dioxin/furan migration from the waste pits to the San Jacinto River following storms occurring since the last cap passive pore-water sampling event in 2012.
- 2. Determine whether there has been any migration of dioxin/furan contaminated sediment from under the toe of the cap using sediment samples.
- 3. Determine whether there is any migration of dioxin/furan from the alluvial aquifer at the waste pits or the Southern Impoundment into the San Jacinto River at levels above the Texas Surface Water Quality Standard (SWQS) for dioxin/furan using passive pore-water samplers and surface water samples.
- 4. Determine whether there is any migration of groundwater in the Southern Impoundment to the surface water in the Old River channel at levels above the SWQS using groundwater samples.

## Sampling Approach:

- 1. Surface water samples: minimum 2 samples each in the San Jacinto River at an upstream location (Banana Bend), at a location directly over the waste pits cap, and at a location immediately downstream of the northern waste pits (near I-10 Bridge) (minimum 6 samples total); and a minimum 2 samples in the Old River spread across the north/south length of the Southern Impoundment. Sampling to be conducted at a minimum of three times on a weekly basis at each sampling location. Use sampling/analysis methods for the 17 dioxin/furan congeners capable of achieving a method quantitation limit that is less than the Texas Surface Water Quality Standard for dioxin/furan.
- 2. Surface sediment (0 12 inches): minimum 8 samples within 3-feet of tow of cap, with a bias for locations adjacent to the higher sediment dioxin concentrations under the outer edges of the cap, spread across the underwater perimeter of the cap. Sediment samples to be analyzed for the 17 dioxin/furan congeners and organic carbon with detection limits consistent with previous sediment sampling events.
- 3. Passive samplers (cap): minimum 10 locations in the underwater portions of cap with 2 samples (top and bottom of armor material) at each location; select locations with a bias toward the northwest area & areas of higher dioxin concentrations under the cap. Include appropriate reference compound samplers so that equalization status can be determined. Samples to be analyzed for the 17 dioxin/furan congeners. Also include surface water passive samplers at a minimum of three of the locations just above the cap distributed across the cap.
- 4. Passive samplers (un-capped sediment northern pits area): minimum 18 locations at a depth of 0 12 inches around the perimeter of the cap in the sediment at random distances from just beyond (several feet) the toe of the cap, but no further away than 30-feet from the toe of the cap. Include an additional 2 pore-water samplers at the northwest and southeast outlets of the drainage ditch located on the south side of the cap. Samples to be analyzed for the 17 dioxin/furan congeners. Also include surface water passive

samplers at three of the locations just above the sediment distributed at locations across the perimeter of the cap.

- 5. Passive samplers (sediment southern impoundment area): minimum 10 underwater locations along the Old River shoreline spread across the north/south length of the Southern Impoundment, with a bias for locations across and down gradient form the highest monitoring well groundwater concentrations. The pore-water samplers should be placed at random distances from the shoreline ranging from 10 feet away but no further away than 30-feet from the shoreline. Samples to be analyzed for the 17 dioxin/furan congeners. Sampler locations should also be field adjusted if any barges are moored in the planned locations.
- 6. Groundwater samples: collect groundwater samples from each of the existing Southern Impoundment area monitoring wells, both shallow and deep, and perform analysis for the 17 dioxin/furan congeners as well as all other chemicals previously detected in any south area monitoring well. Also install and sample 1 new shallow groundwater monitoring well (depth consistent with previous shallow monitoring wells) located as near the shoreline a practical and located down-gradient from existing monitoring well SJMW001 and soil boring SJSB012.
- 7. The EPA dive team will assist with placement and retrieval of the passive samplers.

# APPENDIX B VALIDATION REPORTS FOR 2016 STUDIES

# **1** INTRODUCTION

Chemical data collected according to sampling and analysis plans (SAPs) for the San Jacinto River Waste Pits Superfund Site remedial investigation and feasibility study require validation according to the specifications within their respective SAPs. This appendix contains the data validation reports for the following sample collection efforts undertaken in 2016:

- Surface sediment sampling
- Porewater of the TCRA armored cap and groundwater beneath the impoundments north of I-10 and south of I-10 sampled with solid phase microextraction (SPME) devices
- High-volume surface water sampling
- Tissue of Gulf killifish (*Fundulus grandis*)

The enclosed collections of validation reports make up the complete data validation record for the chemistry data generated as a result of sampling conducted in 2016 for the San Jacinto River Waste Pits remedial investigation.



# DATA VALIDATION REPORT

# SAN JACINTO RIVER WASTE PITS 2016 SEDIMENT SAMPLING

#### **Prepared for:**

Integral Consulting, Inc. 1205 West Bay Dr. NW Olympia, Washington 98502

#### **Prepared by:**

EcoChem, Inc. 1011 Western Avenue, Suite 1006 Seattle, Washington 98104

EcoChem Project: C22130-21

June 28, 2016

**Approved for Release:** 

Melissa Swanson Project Chemist EcoChem, Inc.

# **PROJECT NARRATIVE**

#### **Basis for Data Validation**

This report summarizes the results of full validation (EPA Stage 3/4) performed on sediment and summary validation (EPA Stage 2A) performed on quality control sample data for the San Jacinto River 2016 Sediment Sampling Study. A complete list of samples is provided in the **Sample Index**.

Samples were analyzed by ALS Environmental, Kelso, Washington and ALS Environmental, Houston, Texas. The analytical methods and EcoChem project chemists are listed below.

Analysis	Method	PRIMARY REVIEW	SECONDARY REVIEW	
Dioxin/Furan Compounds	1613B	M. Swanson	C. Ransom	
Total Organic Carbon	D4129-05ALS		M. Swanson	
Grainsize	PSEP	E. Clayton		

The data were reviewed using guidance and quality control criteria documented in the analytical methods and the following project and guidance documents:

- Sampling and Analysis Plan: Sediment Study San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, April 2010).
- Addendum 3 (to the Sediment Sampling and Analysis Plan) Additional Sediment Sampling within the USEPA's Preliminary Site Perimeter, San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, March 2016).
- USEPA National Functional Guidelines for Organic Data Review (USEPA 2008).
- USEPA National Functional Guidelines for Inorganic Data Review (USEPA October 2004).

EcoChem's goal in assigning data assessment qualifiers is to assist in proper data interpretation. If values are estimated (J or UJ), data may be used for site evaluation and risk assessment purposes but reasons for data qualification should be taken into consideration when interpreting sample concentrations. If values are assigned an R, the data are to be rejected and should not be used for any site evaluation purposes. If values have no data qualifier assigned, then the data meet the data quality objectives as stated in the documents and methods referenced above.

Data qualifier definitions, reason codes, and validation criteria are included as **APPENDIX A**. A Qualified Data Summary Table is included in **APPENDIX B**. Data Validation Worksheets and project associated communications will be kept on file at EcoChem, Inc. A qualified laboratory electronic data deliverable (EDD) is also submitted with this report.

# Sample Index

San lacinto	River Waste	Dite TCRA	2016 Sediment
Jan Jacinio			ZUIO Seument

San Jacinto River Waste Pits TCRA 2016 Sediment           Dioxin         Conventionals									
	Sample ID		Dioxins		Conventionals				
	•		,		,				
					<ul> <li>✓</li> </ul>				
					✓				
K1604875	SD0129			K1604875-003	$\checkmark$				
K1604875	SD0113	E1600405-004		K1604875-004	$\checkmark$				
K1604875	SD0112	E1600405-005		K1604875-005	$\checkmark$				
K1604875	SD0127	E1600405-006		K1604875-006	$\checkmark$				
K1604875	SD0126	E1600405-007	$\checkmark$	K1604875-007	$\checkmark$				
K1604875	SD0109	E1600405-008	$\checkmark$	K1604875-008	$\checkmark$				
K1604875	SD0125	E1600405-009	$\checkmark$	K1604875-009	$\checkmark$				
K1604875	SD0124	E1600405-010	$\checkmark$	K1604875-010	$\checkmark$				
K1604875	SD0106	E1600405-011	$\checkmark$	K1604875-011	$\checkmark$				
K1604875	SD0107	E1600405-012	$\checkmark$	K1604875-012	$\checkmark$				
K1604875	SD0108	E1600405-013	$\checkmark$	K1604875-013	$\checkmark$				
K1604875	SD0123	E1600405-014	$\checkmark$	K1604875-014	$\checkmark$				
K1604875	SD0122	E1600405-015	$\checkmark$	K1604875-015	$\checkmark$				
K1604875	SD0105	E1600405-016	$\checkmark$	K1604875-016	$\checkmark$				
K1604875	SD0104	E1600405-017	$\checkmark$	K1604875-017	$\checkmark$				
K1604875	SD0103	E1600405-018	$\checkmark$	K1604875-018	$\checkmark$				
K1604875	SD0115	E1600405-019	$\checkmark$	K1604875-019	$\checkmark$				
K1604875	SD0120	E1600405-020	$\checkmark$	K1604875-020	$\checkmark$				
K1604875	SD0121	E1600405-021	$\checkmark$	K1604875-021	$\checkmark$				
K1604875	SD0102	E1600405-022	$\checkmark$	K1604875-022	$\checkmark$				
K1604875	SD0101	E1600405-023	$\checkmark$	K1604875-023	$\checkmark$				
	FW0100	E1600405-024	$\checkmark$						
K1604875	SD0100	E1600405-025	$\checkmark$	K1604875-024	$\checkmark$				
K1604875	SD0119	E1600405-026	$\checkmark$	K1604875-025	$\checkmark$				
K1604875	SD0118	E1600405-027	$\checkmark$	K1604875-026	$\checkmark$				
K1604875	SD0117	E1600405-028	$\checkmark$	K1604875-027	$\checkmark$				
	FW0012	E1600406-001	$\checkmark$						
	FB0013	E1600406-002	$\checkmark$						
		E1600406-003	$\checkmark$						
			$\checkmark$						
	Conventionals SDG SDG K1604875	Conventionals SDG         Sample ID           K1604875         SD0114           K1604875         SD0128           K1604875         SD0129           K1604875         SD0129           K1604875         SD0129           K1604875         SD0121           K1604875         SD0127           K1604875         SD0126           K1604875         SD0125           K1604875         SD0107           K1604875         SD0107           K1604875         SD0103           K1604875         SD0123           K1604875         SD0103           K1604875         SD0103           K1604875         SD0120           K1604875         SD0120           K1604875         SD0102           K1604875         SD0102           K1604875         SD0102           K1604875         SD0100           K1604875 </td <td>Conventionals SDG         Sample ID         Dioxin Laboratory ID           K1604875         SD0114         E1600405-001           K1604875         SD0128         E1600405-002           K1604875         SD0129         E1600405-003           K1604875         SD0112         E1600405-004           K1604875         SD0112         E1600405-004           K1604875         SD0112         E1600405-005           K1604875         SD0127         E1600405-006           K1604875         SD0126         E1600405-007           K1604875         SD0126         E1600405-008           K1604875         SD0125         E1600405-009           K1604875         SD0124         E1600405-010           K1604875         SD0102         E1600405-012           K1604875         SD0107         E1600405-013           K1604875         SD0108         E1600405-013           K1604875         SD0123         E1600405-014           K1604875         SD0104         E1600405-017           K1604875         SD0103         E1600405-017           K1604875         SD0103         E1600405-018           K1604875         SD0104         E1600405-020           K1604875         &lt;</td> <td>Conventionals SDG         Sample ID         Dioxin Laboratory ID         Dioxins           K1604875         SD0114         E1600405-001         ✓           K1604875         SD0128         E1600405-002         ✓           K1604875         SD0129         E1600405-003         ✓           K1604875         SD0112         E1600405-004         ✓           K1604875         SD0112         E1600405-006         ✓           K1604875         SD0127         E1600405-007         ✓           K1604875         SD0126         E1600405-008         ✓           K1604875         SD0126         E1600405-009         ✓           K1604875         SD0125         E1600405-009         ✓           K1604875         SD0124         E1600405-010         ✓           K1604875         SD0106         E1600405-011         ✓           K1604875         SD0107         E1600405-013         ✓           K1604875         SD0108         E1600405-014         ✓           K1604875         SD0122         E1600405-015         ✓           K1604875         SD0103         E1600405-016         ✓           K1604875         SD0103         E1600405-018         ✓</td> <td>Conventionals SDG         Sample ID         Dioxin Laboratory ID         Dioxins         Conventionals Laboratory ID           K1604875         SD0114         E1600405-001         ✓         K1604875-001           K1604875         SD0128         E1600405-002         ✓         K1604875-002           K1604875         SD0129         E1600405-003         ✓         K1604875-004           K1604875         SD0113         E1600405-004         ✓         K1604875-004           K1604875         SD0127         E1600405-006         ✓         K1604875-005           K1604875         SD0127         E1600405-007         ✓         K1604875-006           K1604875         SD0126         E1600405-008         ✓         K1604875-008           K1604875         SD0125         E1600405-009         ✓         K1604875-009           K1604875         SD0124         E1600405-010         ✓         K1604875-010           K1604875         SD0106         E1600405-011         ✓         K1604875-012           K1604875         SD0107         E1600405-011         ✓         K1604875-012           K1604875         SD0102         E1600405-013         ✓         K1604875-013           K1604875         SD0102         E1600405</td>	Conventionals SDG         Sample ID         Dioxin Laboratory ID           K1604875         SD0114         E1600405-001           K1604875         SD0128         E1600405-002           K1604875         SD0129         E1600405-003           K1604875         SD0112         E1600405-004           K1604875         SD0112         E1600405-004           K1604875         SD0112         E1600405-005           K1604875         SD0127         E1600405-006           K1604875         SD0126         E1600405-007           K1604875         SD0126         E1600405-008           K1604875         SD0125         E1600405-009           K1604875         SD0124         E1600405-010           K1604875         SD0102         E1600405-012           K1604875         SD0107         E1600405-013           K1604875         SD0108         E1600405-013           K1604875         SD0123         E1600405-014           K1604875         SD0104         E1600405-017           K1604875         SD0103         E1600405-017           K1604875         SD0103         E1600405-018           K1604875         SD0104         E1600405-020           K1604875         <	Conventionals SDG         Sample ID         Dioxin Laboratory ID         Dioxins           K1604875         SD0114         E1600405-001         ✓           K1604875         SD0128         E1600405-002         ✓           K1604875         SD0129         E1600405-003         ✓           K1604875         SD0112         E1600405-004         ✓           K1604875         SD0112         E1600405-006         ✓           K1604875         SD0127         E1600405-007         ✓           K1604875         SD0126         E1600405-008         ✓           K1604875         SD0126         E1600405-009         ✓           K1604875         SD0125         E1600405-009         ✓           K1604875         SD0124         E1600405-010         ✓           K1604875         SD0106         E1600405-011         ✓           K1604875         SD0107         E1600405-013         ✓           K1604875         SD0108         E1600405-014         ✓           K1604875         SD0122         E1600405-015         ✓           K1604875         SD0103         E1600405-016         ✓           K1604875         SD0103         E1600405-018         ✓	Conventionals SDG         Sample ID         Dioxin Laboratory ID         Dioxins         Conventionals Laboratory ID           K1604875         SD0114         E1600405-001         ✓         K1604875-001           K1604875         SD0128         E1600405-002         ✓         K1604875-002           K1604875         SD0129         E1600405-003         ✓         K1604875-004           K1604875         SD0113         E1600405-004         ✓         K1604875-004           K1604875         SD0127         E1600405-006         ✓         K1604875-005           K1604875         SD0127         E1600405-007         ✓         K1604875-006           K1604875         SD0126         E1600405-008         ✓         K1604875-008           K1604875         SD0125         E1600405-009         ✓         K1604875-009           K1604875         SD0124         E1600405-010         ✓         K1604875-010           K1604875         SD0106         E1600405-011         ✓         K1604875-012           K1604875         SD0107         E1600405-011         ✓         K1604875-012           K1604875         SD0102         E1600405-013         ✓         K1604875-013           K1604875         SD0102         E1600405				

# DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 Sediment Sampling Dioxin/Furan Compounds by EPA 1613B

This report documents the review of analytical data from the analyses of sediment samples and the associated laboratory quality control (QC) samples. ALS Environmental, Houston, Texas, analyzed the samples. Refer to the **Sample Index** for a complete list of samples.

SDG	Number of Samples	VALIDATION LEVEL		
E1600405	27 Sediment 1 Wipe	EPA Stage 4 EPA Stage 2A		
E1600406	4 Wipe	EPA Stage 2A		

#### DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

#### EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package. No errors were noted.

#### **TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

1	Sample Receipt, Preservation, and Holding Times	$\checkmark$	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)
$\checkmark$	System Performance and Resolution Checks	1	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
$\checkmark$	Initial Calibration (ICAL)	1	Field Replicate Samples
$\checkmark$	Calibration Verification	>	Target Analyte List
2	Laboratory Blanks	$\checkmark$	Reported Results
1	Field Blanks	2	Compound Identification
$\checkmark$	Labeled Compound Recovery	1	Calculation Verification

✓ Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

#### Sample Receipt, Preservation, and Holding Times

As stated in validation guidance documents, sample coolers should arrive at the laboratory within the advisory temperature range of 2°C to 6°C. Several cooler temperatures were greater than the upper control limit, the highest at 11.5°C. Dioxin compounds have been found to remain stable at a

wide range of temperatures. These temperature outliers did not impact data quality; therefore, no data were qualified.

#### Laboratory Blanks

To assess the impact of any blank contaminant on the reported sample results, an action level was established at five times (5x) the concentration reported in the blank. If a contaminant was reported in an associated field sample and the concentration was less than the action level, the result was qualified as not detected (U-7). No action was taken if the sample result was greater than the action level or for non-detected results. The laboratory assigned K-flags to values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are "estimated maximum possible concentrations". When these occurred in the method blank the results were considered as false positives. No action levels were established for these analytes.

Method blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method blanks, however only the results noted below required qualification; all other associated sample results were either not detected or were detected at concentrations greater than the action levels.

*SDG E1600405:* The results for 2,3,4,6,7,8-HxCDF in Samples SD0117, SD0124, and SD0125 were qualified as not detected (U-7).

#### Field Blanks

The field blanks for this project are filter wipe samples. To evaluate the effect of field blank contamination on the sample data, action levels of 5x the blank concentrations were established. For the purposes of evaluation, the reported filter wipe values (in total pg) were converted to the same units as the field samples (ng/kg) using the average sample mass and volume collected. If a contaminant was detected in an associated field sample and the concentration was less than the action level, the result was qualified (U-6) at the reported concentration. No action was taken if the sample result was greater than the action level, or for non-detected results.

*SDG E1600405*: Wipe sample FW0100 was reported in this SDG. No target analytes were detected in this blank.

*SDG E1600406:* Wipe samples FW0100 and FB0100 associated with the sediment samples were submitted in this SDG. There was a positive result for 1,2,3,4,6,7,8-HpCDF in Sample FB0100. All associated sample results were either not detected or were detected at concentrations greater than the action level. No data were qualified based on field blank contamination.

Wipe samples FW0012 and FB0013 were also submitted in this SDG, but are associated with tissue samples that will be discussed in a separate report.

#### Matrix Spike/Matrix Spike Duplicate

Matrix spike/matrix spike duplicate (MS/MSD) samples are not required by the method and were not analyzed. Accuracy and precision were evaluated using the labeled compound and laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results.

#### Field Replicates

The following acceptance criteria were used to evaluate precision: the relative percent difference (RPD) control limit is 50% for results greater than 5x the reporting limit (RL). For results less than 5x the RL, the difference between the sample and replicate must be less than twice the RL. No data were qualified based on field replicate precision outliers. Data users should consider the impact of field precision outliers on the reported results. With the exceptions noted below, field precision was acceptable.

*SDG E1600405:* Two pair of field replicates were submitted with this SDG: SD0120 & SD0121 and SD0128 & SD0129. For Samples SD0120 & SD0121 the difference values for Total HpCDF and OCDF were greater than the control limit.

#### **Compound Identification**

The laboratory assigned K-flags to results where a peak was detected but did not meet ion ratio quantitation criteria. The reported values cannot be considered as positive identifications for these analytes. These results were considered potential false positives or estimated maximum possible concentrations (EMPC) and were qualified as not detected (U-25) at the reported values.

The method requires the confirmation of 2,3,7,8-TCDF using an alternate GC column as the DB5 column that is typically used cannot fully separate 2,3,7,8-TCDF from closely eluting non-target TCDF isomers. The laboratory did not perform a second column confirmation; however, the laboratory uses a DB-5SMUI column. This column provides adequate resolution of the TCDF isomers as indicated by the acceptable peak to valley ratios. Since the 2,3,7,8-TCDF resolution was acceptable, no action was necessary.

*SDG E1600405:* Results were assigned P-flags by the laboratory to indicate the presence of diphenyl ether interference. The results for 1,2,3,4,6,7,8-HpCDF in Samples SD0106, SD0107, SD0108, SD0109, SD0123, and SD0124 were estimated (J-23H) to indicate a potential high bias. In addition to the P-flag the result for 1,2,3,4,6,7,8-HpCDF in Sample SD0125 was also K-flagged by the laboratory, this result was estimated (UJ-23,25) to indicate an elevated reporting limit.

#### **Calculation Verification**

*SDG E1600405*: Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

#### OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable as demonstrated by labeled compound and LCS/LCSD %R values. With the exceptions noted above, precision was acceptable as demonstrated by the LCS/LCSD and field replicate RPD values.

Detection limits were elevated due to ion ratio outliers and method blank contamination. Data were estimated due to diphenyl ether interferences.

All data, as qualified, are acceptable for use.

# DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 Sediment Sampling Conventional Parameters

This report documents the review of analytical data from the analyses of sediment samples and the associated laboratory quality control (QC) samples. ALS Environmental, Kelso, Washington, analyzed the samples. Refer to the **Sample Index** for a complete list of samples.

SDG	Number of Samples	VALIDATION LEVEL
K1604875	27 Sediment	EPA Stage 3

The analytical tests that were performed are summarized below.

Parameter	Method
Grain Size	PSEP
Total Organic Carbon	ASTM D4129-05M
Total Solids	160.3M

#### DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

#### EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package. No errors were noted.

#### **TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below:

1	Sample Receipt, Preservation, and Holding Times	$\checkmark$	Matrix Spikes (MS)
$\checkmark$	Initial Calibration	>	Laboratory Replicates
$\checkmark$	Calibration Verification	1	Field Replicates
$\checkmark$	Laboratory Blanks	>	Reporting Limits
1	Field Blanks	$\checkmark$	Reported Results
$\checkmark$	Laboratory Control Samples (LCS)	1	Calculation Verification

 $\checkmark$  Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

#### Sample Receipt, Preservation, and Holding Times

As stated in validation guidance documents, sample coolers should arrive at the laboratory within the advisory temperature range of 2°C to 6°C. The cooler temperatures upon receipt at the laboratory were less than the lower control limit, the lowest at 1.0°C. These temperature outliers did not impact data quality; therefore, no data were qualified.

One sample jar each for Samples SD0124 and SD0106 arrived at the laboratory broken. The contents were transferred to new containers. Multiple jars arrived for each of these samples, no further action was taken.

#### Field Blanks

No field blanks were submitted.

#### **Field Replicates**

The following acceptance criteria were used to evaluate precision: the relative percent difference (RPD) value control limit is 50% for results greater than 5x the reporting limit (RL). The difference between the sample and replicate must be less than twice the RL for results less than 5x the RL.

Two pair of field replicates were submitted with this SDG, Samples SD0120 & SD0121 and SD0128 & SD0129. Field precision was acceptable.

#### **Calculation Verification**

Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

#### **OVERALL ASSESSMENT**

As determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable as demonstrated by the laboratory control sample percent recovery values. Precision was acceptable as demonstrated by the laboratory and field duplicate RPD values.

No data were qualified for any reason.

All data, as reported, are acceptable for use.



# **APPENDIX A**

# DATA QUALIFIER DEFINITIONS REASON CODES AND CRITERIA TABLES

# DATA VALIDATION QUALIFIER CODES Based on National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents the approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
The following is an EcoChem	qualifier that may also be assigned during the data review process:

DNR Do not report; a more appropriate result is reported from another analysis or dilution.

### DATA QUALIFIER REASON CODES

Group	Code	Reason for Qualification			
Sample Handling	1	Improper Sample Handling or Sample Preservation (i.e., headspace, cooler temperature, pH, summa canister pressure); Exceeded Holding Times			
	24	Instrument Performance (i.e., tune, resolution, retention time window, endrin breakdown, lock-mass)			
	5A	Initial Calibration (RF, %RSD, r <sup>2</sup> )			
Instrument Performance	5B	Calibration Verification (CCV, CCAL; RF, %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate			
	5C	Initial Calibration Verification (ICV %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate			
	6	Field Blank Contamination (Equipment Rinsate, Trip Blank, etc.)			
Blank Contamination	7	Lab Blank Contamination (i.e., method blank, instrument blank, etc.) Use low bias flag (L) <sup>1</sup> for negative instrument blanks			
	8	Matrix Spike (MS and/or MSD) Recoveries Use bias flags (H,L) <sup>1</sup> where appropriate			
Precision and Accuracy	9	Precision (all replicates: LCS/LCSD, MS/MSD, Lab Replicate, Field Replicate)			
	10	Laboratory Control Sample Recoveries (a.k.a. Blank Spikes) Use bias flags (H,L) <sup>1</sup> where appropriate			
	12	Reference Material Use bias flags (H,L) <sup>1</sup> where appropriate			
	13	Surrogate Spike Recoveries (a.k.a. labeled compounds, recovery standards) Use bias flags (H,L) <sup>1</sup> where appropriate			
	16	ICP/ICP-MS Serial Dilution Percent Difference			
	17	ICP/ICP-MS Interference Check Standard Recovery Use bias flags (H,L) <sup>1</sup> where appropriate			
Interferences	19	Internal Standard Performance (i.e., area, retention time, recovery)			
	22	Elevated Detection Limit due to Interference (i.e., chemical and/or matrix)			
	23	Bias from Matrix Interference (i.e. diphenyl ether, PCB/pesticides)			
	2	Chromatographic pattern in sample does not match pattern of calibration standard			
	3	2 <sup>nd</sup> column confirmation (RPD or %D)			
Identification and Quantitation	4	Tentatively Identified Compound (TIC) (associated with NJ only)			
	20	Calibration Range or Linear Range Exceeded			
	25	Compound Identification (i.e., ion ratio, retention time, relative abundance, etc.)			
N.C II	11	A more appropriate result is reported (multiple reported analyses i.e., dilutions, re- extractions, etc. Associated with "R" and "DNR" only)			
Miscellaneous	14	Other (See DV report for details)			
	26	Method QC information not provided			

<sup>1</sup>H = high bias indicated

L = low bias indicated

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Waters/Solids $\leq 6^{\circ}C \&$ in the dark Tissues <-10°C & in the dark <b>Preservation Aqueous:</b> If Cl <sub>2</sub> is present Thiosulfate must be added and if pH > 9 it must be adjusted to 7 - 9	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/R(ND) if thiosulfate not added if Cl <sub>2</sub> present; J(pos)/UJ(ND) if pH not adjusted J(pos)/UJ(ND) if temp > 20°C		EcoChem PJ, see TM-05
Holding Time	If properly stored, 1 year or: Extraction (all matrices): 30 days from collection Analysis (all matrices): 45 days from extraction	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If not properly stored or HT exceedance: J(pos)/UJ(ND)	1	EcoChem PJ, see TM-05 Gross exceedance = > 1 year 2011 NFG Note: Under CWA, SDWA, and RCRA the HT for H2O is 7 days.
Instrument Performa	nce				
Mass Resolution (Tuning)	PFK (Perfluorokerosene) ≥10,000 resolving power at m/z 304.9824. Exact mass of m/z 380.9760 w/in 5 ppm of theoretical value (380.97410 to 380.97790) . Analyzed prior to ICAL and at the start and end of each 12 hr. shift.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	R(pos/ND) all analytes in all samples associated with the tune	24	Notify PM
Windows Defining Mix	Peaks for first and last eluters must be within established retention time windows for each selector group (chlorination level)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If peaks are not completely within windows (clipped): If natives are ok, J(pos)/UJ(ND) homologs (Totals) If natives are affected, R all results for that selector group	24	Notify PM
Column Performance Mix	Both mixes must be analyzed before ICAL and CCAL Valley < 25% (valley = (x/y)*100%) where x = ht. of TCDD (or TCDF) & y = baseline to bottom of valley For all isomers eluting near the 2378-TCDD (TCDF) peak (TCDD only for 8290)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if valley > 25%	24	EcoChem PJ, see TM-05, Rev. 2; Note: TCDF is evaluated only if second column confirmation is performed
Initial Calibration Sensitivity	S/N ratio > 10 for all native and labeled compounds in CS1 std.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5A	
Initial Calibration <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If 2 or more ion ratios are out for one compound in ICAL, J(pos)	5A	EcoChem PJ, see TM-05, Rev. 2

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Instrument Performa	nce (continued)				
Initial Calibration (Minimum 5 stds.)	%RSD < 20% for native compounds %RSD <30% for labeled compounds (%RSD < 35% for labeled compounds under 1613b)	NFG <sup>(1)</sup> J(pos) natives if %RSD > 20%           Method <sup>(2)</sup> 54		5A	
Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD >25 min on DB5 & >15 min on DB-225	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action		EcoChem PJ, see TM-05, Rev. 2
Continuing Calibration (Prior to each 12 hr. shift) <b>Sensitivity</b>	S/N ratio for CS3 standard > 10	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5B	
Continuing Calibration (Prior to each 12 hr. shift) <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	For congener with ion ratio outlier, J(pos) natives in all samples associated with CCAL. No action for labeled congener ion ratio outliers.	25	EcoChem PJ, see TM-05
Continuing Calibration (Prior to each 12 hr.	%D+/-20% for native compounds %D +/-30% for labeled compounds (Must meet limits in Table 6, Method 1613B) If %D in the closing CCAL are within 25%/35%, the mean RF from the two CCAL may be used to calculate samples (Section 8.3.2.4 of 8290).	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Labeled compounds: Narrate, no action. Native compounds: 1613: J(pos)/UJ(ND)if %D is outside Table 6 limits J(pos)/R(ND) if %D is +/-75% of Table 6 limits 8290: J(pos)/UJ(ND) if %D = 20% - 75% J(pos)/R(ND) if %D > 75%	5B (H,L) <sup>3</sup>	
shift) <b>Stability</b>	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD and <sup>13</sup> C <sub>12</sub> -123789-HxCDD should be ± 15 seconds of ICAL RRT for all other compounds must meet criteria listed in Table 2 Method 1316.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action	5B	EcoChem PJ, see TM-05
Blank Contamination					
Method Blank (MB)	MB: One per matrix per batch of (of ≤ 20 samples) No detected compounds > RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U(pos) if result is < 5X action level.	7	Hierarchy of blank review: #1 - Review MB, qualify as needed
Field Blank (FB)	FB: frequency as per QAPP No detected compounds > RL	WELTOU	U(pos) if result is < 5X action level.	6	#2 - Review FB , qualify as needed

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Precision and Accura	cy				
MS/MSD (recovery)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of $\leq$ 20 samples)	EcoChem standard policy	J(pos) if both %R > UCL - high bias J(pos)/UJ(ND) if both %R < LCL - low bias J(pos)/R(ND) if both %R < 10% - very low bias	8 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria. No action if parent concentration is >4x the amount spiked.
	Use most current laboratory control limits		J(pos)/UJ(ND) if one > UCL & one < LCL, with no bias PJ if only one %R outlier		Qualify parent sample only unless other QC indicates systematic problems.
MS/MSD (RPD)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) in parent sample if RPD > CL	9	Qualify parent sample only.
LCS (or OPR)	One per lab batch (of ≤ 20 samples) Use most current laboratory control limits <b>or</b> Limits from Table 6 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	10 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria, when LCSD is analyzed. Qualify all associated samples.
LCS/LCSD (RPD)	LCSD not typically required for HRMS analyses. One set per matrix and batch of 20 samples RPD < 35%	Method <sup>(2)</sup> Ecochem standard policy	J(pos) assoc. compound in all samples if RPD > CL	9	Qualify all associated samples.
Lab Duplicate (RPD)	Lab Dup not typically required for HRMS analyses. One per lab batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos)/UJ(ND) if RPD > CL	9	
Labeled Compounds (Internal Standards)	Added to all samples %R = 40% - 135% in all samples 8290 %R must meet limits in Table 7 Method 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	13 (H,L) <sup>3</sup>	
Field Duplicates	Solids: RPD <50% OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Narrate and qualify if required by project	9	Use professional judgment

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason	Discussion and Comments
Compound ID and Ca	•			Code	
Quantitation/ Identification	All ions for each isomer must maximize within ± 2 seconds. S/N ratio >2.5 Ion ratios must meet criteria listed in Table 8 Method 8290, or Table 9 of 1613B; RRTs w/in limits in Table 2 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate in report; qualify if necessary NJ(pos) for retention time outliers. U(pos) for ion ratio outliers.	25	EcoChem PJ, see TM-05
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If laboratory correctly reported an EMPC value, qualify the native compound U(pos) to indicate that the value is a detection limit and qualify total homolog groups J (pos)	25	Use professional judgment See TM-18
Interferences	Interferences from chlorodiphenyl ether compounds	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if present	23	See TM-16
Interferences	Lock masses must not deviate ± 20% from values in Table 8 of 1613B	Method (2)	J(pos)/UJ(ND) if present	24	See TM-17
Second Column Confirmation	All 2,3,7,8-TCDF hits must be confirmed on a DB-225 (or equiv) column. All QC criteria must also be met for the confirmation analysis.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Report the DB-225 value. If not performed use PJ.	3	DNR-11 DB5 result if both results from both columns are reported. <b>EcoChem PJ, see TM-05</b>
Calculation Check	Check 10% of field & QC sample results	EcoChem standard policy	Contact laboratory for resolution and/or corrective action	na	Full data validation only.
Electronic Data Delive	erable (EDD)				
Verification of EDD to hardcopy data	EcoChem verify @ 10% unless problems noted; then increase level up to 100% for next several packages.		Depending on scope of problem, correct at EcoChem (minor issues) to resubmittal by laboratory (major issues).	na	EcoChem Project Manager and/or Database Administrator will work with lab to provide long-term corrective action.
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	Standard reporting policy	Use "DNR" to flag results that will not be reported.	11	

(pos) - positive (detected) results; (ND) - not detected results

<sup>1</sup> National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) & Chlorinated Dibenzofurans (CDFs) Data Review, September 2011

<sup>2</sup> Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), USEPA SW-846, Method 8290

<sup>2</sup> EPA Method 1613, Rev.B, Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGS/HRMS, October 1994

<sup>3</sup> NFG 2013 suggests using "+ / -" to indicate bias; EcoChem has chosen "H" = high bias indicated; "L" = low bias indicated.

#### Conventional Methods by Gravimetric Analysis (i.e., Total Solids, Total Dissolved Solids, Total Suspended Solids, Grain Size) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	EcoChem Acceptance Criteria	Source of Criteria	EcoChem Action for Non- Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Cooler temperature: 4°C±2°C Preservation: Analyte/Method Specific	Method <sup>(1)</sup> NFG <sup>(2)</sup>	J (pos)/UJ (ND) if preservation requirements not met	1	Use <b>PJ</b> to qualify for cooler temp outliers.
Holding Time	Analyte/Method Specific	Method NFG <sup>(2)</sup>	J (pos)/UJ (ND) if holding time exceeded	1	
<b>Blank Contamination</b>					
Method Blank (MB)	If required by method,one per matrix per batch of (of ≤ 20 samples) Blank conc < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U (pos) if result is < 5X method blank concentration	7	Refer to <b>TM-02</b> for additional information. Blank Evaluation based on NFG 1994
Precision and Accurat	cy			•	
LCS (If appropriate to method)	One per matrix per batch (of ≤ 20 samples) %R between 80-120%	Method <sup>(2)</sup>	J (pos)/R (ND) if %R <50% J (pos)/UJ (ND) if %R 50% - 79% J (pos) if %R > 120%	10 (H,L) <sup>3</sup>	Qualify all samples in batch QAPP may have overriding accuracy limits.
Reference Material (RM, SRM, or CRM)	Result $\pm 20\%$ of the 95% confidence interval of the true value for analytes	EcoChem standard policy	J (pos)/UJ (ND) if < LCL J (pos) if > UCL	12 (H,L) <sup>3</sup>	QAPP may have overriding accuracy limits. Some manufacturers may have different RM control limits

#### **Conventional Methods by Gravimetric Analysis** (i.e., Total Solids, Total Dissolved Solids, Total Suspended Solids, Grain Size) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	EcoChem Acceptance Criteria	Source of Criteria	EcoChem Action for Non- Conformance	Reason Code	Discussion and Comments
Laboratory Duplicate	One per matrix per batch (of $\leq 20$ samples) RPD $\leq 20\%$ for results $\geq 5x$ RL Solids: difference $< 2X$ RL for results $< 5X$ RL Aqueous: difference $< 1X$ RL for results $< 5X$ RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if RPD > 20% For Grain Size, no action if results for fraction are less than 5%	9	Qualify all samples in batch, except Grain Size - qualify parent only. QAPP may have overriding precision limits.
Field Duplicate	Solids: RPD <50% (for results $\ge$ 5x RL) OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% (for results $\ge$ 5x RL) OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Qualify only parent and field duplicate samples J (pos)/UJ (ND)	9	QAPP may have overriding precision limits. Client/QAPP may not require qualification based on field precision.
Compound Quantitati	on	•			
Dilutions, Re-extractions and/or Reanalyses	Report only one result per analyte per sample	EcoChem standard policy	Use "DNR" to flag results that will not be reported.	11	

<sup>1</sup> National Functional Guidelines for Inorganic Superfund Data Review, January 2010.
 <sup>2</sup> SW846 or EPA Standard Methods

(pos): Positive Result (ND): Not Detected

<sup>3</sup> "H" = high bias indicated; "L" = low bias indicated

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments					
Sample Handling										
Cooler/Storage Temperature Preservation	Cooler temperature: 4°C±2°C Preservation: Analyte/Method Specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if preservation requirements not met	1	Use <b>PJ</b> to qualify for cooler temp outliers.					
Holding Time	Analyte/Method Specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if holding time exceeded	1						
Instrument Performance										
Initial Calibration (ICAL)	blank + multiple standards as per method requirements r ≥ 0.995	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) for r < 0.995	5A						
Initial Calibration Verification (ICV)	Independent source analyzed immediately after calibration %R method specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < lower control limit (LCL) J (pos) if %R > upper control limit (UCL)	5A (H,L) <sup>3</sup>	Qualify all samples in run					
Continuing Calibration Verification (CCV)	Immediately following ICV, every 10 samples, and end of run %R method specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if %R < LCL J(pos) if %R > UCL	5B (H,L) <sup>3</sup>	Qualify samples bracketed by CCV outliers					
Blank Contamination		•	•							
Method Blank (MB)	One per matrix per batch of (of $\leq$ 20 samples) Blank conc < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U (pos) if result is < 5X method blank concentration	7	Refer to <b>TM-02</b> for additional information. Blank Evaluation based on NFG 1994					

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Instrument Blanks (ICB/CCB)	After each ICV & CCV   blank concentration   < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Action level is 5x absolute value of blank conc. For positive blanks: U (pos) results < action level For negative blanks: J (pos)/UJ (ND) results < action level	Pos Blanks: 7 Neg Blanks: 7L <sup>3</sup>	Use blanks bracketing samples for Qualification Refer to <b>TM-02</b> for additional information. <b>Hierarchy of blank review:</b> <b>#1 - Review MB, qualify as needed</b> <b>#2 - Review IB , qualify as needed</b> <b>#3 - Review FB , qualify as needed</b>
Field Blank (FB)	Blank conc < MDL	EcoChem standard policy	U (pos) if result is < 5x action level, as per analyte.	6	Qualify in associated field samples only. Refer to <b>TM-02</b> for additional information.
Precision and Accura	су				
Laboratory Control Sample (LCS)	One per matrix per batch (of ≤ 20 samples) %R within Method control limits (or Laboratory control limtis if none specified in method)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < LCL J (pos) if %R > UCL	10 (H,L) <sup>3</sup>	Qualify all samples in batch QAPP may have overriding accuracy limits.
Reference Materials (RM, CRM, SRM)	Result $\pm 20\%$ of the 95% confidence interval of the true value for analytes	EcoChem standard policy	J (pos)/UJ (ND) if < LCL J (pos) if > UCL	12 (H,L) <sup>3</sup>	QAPP may have overriding accuracy limits. Some manufacturers may have different RM control limits

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Where applicable to method; MSD may not be required One per matrix per batch (of ≤ 20 samples) For samples <4x spike level, %R within method control limits (or Laboratory control limits if none specified in method)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < LCL J (pos) if %R > UCL	8 (H,L)3	Qualify all samples in batch No action if native analyte concentration ≥ 4x spike added. Qualify all samples in batch. QAPP may have overriding accuracy limits.
Laboratory Duplicate (or MS/MSD)	One per matrix per batch (of ≤ 20 samples) RPD ≤ 20% for results ≥ 5x RL Solids: difference < 2X RL for results < 5X RL Aqueous: difference < 1X RL for results < 5X RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if RPD > 20% or if difference > control limit	9	Qualify all samples in batch. QAPP may have overriding precision limits.
Field Duplicate	Solids: RPD <50% (for results ≥ 5x RL) OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% (for results ≥ 5x RL) OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Qualify only parent and field duplicate samples J (pos)/UJ (ND)	9	QAPP may have overriding precision limits. Client/QAPP may not require qualification based on field precision.
Compound Quantita	tion				
Linear Range	Sample concentrations less than highest calibration standard	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If result exceeds linear range & sample was not diluted J (pos)	20	
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	EcoChem standard policy	Use "DNR" to flag results that will not be reported.	11	TM-04 EcoChem Policy for Rejection/Selection Process for Multiple Results

<sup>1</sup> National Functional Guidelines for Inorganic Superfund Data Review, January 2010.

<sup>2</sup> SW846 or EPA Standard Methods

<sup>3</sup> "H" = high bias indicated; "L" = low bias indicated

(pos): Positive Result (ND): Not Detected



# **APPENDIX B**

# **QUALIFIED DATA SUMMARY TABLE**

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
E1600405	SD0114	E1600405-001	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.64	ng/kg	JK	U	25
E1600405	SD0114	E1600405-001	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	1.77	ng/kg	JK	U	25
E1600405	SD0114	E1600405-001	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.339	ng/kg	JK	U	25
E1600405	SD0114	E1600405-001	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	2.53	ng/kg	K	U	25
E1600405	SD0114	E1600405-001	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.686	ng/kg	JK	U	25
E1600405	SD0128	E1600405-002	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	3.47	ng/kg	JK	U	25
E1600405	SD0128	E1600405-002	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.36	ng/kg	JK	U	25
E1600405	SD0128	E1600405-002	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.46	ng/kg	JK	U	25
E1600405	SD0128	E1600405-002	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.541	ng/kg	JK	U	25
E1600405	SD0128	E1600405-002	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	2.46	ng/kg	JK	U	25
E1600405	SD0128	E1600405-002	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.715	ng/kg	JK	U	25
E1600405	SD0129	E1600405-003	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.713	ng/kg	JK	U	25
E1600405	SD0129	E1600405-003	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.3	ng/kg	JK	U	25
E1600405	SD0129	E1600405-003	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.52	ng/kg	JK	U	25
E1600405	SD0129	E1600405-003	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	1.61	ng/kg	JK	U	25
E1600405	SD0129	E1600405-003	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.998	ng/kg	JK	U	25
E1600405	SD0113	E1600405-004	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.21	ng/kg	JK	U	25
E1600405	SD0113	E1600405-004	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.713	ng/kg	JK	U	25
E1600405	SD0113	E1600405-004	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.405	ng/kg	JK	U	25
E1600405	SD0113	E1600405-004	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.54	ng/kg	JK	U	25
E1600405	SD0113	E1600405-004	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	0.421	ng/kg	JK	U	25
E1600405	SD0112	E1600405-005	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	5.16	ng/kg	JK	U	25
E1600405	SD0112	E1600405-005	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.693	ng/kg	JK	U	25
E1600405	SD0112	E1600405-005	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.653	ng/kg	JK	U	25
E1600405	SD0112	E1600405-005	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	1.49	ng/kg	JK	U	25
E1600405	SD0112	E1600405-005	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	0.832	ng/kg	JK	U	25
E1600405	SD0112	E1600405-005	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.367	ng/kg	JK	U	25
E1600405	SD0112	E1600405-005	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.521	ng/kg	JK	U	25
E1600405	SD0127	E1600405-006	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.732	ng/kg	JK	U	25
E1600405	SD0127	E1600405-006	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.16	ng/kg	JK	U	25
E1600405	SD0127	E1600405-006	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.376	ng/kg	JK	U	25

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
E1600405	SD0126	E1600405-007	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.02	ng/kg	JK	U	25
E1600405	SD0109	E1600405-008	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	12.5	ng/kg	Р	J	23H
E1600405	SD0109	E1600405-008	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.818	ng/kg	JK	U	25
E1600405	SD0125	E1600405-009	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.73	ng/kg	JKP	UJ	23,25
E1600405	SD0125	E1600405-009	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.274	ng/kg	BJK	U	25
E1600405	SD0125	E1600405-009	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.326	ng/kg	JK	U	25
E1600405	SD0125	E1600405-009	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.315	ng/kg	BJK	U	25
E1600405	SD0125	E1600405-009	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.209	ng/kg	JK	U	25
E1600405	SD0125	E1600405-009	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.355	ng/kg	BJ	U	7
E1600405	SD0125	E1600405-009	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	0.423	ng/kg	JK	U	25
E1600405	SD0125	E1600405-009	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	1.86	ng/kg	К	U	25
E1600405	SD0124	E1600405-010	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	2.18	ng/kg	JP	J	23H
E1600405	SD0124	E1600405-010	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.451	ng/kg	BJK	U	25
E1600405	SD0124	E1600405-010	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.474	ng/kg	JK	U	25
E1600405	SD0124	E1600405-010	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.45	ng/kg	BJ	U	7
E1600405	SD0106	E1600405-011	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	5.37	ng/kg	Р	J	23H
E1600405	SD0106	E1600405-011	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.352	ng/kg	JK	U	25
E1600405	SD0107	E1600405-012	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	7.79	ng/kg	Р	J	23H
E1600405	SD0107	E1600405-012	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.408	ng/kg	BJK	U	25
E1600405	SD0108	E1600405-013	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	8.58	ng/kg	Р	J	23H
E1600405	SD0123	E1600405-014	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	9.47	ng/kg	Р	J	23H
E1600405	SD0123	E1600405-014	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.1	ng/kg	JK	U	25
E1600405	SD0123	E1600405-014	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.724	ng/kg	JK	U	25
E1600405	SD0123	E1600405-014	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	1.8	ng/kg	JK	U	25
E1600405	SD0122	E1600405-015	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	2.12	ng/kg	JK	U	25
E1600405	SD0122	E1600405-015	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	2.59	ng/kg	JK	U	25
E1600405	SD0122	E1600405-015	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	1.89	ng/kg	JK	U	25
E1600405	SD0105	E1600405-016	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	9.97	ng/kg	К	U	25
E1600405	SD0105	E1600405-016	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.748	ng/kg	JK	U	25
E1600405	SD0105	E1600405-016	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.21	ng/kg	JK	U	25
E1600405	SD0105	E1600405-016	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	2.08	ng/kg	JK	U	25

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
E1600405	SD0105	E1600405-016	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.912	ng/kg	JK	U	25
E1600405	SD0105	E1600405-016	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	1.64	ng/kg	JK	U	25
E1600405	SD0104	E1600405-017	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.46	ng/kg	JK	U	25
E1600405	SD0104	E1600405-017	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	3.23	ng/kg	JK	U	25
E1600405	SD0104	E1600405-017	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	3.9	ng/kg	JK	U	25
E1600405	SD0104	E1600405-017	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	2.42	ng/kg	JK	U	25
E1600405	SD0103	E1600405-018	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.33	ng/kg	JK	U	25
E1600405	SD0103	E1600405-018	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.911	ng/kg	JK	U	25
E1600405	SD0115	E1600405-019	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.23	ng/kg	JK	U	25
E1600405	SD0115	E1600405-019	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	3.03	ng/kg	JK	U	25
E1600405	SD0115	E1600405-019	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.11	ng/kg	JK	U	25
E1600405	SD0115	E1600405-019	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	3.13	ng/kg	JK	U	25
E1600405	SD0115	E1600405-019	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	1.6	ng/kg	JK	U	25
E1600405	SD0115	E1600405-019	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.936	ng/kg	JK	U	25
E1600405	SD0120	E1600405-020	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.26	ng/kg	JK	U	25
E1600405	SD0120	E1600405-020	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.06	ng/kg	JK	U	25
E1600405	SD0120	E1600405-020	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.08	ng/kg	JK	U	25
E1600405	SD0120	E1600405-020	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	2.93	ng/kg	JK	U	25
E1600405	SD0120	E1600405-020	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.845	ng/kg	JK	U	25
E1600405	SD0120	E1600405-020	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.942	ng/kg	JK	U	25
E1600405	SD0121	E1600405-021	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	11.4	ng/kg	К	U	25
E1600405	SD0121	E1600405-021	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.984	ng/kg	JK	U	25
E1600405	SD0121	E1600405-021	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.25	ng/kg	JK	U	25
E1600405	SD0121	E1600405-021	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	1.18	ng/kg	JK	U	25
E1600405	SD0121	E1600405-021	EPA1613B	Octachlorodibenzofuran	36	ng/kg	К	U	25
E1600405	SD0102	E1600405-022	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	1.27	ng/kg	JK	U	25
E1600405	SD0102	E1600405-022	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	2.61	ng/kg	JK	U	25
E1600405	SD0102	E1600405-022	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.32	ng/kg	JK	U	25
E1600405	SD0102	E1600405-022	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	1.99	ng/kg	JK	U	25
E1600405	SD0102	E1600405-022	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	1.85	ng/kg	JK	U	25
E1600405	SD0102	E1600405-022	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	1.63	ng/kg	JK	U	25

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
E1600405	SD0102	E1600405-022	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	15.5	ng/kg	К	U	25
E1600405	SD0101	E1600405-023	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.965	ng/kg	JK	U	25
E1600405	SD0101	E1600405-023	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1	ng/kg	JK	U	25
E1600405	SD0101	E1600405-023	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	2.67	ng/kg	JK	U	25
E1600405	SD0101	E1600405-023	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.973	ng/kg	JK	U	25
E1600405	FW0100	E1600405-024	EPA1613B	Octachlorodibenzo-p-dioxin	13.4	pg	BJK	U	25
E1600405	SD0100	E1600405-025	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.46	ng/kg	JK	U	25
E1600405	SD0100	E1600405-025	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.44	ng/kg	JK	U	25
E1600405	SD0100	E1600405-025	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	2.22	ng/kg	JK	U	25
E1600405	SD0100	E1600405-025	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	2.08	ng/kg	JK	U	25
E1600405	SD0100	E1600405-025	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	1.22	ng/kg	JK	U	25
E1600405	SD0100	E1600405-025	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	2.08	ng/kg	JK	U	25
E1600405	SD0119	E1600405-026	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.54	ng/kg	JK	U	25
E1600405	SD0119	E1600405-026	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.33	ng/kg	JK	U	25
E1600405	SD0119	E1600405-026	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	2.23	ng/kg	JK	U	25
E1600405	SD0119	E1600405-026	EPA1613B	1,2,3,7,8-Pentachlorodibenzofuran	2.03	ng/kg	JK	U	25
E1600405	SD0119	E1600405-026	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	1.21	ng/kg	JK	U	25
E1600405	SD0119	E1600405-026	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	2.62	ng/kg	JK	U	25
E1600405	SD0118	E1600405-027	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	2.83	ng/kg	JK	U	25
E1600405	SD0118	E1600405-027	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.526	ng/kg	BJK	U	25
E1600405	SD0118	E1600405-027	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.384	ng/kg	BJK	U	25
E1600405	SD0118	E1600405-027	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.33	ng/kg	BJK	U	25
E1600405	SD0117	E1600405-028	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	1.25	ng/kg	JK	U	25
E1600405	SD0117	E1600405-028	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.402	ng/kg	BJK	U	25
E1600405	SD0117	E1600405-028	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.51	ng/kg	BJK	U	25
E1600405	SD0117	E1600405-028	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.409	ng/kg	BJ	U	7
E1600406	FB0013	E1600406-002	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	6.66	pg	BJK	U	25
E1600406	FW0100	E1600406-003	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	6.56	pg	BJK	U	25
E1600406	FW0100	E1600406-003	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	2.53	pg	JK	U	25
E1600406	FW0100	E1600406-003	EPA1613B	Octachlorodibenzo-p-dioxin	16.1	pg	BJK	U	25
E1600406	FB0100	E1600406-004	EPA1613B	Octachlorodibenzo-p-dioxin	10.6	pg	BJK	U	25



# DATA VALIDATION REPORT

# SAN JACINTO RIVER WASTE PITS 2016 SEDIMENT SAMPLING

#### **Prepared for:**

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EcoChem Project: C22130-23

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**Approved for Release:** 

Melissa Swanson Project Chemist EcoChem, Inc.

# **PROJECT NARRATIVE**

#### **Basis for Data Validation**

This report summarizes the results of full validation (EPA Stage 3/4) performed on sediment sample data and compliance validation (EPA Stage 2A) performed on wipe blank sample data for the San Jacinto River 2016 Sediment Sampling Study. A complete list of samples is provided in the **Sample Index**.

Samples were analyzed for conventional parameters by ALS Environmental, Kelso, Washington. ALS Environmental, Houston, Texas performed the dioxin analyses. The analytical methods and EcoChem project chemists are listed below.

Analysis	Method	PRIMARY REVIEW	Secondary Review	
Dioxin/Furan Compounds	1613B	M. Swanson		
Total Organic Carbon	D4129-05ALS	E Clautan	A. Bodkin	
Grainsize	PSEP	E. Clayton		

The data were reviewed using guidance and quality control criteria documented in the analytical methods and the following project and guidance documents:

- Sampling and Analysis Plan: Sediment Study San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, April 2010).
- Addendum 3 (to the Sediment Sampling and Analysis Plan) Additional Sediment Sampling within the USEPA's Preliminary Site Perimeter, San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, March 2016).
- USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review (USEPA 2011).
- USEPA National Functional Guidelines for Inorganic Data Review (USEPA October 2004).

EcoChem's goal in assigning data assessment qualifiers is to assist in proper data interpretation. If values are estimated (J or UJ), data may be used for site evaluation and risk assessment purposes but reasons for data qualification should be taken into consideration when interpreting sample concentrations. If values are assigned an R, the data are to be rejected and should not be used for any site evaluation purposes. If values have no data qualifier assigned, then the data meet the data quality objectives as stated in the documents and methods referenced above.

Data qualifier definitions, reason codes, and validation criteria are included as **APPENDIX A**. A Qualified Data Summary Table is included in **APPENDIX B**. Data Validation Worksheets and project associated communications will be kept on file at EcoChem, Inc. A qualified laboratory electronic data deliverable (EDD) is also submitted with this report.

# Qualified Data Summary Table

Dioxin	Conventionals	Sample ID	Dioxin	Dioxins	Conventionals	Conventionals
SDG	SDG		Laboratory ID		Laboratory ID	
E1600405	K1608142	SD0110	E1600726-001	$\checkmark$	K1608142-001	$\checkmark$
E1600405	K1608142	SD0111	E1600726-002	$\checkmark$	K1608142-002	$\checkmark$
E1600405	K1608142	SD0116	E1600726-003	$\checkmark$	K1608142-003	$\checkmark$
E1600405		FW0102	E1600726-004	$\checkmark$		
E1600405		FB0101	E1600726-005	$\checkmark$		

### San Jacinto River Waste Pits Sediments 2016

# DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 Sediment Sampling Dioxin/Furan Compounds by EPA 1613B

This report documents the review of analytical data from the analyses of sediment samples and the associated laboratory and field quality control (QC) samples. ALS Environmental, Houston, Texas, analyzed the samples. Refer to the **Sample Index** for a complete list of samples.

SDG	Number of Samples	VALIDATION LEVEL	
E1600726	3 Sediment	EPA Stage 4	
E1000720	2 Wipe	EPA Stage 2A	

#### DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

#### EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package. No errors were noted.

#### TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below.

~	Sample Receipt, Preservation, and Holding Times	~	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	
$\checkmark$	✓ System Performance and Resolution Checks		Matrix Spikes/Matrix Spike Duplicates (MS/MSD)	
$\checkmark$	Initial Calibration (ICAL)	1	Field Replicate Samples	
$\checkmark$	Calibration Verification	$\checkmark$	Target Analyte List	
2	Laboratory Blanks	$\checkmark$	Reported Results	
2	Field Blanks	2	Compound Identification	
$\checkmark$	Labeled Compound Recovery	1	Calculation Verification	

✓ Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

#### **Laboratory Blanks**

To assess the impact of any blank contaminant on the reported sample results, an action level was established at five times (5x) the concentration reported in the blank. If a contaminant was reported in an associated field sample and the concentration was less than the action level, the result was gualified as not detected (U-7). No action was taken if the sample result was greater than the action

level or for non-detected results. The laboratory assigned K-flags to values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are "estimated maximum possible concentrations". When these occurred in the method blank the results were considered as false positives. No action levels were established for these analytes.

Method blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method blanks, however only the results noted below required qualification; all other associated sample results were either not detected or were detected at concentrations greater than the action levels.

The results for 1,2,3,4,6,7,8-HpCDD in Samples FB0101 and FW0102 and 1,2,3,4,6,7,8-HpCDF in Sample FB0101 were qualified as not detected (U-7).

#### Field Blanks

The field blanks for this project are filter blank and filter wipe samples. To evaluate the effect of field blank contamination on the sample data, action levels of 5x the blank concentrations were established. After qualification based on method blank contamination, any remaining positive results in the filter blank are applied to the filter wipe and any remaining positive results in the filter wipe are applied to the sediment samples. For the purposes of evaluation, the reported filter wipe values (in total pg) were converted to the same units as the field samples (ng/kg) using the average sample mass and volume collected. If a contaminant was detected in an associated field sample and the concentration was less than the action level, the result was qualified (U-6) at the reported concentration. No action was taken if the sample result was greater than the action level, or for non-detected results.

Wipe samples FB0101 and FW0102 were submitted with this data package. After qualification due to method blank contamination, positive results remained for OCDD, 1,2,3,6,7,8-HxCDF, and OCDF in Sample FB0101. Results for these compounds were qualified as not detected (U-6) in Sample FW0102.

After qualification due to method blank contamination and filter blank contamination, a positive result remained for 1,2,3,4,7,8,9-HpCDF in Sample FW0102. All results in the associated sediment samples were detected at concentrations greater than the action level. No data were qualified based on filter wipe contamination.

#### Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) samples are not required by the method and were not analyzed. Accuracy and precision were evaluated using the labeled compound and laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results.

#### **Field Replicates**

No field replicates were submitted with this data package.

#### **Compound Identification**

The laboratory assigned K-flags to results where a peak was detected but did not meet ion ratio quantitation criteria. The reported values cannot be considered as positive identifications for these analytes. These results were considered potential false positives or estimated maximum possible concentrations (EMPC) and were qualified as not detected (U-25) at the reported values.

The method requires the confirmation of 2,3,7,8-TCDF using an alternate GC column as the DB5 column that is typically used cannot fully separate 2,3,7,8-TCDF from closely eluting non-target TCDF isomers. The laboratory did not perform a second column confirmation; however, the laboratory uses a DB-5MSUI column. This column provides adequate resolution of the TCDF isomers as indicated by the acceptable peak to valley ratios. Since the 2,3,7,8-TCDF resolution was acceptable, no action was necessary.

#### **Calculation Verification**

Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

#### OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable as demonstrated by labeled compound and LCS/LCSD %R values. Precision was acceptable as demonstrated by the LCS/LCSD RPD values.

Detection limits were elevated due to ion ratio outliers, method blank contamination and field blank contamination.

All data, as qualified, are acceptable for use.

# DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 Sediment Sampling Conventional Parameters

This report documents the review of analytical data from the analyses of sediment samples and the associated laboratory quality control (QC) samples. ALS Environmental, Kelso, Washington, analyzed the samples. Refer to the **Sample Index** for a complete list of samples.

SDG	Number of Samples	VALIDATION LEVEL
K1608142	3 Sediment	EPA Stage 3

The analytical tests that were performed are summarized below.

Parameter	Method
Grain Size	PSEP
Total Organic Carbon	ASTM D4129-05M
Total Solids	160.3M

#### DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

#### EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package. No errors were noted.

#### **TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below:

$\checkmark$	Sample Receipt, Preservation, and Holding Times	$\checkmark$	Matrix Spikes (MS)
$\checkmark$	Initial Calibration	2	Laboratory Replicates
$\checkmark$	Calibration Verification	1	Field Replicates
$\checkmark$	Laboratory Blanks	$\checkmark$	Reporting Limits
1	Field Blanks	$\checkmark$	Reported Results
$\checkmark$	Laboratory Control Samples (LCS)	1	Calculation Verification

 $\checkmark$  Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

<sup>1</sup> Quality control results are discussed below, but no data were qualified.

#### **Field Blanks**

No field blanks were submitted.

#### **Laboratory Replicates**

Sample SD0116 analyzed in triplicate for grain size and Sample SD0110 was analyzed in quadruplicate for total organic carbon (TOC). Percent relative standard deviation (%RSD) values were evaluated for results greater than five times the method reporting limit (MRL). With the exceptions noted below, the %RSD values were less than the 20% control limit.

The %RSD values for grain size (gravel, fine sand, and silt) were greater than the control limit. Results for these analytes were estimated (J-9) in the parent, duplicate, and triplicate samples.

#### **Field Replicates**

No field replicates were included with this data set.

#### **Calculation Verification**

Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

#### OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable as demonstrated by the laboratory control sample percent recovery values. With the exceptions noted above, precision was acceptable as demonstrated by the laboratory and field replicate %RSD values.

Data were qualified due to laboratory precision outliers.

All data, as qualified, are acceptable for use.



# **APPENDIX A**

# DATA QUALIFIER DEFINITIONS REASON CODES AND CRITERIA TABLES

## DATA VALIDATION QUALIFIER CODES Based on National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents the approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
The following is an EcoChem	qualifier that may also be assigned during the data review process:

DNR Do not report; a more appropriate result is reported from another analysis or dilution.

## DATA QUALIFIER REASON CODES

Group	Code	Reason for Qualification
Sample Handling	1	Improper Sample Handling or Sample Preservation (i.e., headspace, cooler temperature, pH, summa canister pressure); Exceeded Holding Times
	24	Instrument Performance (i.e., tune, resolution, retention time window, endrin breakdown, lock-mass)
	5A	Initial Calibration (RF, %RSD, r <sup>2</sup> )
Instrument Performance Blank Contamination Precision and Accuracy Interferences	5B	Calibration Verification (CCV, CCAL; RF, %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	5C	Initial Calibration Verification (ICV %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	6	Field Blank Contamination (Equipment Rinsate, Trip Blank, etc.)
Blank Contamination	7	Lab Blank Contamination (i.e., method blank, instrument blank, etc.) Use low bias flag (L) <sup>1</sup> for negative instrument blanks
	8	Matrix Spike (MS and/or MSD) Recoveries Use bias flags (H,L) <sup>1</sup> where appropriate
Precision and Accuracy	9	Precision (all replicates: LCS/LCSD, MS/MSD, Lab Replicate, Field Replicate)
	10	Laboratory Control Sample Recoveries (a.k.a. Blank Spikes) Use bias flags (H,L) <sup>1</sup> where appropriate
	12	Reference Material Use bias flags (H,L) <sup>1</sup> where appropriate
	13	Surrogate Spike Recoveries (a.k.a. labeled compounds, recovery standards) Use bias flags (H,L) <sup>1</sup> where appropriate
	16	ICP/ICP-MS Serial Dilution Percent Difference
	17	ICP/ICP-MS Interference Check Standard Recovery Use bias flags (H,L) <sup>1</sup> where appropriate
Interferences	19	Internal Standard Performance (i.e., area, retention time, recovery)
	22	Elevated Detection Limit due to Interference (i.e., chemical and/or matrix)
	23	Bias from Matrix Interference (i.e. diphenyl ether, PCB/pesticides)
	2	Chromatographic pattern in sample does not match pattern of calibration standard
Identification and	3	2 <sup>nd</sup> column confirmation (RPD or %D)
Identification and Quantitation	4	Tentatively Identified Compound (TIC) (associated with NJ only)
	20	Calibration Range or Linear Range Exceeded
	25	Compound Identification (i.e., ion ratio, retention time, relative abundance, etc.)
MC	11	A more appropriate result is reported (multiple reported analyses i.e., dilutions, re- extractions, etc. Associated with "R" and "DNR" only)
Miscellaneous	14	Other (See DV report for details)
	26	Method QC information not provided

<sup>1</sup>H = high bias indicated

L = low bias indicated

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Waters/Solids $\leq 6^{\circ}C \&$ in the dark Tissues <-10°C & in the dark <b>Preservation Aqueous:</b> If Cl <sub>2</sub> is present Thiosulfate must be added and if pH > 9 it must be adjusted to 7 - 9	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/R(ND) if thiosulfate not added if Cl <sub>2</sub> present; J(pos)/UJ(ND) if pH not adjusted J(pos)/UJ(ND) if temp > 20°C	1	EcoChem PJ, see TM-05
Holding Time	If properly stored, 1 year or: Extraction (all matrices): 30 days from collection Analysis (all matrices): 45 days from extraction	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If not properly stored or HT exceedance: J(pos)/UJ(ND)	1	EcoChem PJ, see TM-05 Gross exceedance = > 1 year 2011 NFG Note: Under CWA, SDWA, and RCRA the HT for H2O is 7 days.
Instrument Performa	nce				
Mass Resolution (Tuning)	PFK (Perfluorokerosene) ≥10,000 resolving power at m/z 304.9824. Exact mass of m/z 380.9760 w/in 5 ppm of theoretical value (380.97410 to 380.97790) . Analyzed prior to ICAL and at the start and end of each 12 hr. shift.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	R(pos/ND) all analytes in all samples associated with the tune	24	Notify PM
Windows Defining Mix	Peaks for first and last eluters must be within established retention time windows for each selector group (chlorination level)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If peaks are not completely within windows (clipped): If natives are ok, J(pos)/UJ(ND) homologs (Totals) If natives are affected, R all results for that selector group	24	Notify PM
Column Performance Mix	Both mixes must be analyzed before ICAL and CCAL Valley < 25% (valley = (x/y)*100%) where x = ht. of TCDD (or TCDF) & y = baseline to bottom of valley For all isomers eluting near the 2378-TCDD (TCDF) peak (TCDD only for 8290)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if valley > 25%	24	EcoChem PJ, see TM-05, Rev. 2; Note: TCDF is evaluated only if second column confirmation is performed
Initial Calibration Sensitivity	S/N ratio > 10 for all native and labeled compounds in CS1 std.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5A	
Initial Calibration <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If 2 or more ion ratios are out for one compound in ICAL, J(pos)	5A	EcoChem PJ, see TM-05, Rev. 2

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
nstrument Performa	ance (continued)				
Initial Calibration (Minimum 5 stds.)	%RSD < 20% for native compounds %RSD <30% for labeled compounds (%RSD < 35% for labeled compounds under 1613b)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) natives if %RSD > 20%	5A	
Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD >25 min on DB5 & >15 min on DB-225	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action		EcoChem PJ, see TM-05, Rev. 2
Continuing Calibration (Prior to each 12 hr. shift) <b>Sensitivity</b>	S/N ratio for CS3 standard > 10	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5B	
Continuing Calibration (Prior to each 12 hr. shift) <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	For congener with ion ratio outlier, J(pos) natives in all samples associated with CCAL. No action for labeled congener ion ratio outliers.	25	EcoChem PJ, see TM-05
Continuing Calibration (Prior to each 12 hr.	%D+/-20% for native compounds %D +/-30% for labeled compounds (Must meet limits in Table 6, Method 1613B) If %D in the closing CCAL are within 25%/35%, the mean RF from the two CCAL may be used to calculate samples (Section 8.3.2.4 of 8290).	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Labeled compounds: Narrate, no action. Native compounds: 1613: J(pos)/UJ(ND)if %D is outside Table 6 limits J(pos)/R(ND) if %D is +/-75% of Table 6 limits 8290: J(pos)/UJ(ND) if %D = 20% - 75%	5B (H,L) <sup>3</sup>	
shift) <b>Stability</b>	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD and <sup>13</sup> C <sub>12</sub> -123789-HxCDD should be ± 15 seconds of ICAL RRT for all other compounds must meet criteria listed in Table 2 Method 1316.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/R(ND) if %D > 75% Narrate, no action	5B	EcoChem PJ, see TM-05
Blank Contamination	1 I				
Method Blank (MB)	MB: One per matrix per batch of (of ≤ 20 samples) No detected compounds > RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U(pos) if result is < 5X action level.	7	Hierarchy of blank review: #1 - Review MB, qualify as needed
Field Blank (FB)	FB: frequency as per QAPP No detected compounds > RL	метной	U(pos) if result is < 5X action level.	6	#2 - Review FB , qualify as needed

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Precision and Accura	cy				
MS/MSD (recovery)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples)	EcoChem standard policy	J(pos) if both %R > UCL - high bias J(pos)/UJ(ND) if both %R < LCL - low bias J(pos)/R(ND) if both %R < 10% - very low bias	8 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria. No action if parent concentration is >4x the amount spiked.
	Use most current laboratory control limits		J(pos)/UJ(ND) if one > UCL & one < LCL, with no bias PJ if only one %R outlier		Qualify parent sample only unless other QC indicates systematic problems.
MS/MSD (RPD)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) in parent sample if RPD > CL	9	Qualify parent sample only.
LCS (or OPR)	One per lab batch (of ≤ 20 samples) Use most current laboratory control limits <b>or</b> Limits from Table 6 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	10 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria, when LCSD is analyzed. Qualify all associated samples.
LCS/LCSD (RPD)	LCSD not typically required for HRMS analyses. One set per matrix and batch of 20 samples RPD < 35%	Method <sup>(2)</sup> Ecochem standard policy	J(pos) assoc. compound in all samples if RPD > CL	9	Qualify all associated samples.
Lab Duplicate (RPD)	Lab Dup not typically required for HRMS analyses. One per lab batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos)/UJ(ND) if RPD > CL	9	
Labeled Compounds (Internal Standards)	Added to all samples %R = 40% - 135% in all samples 8290 %R must meet limits in Table 7 Method 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	13 (H,L) <sup>3</sup>	
Field Duplicates	Solids: RPD <50% OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Narrate and qualify if required by project	9	Use professional judgment

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason	Discussion and Comments
Compound ID and Ca	•			Code	
Quantitation/ Identification	All ions for each isomer must maximize within ± 2 seconds. S/N ratio > 2.5 Ion ratios must meet criteria listed in Table 8 Method 8290, or Table 9 of 1613B; RRTs w/in limits in Table 2 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate in report; qualify if necessary NJ(pos) for retention time outliers. U(pos) for ion ratio outliers.	25	EcoChem PJ, see TM-05
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If laboratory correctly reported an EMPC value, qualify the native compound U(pos) to indicate that the value is a detection limit and qualify total homolog groups J (pos)	25	Use professional judgment See TM-18
Interferences	Interferences from chlorodiphenyl ether compounds	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if present	23	See TM-16
Interferences	Lock masses must not deviate ± 20% from values in Table 8 of 1613B	Method <sup>(2)</sup>	J(pos)/UJ(ND) if present	24	See TM-17
Second Column Confirmation	All 2,3,7,8-TCDF hits must be confirmed on a DB-225 (or equiv) column. All QC criteria must also be met for the confirmation analysis.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Report the DB-225 value. If not performed use PJ.	3	DNR-11 DB5 result if both results from both columns are reported. <b>EcoChem PJ, see TM-05</b>
Calculation Check	Check 10% of field & QC sample results	EcoChem standard policy	Contact laboratory for resolution and/or corrective action	na	Full data validation only.
Electronic Data Delive	erable (EDD)				
Verification of EDD to hardcopy data	EcoChem verify @ 10% unless problems noted; then increase level up to 100% for next several packages.		Depending on scope of problem, correct at EcoChem (minor issues) to resubmittal by laboratory (major issues).	na	EcoChem Project Manager and/or Database Administrator will work with lab to provide long-term corrective action.
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	Standard reporting policy	Use "DNR" to flag results that will not be reported.	11	

(pos) - positive (detected) results; (ND) - not detected results

<sup>1</sup> National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) & Chlorinated Dibenzofurans (CDFs) Data Review, September 2011

<sup>2</sup> Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), USEPA SW-846, Method 8290

<sup>2</sup> EPA Method 1613, Rev.B, Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGS/HRMS, October 1994

<sup>3</sup> NFG 2013 suggests using "+ / -" to indicate bias; EcoChem has chosen "H" = high bias indicated; "L" = low bias indicated.

#### Conventional Methods by Gravimetric Analysis (i.e., Total Solids, Total Dissolved Solids, Total Suspended Solids, Grain Size) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	EcoChem Acceptance Criteria	Source of Criteria			Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Cooler temperature: 4°C±2°C Preservation: Analyte/Method Specific	Method <sup>(1)</sup> NFG <sup>(2)</sup>	J (pos)/UJ (ND) if preservation requirements not met	1	Use <b>PJ</b> to qualify for cooler temp outliers.
Holding Time	Analyte/Method Specific	Method NFG <sup>(2)</sup>	J (pos)/UJ (ND) if holding time exceeded	1	
<b>Blank Contamination</b>					
Method Blank (MB)	k (MB) If required by method,one per matrix per batch of (of ≤ 20 samples) Blank conc < MDL		U (pos) if result is < 5X method blank concentration	7	Refer to <b>TM-02</b> for additional information. Blank Evaluation based on NFG 1994
Precision and Accurat	cy			•	
LCS (If appropriate to method)	One per matrix per batch (of ≤ 20 samples) %R between 80-120%	Method <sup>(2)</sup>	J (pos)/R (ND) if %R <50% J (pos)/UJ (ND) if %R 50% - 79% J (pos) if %R > 120%	10 (H,L) <sup>3</sup>	Qualify all samples in batch QAPP may have overriding accuracy limits.
Reference Material (RM, SRM, or CRM)	Result $\pm 20\%$ of the 95% confidence interval of the true value for analytes	EcoChem standard policy	J (pos)/UJ (ND) if < LCL J (pos) if > UCL	12 (H,L) <sup>3</sup>	QAPP may have overriding accuracy limits. Some manufacturers may have different RM control limits

#### **Conventional Methods by Gravimetric Analysis** (i.e., Total Solids, Total Dissolved Solids, Total Suspended Solids, Grain Size) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	EcoChem Acceptance Criteria	Source of Criteria	EcoChem Action for Non- Conformance	Reason Code	Discussion and Comments
Laboratory Duplicate	One per matrix per batch (of $\leq 20$ samples) RPD $\leq 20\%$ for results $\geq 5x$ RL Solids: difference $< 2X$ RL for results $< 5X$ RL Aqueous: difference $< 1X$ RL for results $< 5X$ RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if RPD > 20% For Grain Size, no action if results for fraction are less than 5%	9	Qualify all samples in batch, except Grain Size - qualify parent only. QAPP may have overriding precision limits.
Field Duplicate	Solids: RPD <50% (for results $\ge$ 5x RL) OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% (for results $\ge$ 5x RL) OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Qualify only parent and field duplicate samples J (pos)/UJ (ND)	9	QAPP may have overriding precision limits. Client/QAPP may not require qualification based on field precision.
Compound Quantitati	on	•			
Dilutions, Re-extractions and/or Reanalyses		EcoChem standard policy	Use "DNR" to flag results that will not be reported.	11	

<sup>1</sup> National Functional Guidelines for Inorganic Superfund Data Review, January 2010.
 <sup>2</sup> SW846 or EPA Standard Methods

(pos): Positive Result (ND): Not Detected

<sup>3</sup> "H" = high bias indicated; "L" = low bias indicated

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Cooler temperature: 4°C±2°C Preservation: Analyte/Method Specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if preservation requirements not met	1	Use <b>PJ</b> to qualify for cooler temp outliers.
Holding Time	Analyte/Method Specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if holding time exceeded	1	
Instrument Performa	nce	•			
Initial Calibration (ICAL)	blank + multiple standards as per method requirements r ≥ 0.995	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) for r < 0.995	5A	
Initial Calibration Verification (ICV)	Independent source analyzed immediately after calibration %R method specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < lower control limit (LCL) J (pos) if %R > upper control limit (UCL)	5A (H,L) <sup>3</sup>	Qualify all samples in run
Continuing Calibration Verification (CCV)	Immediately following ICV, every 10 samples, and end of run %R method specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if %R < LCL J(pos) if %R > UCL	5B (H,L) <sup>3</sup>	Qualify samples bracketed by CCV outliers
Blank Contamination		•	•		
Method Blank (MB)	One per matrix per batch of (of $\leq$ 20 samples) Blank conc < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U (pos) if result is < 5X method blank concentration	7	Refer to <b>TM-02</b> for additional information. Blank Evaluation based on NFG 1994

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Instrument Blanks (ICB/CCB)	After each ICV & CCV   blank concentration   < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Action level is 5x absolute value of blank conc. For positive blanks: U (pos) results < action level For negative blanks: J (pos)/UJ (ND) results < action level	Pos Blanks: 7 Neg Blanks: 7L <sup>3</sup>	Use blanks bracketing samples for Qualification Refer to <b>TM-02</b> for additional information. <b>Hierarchy of blank review:</b> <b>#1 - Review MB, qualify as needed</b> <b>#2 - Review IB , qualify as needed</b> <b>#3 - Review FB , qualify as needed</b>
Field Blank (FB)	Blank conc < MDL	EcoChem standard policy	U (pos) if result is < 5x action level, as per analyte.	6	Qualify in associated field samples only. Refer to <b>TM-02</b> for additional information.
Precision and Accura	су				
Laboratory Control Sample (LCS)	One per matrix per batch (of ≤ 20 samples) %R within Method control limits (or Laboratory control limtis if none specified in method)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < LCL J (pos) if %R > UCL	10 (H,L) <sup>3</sup>	Qualify all samples in batch QAPP may have overriding accuracy limits.
Reference Materials (RM, CRM, SRM)	Result $\pm 20\%$ of the 95% confidence interval of the true value for analytes	EcoChem standard policy	J (pos)/UJ (ND) if < LCL J (pos) if > UCL	12 (H,L) <sup>3</sup>	QAPP may have overriding accuracy limits. Some manufacturers may have different RM control limits

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Where applicable to method; MSD may not be required One per matrix per batch (of ≤ 20 samples) For samples <4x spike level, %R within method control limits (or Laboratory control limits if none specified in method)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < LCL J (pos) if %R > UCL	8 (H,L)3	Qualify all samples in batch No action if native analyte concentration ≥ 4x spike added. Qualify all samples in batch. QAPP may have overriding accuracy limits.
Laboratory Duplicate (or MS/MSD)	One per matrix per batch (of ≤ 20 samples) RPD ≤ 20% for results ≥ 5x RL Solids: difference < 2X RL for results < 5X RL Aqueous: difference < 1X RL for results < 5X RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if RPD > 20% or if difference > control limit	9	Qualify all samples in batch. QAPP may have overriding precision limits.
Field Duplicate	Solids: RPD <50% (for results ≥ 5x RL) OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% (for results ≥ 5x RL) OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Qualify only parent and field duplicate samples J (pos)/UJ (ND)	9	QAPP may have overriding precision limits. Client/QAPP may not require qualification based on field precision.
Compound Quantita	tion				
Linear Range	inear Range Sample concentrations less than highest calibration standard		If result exceeds linear range & sample was not diluted J (pos)	20	
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	EcoChem standard policy	Use "DNR" to flag results that will not be reported.	11	TM-04 EcoChem Policy for Rejection/Selection Process for Multiple Results

<sup>1</sup> National Functional Guidelines for Inorganic Superfund Data Review, January 2010.

<sup>2</sup> SW846 or EPA Standard Methods

<sup>3</sup> "H" = high bias indicated; "L" = low bias indicated

(pos): Positive Result (ND): Not Detected



## **APPENDIX B**

# **QUALIFIED DATA SUMMARY TABLE**

## Qualified Data Summary Table San Jacinto River Waste Pits Sediments 2016

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
E1600726	SD0110	E1600726-001	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.476	ng/kg	JK	U	25
E1600726	SD0110	E1600726-001	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.733	ng/kg	JK	U	25
E1600726	SD0110	E1600726-001	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.423	ng/kg	JK	U	25
E1600726	SD0110	E1600726-001	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	1.6	ng/kg	К	U	25
E1600726	SD0110	E1600726-001	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.746	ng/kg	JK	U	25
E1600726	SD0111	E1600726-002	EPA1613B	Octachlorodibenzofuran	9.94	ng/kg	К	U	25
E1600726	SD0116	E1600726-003	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.481	ng/kg	JK	U	25
E1600726	SD0116	E1600726-003	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.458	ng/kg	JK	U	25
E1600726	SD0116	E1600726-003	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.574	ng/kg	JK	U	25
E1600726	SD0116	E1600726-003	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	0.401	ng/kg	JK	U	25
E1600726	FW0102	E1600726-004	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	5.1	pg	BJK	U	25
E1600726	FW0102	E1600726-004	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	27.8	pg	В	U	7
E1600726	FW0102	E1600726-004	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	1.11	pg	JK	U	25
E1600726	FW0102	E1600726-004	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.06	pg	JK	U	25
E1600726	FW0102	E1600726-004	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.897	pg	J	U	6
E1600726	FW0102	E1600726-004	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.44	pg	JK	U	25
E1600726	FW0102	E1600726-004	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	1.17	pg	JK	U	25
E1600726	FW0102	E1600726-004	EPA1613B	Octachlorodibenzofuran	25	pg	J	U	6
E1600726	FW0102	E1600726-004	EPA1613B	Octachlorodibenzo-p-dioxin	126	pg	В	U	6
E1600726	FB0101	E1600726-005	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	5.76	pg	BJ	U	7
E1600726	FB0101	E1600726-005	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	29.2	pg	В	U	7
E1600726	FB0101	E1600726-005	EPA1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.83	pg	JK	U	25
E1600726	FB0101	E1600726-005	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	1.1	pg	JK	U	25
E1600726	FB0101	E1600726-005	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	1.7	pg	JK	U	25
E1600726	FB0101	E1600726-005	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.839	pg	JK	U	25
E1600726	FB0101	E1600726-005	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	1.11	pg	JK	U	25
E1600726	FB0101	E1600726-005	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.433	pg	JK	U	25
K1608142	SD0116	K1608142-003	PSEP	Fine Sand	11.48	percent		J	9
K1608142	SD0116	K1608142-003	PSEP	Gravel	24.36	percent		J	9
K1608142	SD0116	K1608142-003	PSEP	Silt	29.43	percent		J	9
K1608142	SD0116	K1608142-003DUP	PSEP	Fine Sand	12.01	percent		J	9

## Qualified Data Summary Table San Jacinto River Waste Pits Sediments 2016

SDG San	Samula ID	Lab Sample ID	Method	Analyta	Result	Units Lab Flag	DV	DV	
	Sample ID	Lab Sample ID	Method	Analyte	Result		Lab Flag	Qualifier	Reason
K1608142	SD0116	K1608142-003DUP	PSEP	Gravel	16.65	percent		J	9
K1608142	SD0116	K1608142-003DUP	PSEP	Silt	29.94	percent		J	9
K1608142	SD0116	K1608142-003TRP	PSEP	Fine Sand	7.95	percent		J	9
K1608142	SD0116	K1608142-003TRP	PSEP	Gravel	67.74	percent		J	9
K1608142	SD0116	K1608142-003TRP	PSEP	Silt	20.25	percent		J	9



## DATA VALIDATION REPORT

## SAN JACINTO RIVER WASTE PITS 2016 TISSUE SAMPLING

#### **Prepared for:**

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September 12, 2016

**Approved for Release:** 

n D. Bodkin

Alison Bodkin Project Chemist EcoChem, Inc.

## PROJECT NARRATIVE

#### **Basis for Data Validation**

This report summarizes the results of summary and full validation (EPA Stage 2B & 4) performed on tissue sample data and quality control sample data for the San Jacinto River 2016 Tissue Sampling Study. A complete list of samples is provided in the **Sample Index**.

Samples were analyzed by ALS Environmental, Houston, Texas. The analytical methods and EcoChem project chemists are listed below.

ANALYSIS	METHOD	PRIMARY REVIEW	SECONDARY REVIEW
Dioxin/Furan Compounds	1613B	M. Swanson	C. Frans

The data were reviewed using guidance and quality control criteria documented in the analytical methods and the following project and guidance documents:

- Sampling and Analysis Plan: Tissue Study, San Jacinto River Waste Pits Superfund Site (Integral, September 2010);
- Draft Addendum 2 (to the Tissue Sampling and Analysis Plan) For Additional Gulf Killifish Tissue Sampling, San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, March 2016).
- USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review (USEPA 2011).

EcoChem's goal in assigning data assessment qualifiers is to assist in proper data interpretation. If values are estimated (J or UJ), data may be used for site evaluation and risk assessment purposes but reasons for data qualification should be taken into consideration when interpreting sample concentrations. If values are assigned an R, the data are to be rejected and should not be used for any site evaluation purposes. If values have no data qualifier assigned, then the data meet the data quality objectives as stated in the documents and methods referenced above.

Data qualifier definitions, reason codes, and validation criteria are included as **APPENDIX A**. A Qualified Data Summary Table is included in **APPENDIX B**. Data Validation Worksheets and project associated communications will be kept on file at EcoChem, Inc. A qualified laboratory electronic data deliverable (EDD) is also submitted with this report.

## Qualified Data Summary Table San Jacinto River Waste Pits Tissues 2016

SDG	Sample ID	Laboratory ID	Dioxins
E1600395	TS0185	E1600395-001	$\checkmark$
E1600395	TS0186	E1600395-002	$\checkmark$
E1600395	TS0187	E1600395-003	$\checkmark$
E1600395	TS0188	E1600395-004	$\checkmark$
E1600797	TS0184	E1600797-006	$\checkmark$
E1600798	TS0181	E1600798-007	$\checkmark$
E1600799	TS0182	E1600799-007	$\checkmark$
E1600800	TS0183	E1600800-004	$\checkmark$

## DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 Tissue Sampling Dioxin/Furan Compounds by EPA 1613B

This report documents the review of analytical data from the analyses of tissue samples and the associated laboratory quality control (QC) samples. ALS Environmental, Houston, Texas, analyzed the samples. Refer to the **Sample Index** for a complete list of samples.

SDG	Number of Samples	VALIDATION LEVEL
E1600395	4 Tissue	EPA Stage 4
E1600797	1 Tissue	EPA Stage 2B
E1600798	1 Tissue	EPA Stage 2B
E1600799	1 Tissue	EPA Stage 2B
E1600800	1 Tissue	EPA Stage 2B

#### DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

#### EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package. No errors were noted.

#### **TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

1	Sample Receipt, Preservation, and Holding Times	$\checkmark$	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)
$\checkmark$	System Performance and Resolution Checks	1	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
$\checkmark$	Initial Calibration (ICAL)	1	Field Replicate Samples
$\checkmark$	Calibration Verification	$\checkmark$	Target Analyte List
1	Laboratory Blanks	>	Reported Results
1	Field Blanks	2	Compound Identification
$\checkmark$	Labeled Compound Recovery	1	Calculation Verification

✓ Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

#### Sample Receipt, Preservation, and Holding Times

As stated in validation guidance documents, sample coolers should arrive at the laboratory within the advisory temperature range of 2°C to 6°C. Several cooler temperatures were less than the lower control limit, the lowest at -55°C, on dry ice. One sample cooler was delivered to the laboratory at the end of the day they were collected. The coolers had insufficient time to cool to 6°C. Dioxin compounds have been found to remain stable at a wide range of temperatures. These temperature outliers did not impact data quality; therefore, no data were qualified.

#### **Laboratory Blanks**

To assess the impact of any blank contaminant on the reported sample results, an action level was established at five times (5x) the concentration reported in the blank. If a contaminant was reported in an associated field sample and the concentration was less than the action level, the result was qualified as not detected (U-7). No action was taken if the sample result was greater than the action level or for non-detected results. The laboratory assigned K-flags to values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are "estimated maximum possible concentrations". When these occurred in the method blank the results were considered as false positives. No action levels were established for these analytes.

A method blank was analyzed at the appropriate frequency. The only positive results reported did not meet ion abundance ratio criteria and were assigned K-flags by the laboratory. No data were qualified due to method blank contamination.

#### Field Blanks

No field blanks were submitted for this project.

#### Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) samples are not required by the method and were not analyzed. Accuracy and precision were evaluated using the labeled compound and laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results.

#### **Field Replicates**

No field replicates were submitted for this project.

#### **Compound Identification**

The laboratory assigned K-flags to results where a peak was detected but did not meet ion ratio quantitation criteria. The reported values cannot be considered as positive identifications for these analytes. These results were considered potential false positives or estimated maximum possible concentrations (EMPC) and were qualified as not detected (U-25) at the reported values.

The method requires the confirmation of 2,3,7,8-TCDF using an alternate GC column as the DB5 column that is typically used cannot fully separate 2,3,7,8-TCDF from closely eluting non-target TCDF isomers. The laboratory did not perform a second column confirmation; however, the laboratory uses a DB-5MSUI column. This column provides adequate resolution of the TCDF isomers as indicated by the acceptable peak to valley ratios. Because the 2,3,7,8-TCDF resolution was acceptable, no action was necessary.

*SDG E1600395*: The result for 1,2,3,4,7,8-HxCDF in Sample TS0188 was flagged "P" by the laboratory to indicate chlorodiphenyl ether interference. This result was estimated (J-23H), to indicate a potential high bias.

#### **Calculation Verification**

*SDG E1600395:* Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

#### **OVERALL ASSESSMENT**

As determined by this evaluation, the laboratory followed the specified analytical method. Accuracy was acceptable as demonstrated by labeled compound and LCS/LCSD %R values. Precision was acceptable as demonstrated by the LCS/LCSD RPD values.

Detection limits were elevated due to ion abundance ratio outliers. One result was estimated due to chlorodiphenyl ether interferences.

All data, as qualified, are acceptable for use.



# **APPENDIX A**

# DATA QUALIFIER DEFINITIONS REASON CODES AND CRITERIA TABLES

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## DATA VALIDATION QUALIFIER CODES Based on National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents the approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

The following is an EcoChem qualifier that may also be assigned during the data review process:

DNR Do not report; a more appropriate result is reported from another analysis or dilution.

## DATA QUALIFIER REASON CODES

Group	Code	Reason for Qualification
Sample Handling	1	Improper Sample Handling or Sample Preservation (i.e., headspace, cooler temperature, pH, summa canister pressure); Exceeded Holding Times
	24	Instrument Performance (i.e., tune, resolution, retention time window, endrin breakdown, lock-mass)
	5A	Initial Calibration (RF, %RSD, r <sup>2</sup> )
Instrument Performance	5B	Calibration Verification (CCV, CCAL; RF, %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	5C	Initial Calibration Verification (ICV %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	6	Field Blank Contamination (Equipment Rinsate, Trip Blank, etc.)
Blank Contamination	7	Lab Blank Contamination (i.e., method blank, instrument blank, etc.) Use low bias flag (L) <sup>1</sup> for negative instrument blanks
	8	Matrix Spike (MS and/or MSD) Recoveries Use bias flags (H,L) <sup>1</sup> where appropriate
	9	Precision (all replicates: LCS/LCSD, MS/MSD, Lab Replicate, Field Replicate)
Precision and Accuracy	10	Laboratory Control Sample Recoveries (a.k.a. Blank Spikes) Use bias flags (H,L) <sup>1</sup> where appropriate
	12	Reference Material Use bias flags (H,L) <sup>1</sup> where appropriate
	13	Surrogate Spike Recoveries (a.k.a. labeled compounds, recovery standards) Use bias flags (H,L) <sup>1</sup> where appropriate
	16	ICP/ICP-MS Serial Dilution Percent Difference
	17	ICP/ICP-MS Interference Check Standard Recovery Use bias flags (H,L) <sup>1</sup> where appropriate
Interferences	19	Internal Standard Performance (i.e., area, retention time, recovery)
	22	Elevated Detection Limit due to Interference (i.e., chemical and/or matrix)
	23	Bias from Matrix Interference (i.e. diphenyl ether, PCB/pesticides)
	2	Chromatographic pattern in sample does not match pattern of calibration standard
	3	2 <sup>nd</sup> column confirmation (RPD or %D)
Identification and Quantitation	4	Tentatively Identified Compound (TIC) (associated with NJ only)
	20	Calibration Range or Linear Range Exceeded
	25	Compound Identification (i.e., ion ratio, retention time, relative abundance, etc.)
N.C II	11	A more appropriate result is reported (multiple reported analyses i.e., dilutions, re- extractions, etc. Associated with "R" and "DNR" only)
Miscellaneous	14	Other (See DV report for details)
	26	Method QC information not provided

<sup>1</sup>H = high bias indicated

L = low bias indicated

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Waters/Solids $\leq 6^{\circ}C \&$ in the dark Tissues <-10°C & in the dark <b>Preservation Aqueous:</b> If Cl <sub>2</sub> is present Thiosulfate must be added and if pH > 9 it must be adjusted to 7 - 9	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/R(ND) if thiosulfate not added if Cl <sub>2</sub> present; J(pos)/UJ(ND) if pH not adjusted J(pos)/UJ(ND) if temp > 20°C	1	EcoChem PJ, see TM-05
Holding Time	If properly stored, 1 year or: Extraction (all matrices): 30 days from collection Analysis (all matrices): 45 days from extraction	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If not properly stored or HT exceedance: J(pos)/UJ(ND)	1	EcoChem PJ, see TM-05 Gross exceedance = > 1 year 2011 NFG Note: Under CWA, SDWA, and RCRA the HT for H2O is 7 days.
Instrument Performa	nce				
Mass Resolution (Tuning)	PFK (Perfluorokerosene) ≥10,000 resolving power at m/z 304.9824. Exact mass of m/z 380.9760 w/in 5 ppm of theoretical value (380.97410 to 380.97790) . Analyzed prior to ICAL and at the start and end of each 12 hr. shift.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	R(pos/ND) all analytes in all samples associated with the tune	24	Notify PM
Windows Defining Mix	Peaks for first and last eluters must be within established retention time windows for each selector group (chlorination level)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If peaks are not completely within windows (clipped): If natives are ok, J(pos)/UJ(ND) homologs (Totals) If natives are affected, R all results for that selector group	24	Notify PM
Column Performance Mix	Both mixes must be analyzed before ICAL and CCAL Valley < 25% (valley = (x/y)*100%) where x = ht. of TCDD (or TCDF) & y = baseline to bottom of valley For all isomers eluting near the 2378-TCDD (TCDF) peak (TCDD only for 8290)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if valley > 25%	24	EcoChem PJ, see TM-05, Rev. 2; Note: TCDF is evaluated only if second column confirmation is performed
Initial Calibration Sensitivity	S/N ratio > 10 for all native and labeled compounds in CS1 std.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5A	
Initial Calibration <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If 2 or more ion ratios are out for one compound in ICAL, J(pos)	5A	EcoChem PJ, see TM-05, Rev. 2

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Instrument Performa	nce (continued)				
Initial Calibration (Minimum 5 stds.)	%RSD < 20% for native compounds %RSD <30% for labeled compounds (%RSD < 35% for labeled compounds under 1613b)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) natives if %RSD > 20%	5A	
Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD >25 min on DB5 & >15 min on DB-225	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action		EcoChem PJ, see TM-05, Rev. 2
Continuing Calibration (Prior to each 12 hr. shift) <b>Sensitivity</b>	S/N ratio for CS3 standard > 10	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5B	
Continuing Calibration (Prior to each 12 hr. shift) <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	For congener with ion ratio outlier, J(pos) natives in all samples associated with CCAL. No action for labeled congener ion ratio outliers.	25	EcoChem PJ, see TM-05
Continuing Calibration (Prior to each 12 hr.	%D+/-20% for native compounds %D +/-30% for labeled compounds (Must meet limits in Table 6, Method 1613B) If %D in the closing CCAL are within 25%/35%, the mean RF from the two CCAL may be used to calculate samples (Section 8.3.2.4 of 8290).	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Labeled compounds: Narrate, no action. Native compounds: 1613: J(pos)/UJ(ND)if %D is outside Table 6 limits J(pos)/R(ND) if %D is +/-75% of Table 6 limits 8290: J(pos)/UJ(ND) if %D = 20% - 75% J(pos)/R(ND) if %D > 75%	5B (H,L) <sup>3</sup>	
shift) Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD and <sup>13</sup> C <sub>12</sub> -123789-HxCDD should be ± 15 seconds of ICAL RRT for all other compounds must meet criteria listed in Table 2 Method 1316.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action	5B	EcoChem PJ, see TM-05
Blank Contamination					
Method Blank (MB)	MB: One per matrix per batch of (of ≤ 20 samples) No detected compounds > RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U(pos) if result is < 5X action level.	7	Hierarchy of blank review: #1 - Review MB, qualify as needed
Field Blank (FB)	FB: frequency as per QAPP No detected compounds > RL	WELTOU	U(pos) if result is < 5X action level.	6	#2 - Review FB , qualify as needed

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Precision and Accura	cy				
MS/MSD (recovery)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of $\leq$ 20 samples)	EcoChem standard policy	J(pos) if both %R > UCL - high bias J(pos)/UJ(ND) if both %R < LCL - low bias m standard policy J(pos)/R(ND) if both %R < 10% - very low bias	8 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria. No action if parent concentration is >4x the amount spiked.
	Use most current laboratory control limits		J(pos)/UJ(ND) if one > UCL & one < LCL, with no bias <b>PJ if only one %R outlier</b>		Qualify parent sample only unless other QC indicates systematic problems.
MS/MSD (RPD)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) in parent sample if RPD > CL	9	Qualify parent sample only.
LCS (or OPR)	One per lab batch (of ≤ 20 samples) Use most current laboratory control limits <b>or</b> Limits from Table 6 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	10 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria, when LCSD is analyzed. Qualify all associated samples.
LCS/LCSD (RPD)	LCSD not typically required for HRMS analyses. One set per matrix and batch of 20 samples RPD < 35%	Method <sup>(2)</sup> Ecochem standard policy	J(pos) assoc. compound in all samples if RPD > CL	9	Qualify all associated samples.
Lab Duplicate (RPD)	Lab Dup not typically required for HRMS analyses. One per lab batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos)/UJ(ND) if RPD > CL	9	
Labeled Compounds (Internal Standards)	Added to all samples %R = 40% - 135% in all samples 8290 %R must meet limits in Table 7 Method 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	13 (H,L) <sup>3</sup>	
Field Duplicates	Solids: RPD <50% OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Narrate and qualify if required by project	9	Use professional judgment

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason	Discussion and Comments
Compound ID and Ca	•			Code	
Quantitation/ Identification	All ions for each isomer must maximize within ± 2 seconds. S/N ratio >2.5 Ion ratios must meet criteria listed in Table 8 Method 8290, or Table 9 of 1613B; RRTs w/in limits in Table 2 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate in report; qualify if necessary NJ(pos) for retention time outliers. U(pos) for ion ratio outliers.	25	EcoChem PJ, see TM-05
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If laboratory correctly reported an EMPC value, qualify the native compound U(pos) to indicate that the value is a detection limit and qualify total homolog groups J (pos)	25	Use professional judgment See TM-18
Interferences	Interferences from chlorodiphenyl ether compounds	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if present	23	See TM-16
Interferences	Lock masses must not deviate ± 20% from values in Table 8 of 1613B	Method (2)	J(pos)/UJ(ND) if present	24	See TM-17
Second Column Confirmation	All 2,3,7,8-TCDF hits must be confirmed on a DB-225 (or equiv) column. All QC criteria must also be met for the confirmation analysis.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Report the DB-225 value. If not performed use PJ.	3	DNR-11 DB5 result if both results from both columns are reported. <b>EcoChem PJ, see TM-05</b>
Calculation Check	Check 10% of field & QC sample results	EcoChem standard policy	Contact laboratory for resolution and/or corrective action	na	Full data validation only.
Electronic Data Delive	erable (EDD)				
Verification of EDD to hardcopy data	EcoChem verify @ 10% unless problems noted; then increase level up to 100% for next several packages.		Depending on scope of problem, correct at EcoChem (minor issues) to resubmittal by laboratory (major issues).	na	EcoChem Project Manager and/or Database Administrator will work with lab to provide long-term corrective action.
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	Standard reporting policy	Use "DNR" to flag results that will not be reported.	11	

(pos) - positive (detected) results; (ND) - not detected results

<sup>1</sup> National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) & Chlorinated Dibenzofurans (CDFs) Data Review, September 2011

<sup>2</sup> Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), USEPA SW-846, Method 8290

<sup>2</sup> EPA Method 1613, Rev.B, Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGS/HRMS, October 1994

<sup>3</sup> NFG 2013 suggests using "+ / -" to indicate bias; EcoChem has chosen "H" = high bias indicated; "L" = low bias indicated.



## **APPENDIX B**

# **QUALIFIED DATA SUMMARY TABLE**

## Qualified Data Summary Table San Jacinto River Waste Pits Tissues 2016

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV	DV
300	Sample ID		Wethou		Result	Units	Lab Hay	Qualifier	Reason
E1600395	TS0185	E1600395-001	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.139	ng/kg	JK	U	25
E1600395	TS0185	E1600395-001	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.174	ng/kg	BJK	U	25
E1600395	TS0185	E1600395-001	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.0661	ng/kg	JK	U	25
E1600395	TS0186	E1600395-002	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	0.873	ng/kg	JK	U	25
E1600395	TS0186	E1600395-002	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.937	ng/kg	К	U	25
E1600395	TS0187	E1600395-003	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0996	ng/kg	JK	U	25
E1600395	TS0187	E1600395-003	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.129	ng/kg	BJK	U	25
E1600395	TS0187	E1600395-003	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	1.03	ng/kg	К	U	25
E1600395	TS0188	E1600395-004	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	1.62	ng/kg	JP	J	23H
E1600395	TS0188	E1600395-004	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.573	ng/kg	JK	U	25
E1600395	TS0188	E1600395-004	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	1.21	ng/kg	К	U	25
E1600797	TS0184	E1600797-006	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.69	ng/kg	JK	U	25
E1600797	TS0184	E1600797-006	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.396	ng/kg	BJK	U	25
E1600797	TS0184	E1600797-006	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	0.244	ng/kg	JK	U	25
E1600798	TS0181	E1600798-007	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.369	ng/kg	JK	U	25
E1600798	TS0181	E1600798-007	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.271	ng/kg	BJK	U	25
E1600798	TS0181	E1600798-007	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	0.105	ng/kg	JK	U	25
E1600799	TS0182	E1600799-007	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.123	ng/kg	JK	U	25
E1600800	TS0183	E1600800-004	EPA1613B	Octachlorodibenzofuran	1.07	ng/kg	JK	U	25



## DATA VALIDATION REPORT

## SAN JACINTO RIVER WASTE PITS 2016 SPME FIBER SAMPLING

#### **Prepared for:**

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EcoChem Project: C22130-22

September 12, 2016

**Approved for Release:** 

ia J. Bodki

Alison Bodkin Project Chemist EcoChem, Inc.

## **PROJECT NARRATIVE**

#### **Basis for Data Validation**

This report summarizes the results of validation performed on groundwater and pore water solidphase microextraction (SPME) fibers and quality control sample data for the San Jacinto River 2016 GW & PW SPME Sampling Study. The first data packages received an EPA Stage 4 validation. QC samples (SPME blanks, caulk blanks, solvent rinses) received an EPA Stage 2A validation. All other packages received a Stage 2B validation. A complete list of samples is provided in the **Sample Index**.

Samples were analyzed by ALS Environmental, Houston, Texas. The analytical methods and EcoChem project chemists are listed below.

Analysis	Method	PRIMARY REVIEW	SECONDARY REVIEW
Dioxin/Furan Compounds	1613B	M. Swanson	A. Bodkin

The data were reviewed using guidance and quality control criteria documented in the analytical methods and the following project and guidance documents:

- Sampling and Analysis Plan: TCRA Cap Porewater Assessment San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, May 2012).
- Addendum 1 (to the Sampling and Analysis Plan (SAP) TCRA Cap Porewater Assessment) *Additional Assessment of Porewater within the TCRA Armored Cap, San Jacinto River Waste Pits Superfund Site* (Integral/Anchor QEA, February 2016).
- Final Groundwater Study Sampling and Analysis Plan, San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, January 2011).
- Final Addendum 3 Groundwater Study Sampling and Analysis Plan, San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, March 2016).
- USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review (USEPA 2011).

EcoChem's goal in assigning data assessment qualifiers is to assist in proper data interpretation. If values are estimated (J or UJ), data may be used for site evaluation and risk assessment purposes but reasons for data qualification should be taken into consideration when interpreting sample concentrations. If values are assigned an R, the data are to be rejected and should not be used for any site evaluation purposes. If values have no data qualifier assigned, then the data meet the data quality objectives as stated in the documents and methods referenced above.

Data qualifier definitions, reason codes, and validation criteria are included as **APPENDIX A**. A Qualified Data Summary Table is included in **APPENDIX B**. Data Validation Worksheets and project associated communications will be kept on file at EcoChem, Inc. A qualified laboratory electronic data deliverable (EDD) is also submitted with this report.

## Qualified Data Summary Table San Jacinto River Waste Pits GW - PW SPME 2016

SDG	Sample ID	Laboratory ID	Dioxins
E1600282	04052016SJPW10	E1600282-006	$\checkmark$
E1600308	04132016-SJGW200	E1600308-001	$\checkmark$
E1600326	03162016SJGW1	E1600326-001	$\checkmark$
E1600326	04072016SJGW1	E1600326-002	$\checkmark$
E1600326	04072016SJGW2	E1600326-003	$\checkmark$
E1600326	04072016SJGW10	E1600326-004	$\checkmark$
E1600326	04072016SJGW11	E1600326-005	$\checkmark$
E1600326	04072016SJGW12	E1600326-006	$\checkmark$
E1600326	04072016SJGW13	E1600326-007	$\checkmark$
E1600326	04072016SJGW14	E1600326-008	$\checkmark$
E1600326	04072016SJGW15	E1600326-009	$\checkmark$
E1600426	05132016-SJGW300	E1600426-001	$\checkmark$
E1600426	05132016-SJGW301	E1600426-002	$\checkmark$
E1600426	05132016-SJGW302	E1600426-003	$\checkmark$
E1600426	05132016-SJGW303	E1600426-004	$\checkmark$
E1600426	05152013-SJPW201	E1600426-005	$\checkmark$
E1600426	05152016-SJPW202	E1600426-006	$\checkmark$
E1600426	05152016-SJPW203	E1600426-007	$\checkmark$
E1600426	05152016-SJPW204	E1600426-008	$\checkmark$
E1600426	05152016-SJPW205	E1600426-009	$\checkmark$
E1600426	05152016-SJPW206	E1600426-010	$\checkmark$
E1600448	05182016_SJPWDP	E1600448-001	$\checkmark$
E1600542	06142016_SSGWRB	E1600542-001	$\checkmark$
E1600542	SJMW003 GW1015	E1600542-002	$\checkmark$
E1600542	SJMW004 SGW1217	E1600542-003	$\checkmark$
E1600542	SJMW005 GW1318	E1600542-004	$\checkmark$
E1600542	SJMW006 GW1015	E1600542-005	$\checkmark$
E1600542	SJMW007 GW0712	E1600542-006	$\checkmark$
E1600542	SJMW008 GW2025	E1600542-007	$\checkmark$
E1600542	SJMW004D GW77.582.5	E1600542-008	$\checkmark$
E1600542	SJMW1004 GW77.582.5	E1600542-009	$\checkmark$
E1600542	SJMW002 GW07.512.5	E1600542-010	$\checkmark$
E1600542	SJMW001 GW09.514.5	E1600542-011	$\checkmark$
E1600542	SJMW010 GW0510	E1600542-012	$\checkmark$
E1600542	SJMW009 GW19.524.5	E1600542-013	$\checkmark$
E1600542	SJMW011 GW50.555.5	E1600542-014	$\checkmark$
E1600542	SJMW012 GW09.814.8	E1600542-015	$\checkmark$
E1600542	SJMW1012 GW09.814.8	E1600542-016	$\checkmark$
E1600554	SJCPRI-PW-2-A	E1600554-001	$\checkmark$

## Qualified Data Summary Table San Jacinto River Waste Pits GW - PW SPME 2016

SDG	Sample ID	Laboratory ID	Dioxins
E1600554	SJCPRI-PW-2-B	E1600554-002	$\checkmark$
E1600554	SJCPRI-PW-2-C	E1600554-003	$\checkmark$
E1600738	SJCP001SP1ADUP	E1600738-001	$\checkmark$
E1600738	SJCP001SP1BDUP	E1600738-002	$\checkmark$
E1600738	SJCP001SP1CDUP	E1600738-003	$\checkmark$
E1600738	SJCP001SP1A	E1600738-004	$\checkmark$
E1600738	SJCP001SP1B	E1600738-005	$\checkmark$
E1600738	SJCP001SP1C	E1600738-006	$\checkmark$
E1600738	SJCP013SP1A	E1600738-007	$\checkmark$
E1600738	SJCP013SP1B	E1600738-008	$\checkmark$
E1600738	SJCP013SP1C	E1600738-009	$\checkmark$
E1600739	SJMW013GW50.355.3	E1600739-001	$\checkmark$
E1600740	SJCP011SP1A	E1600740-007	$\checkmark$
E1600740	SJCP011SP1B	E1600740-008	$\checkmark$
E1600740	SJCP011SP1C	E1600740-009	$\checkmark$
E1600740	07202016-SJPWRB	E1600740-013	$\checkmark$
E1600740	SJCP014SP1A	E1600740-014	$\checkmark$
E1600740	SJCP014SP1B	E1600740-015	$\checkmark$
E1600740	SJCP014SP1C	E1600740-016	$\checkmark$
E1600740	SJCP008SP1AW	E1600740-017	$\checkmark$
E1600741	SJCP008SP1A	E1600741-001	$\checkmark$
E1600741	SJCP008SP1B	E1600741-002	$\checkmark$
E1600741	SJCP008SP1C	E1600741-003	$\checkmark$
E1600745	SJCP006SP1A	E1600745-001	$\checkmark$
E1600745	SJCP006SP1B	E1600745-002	$\checkmark$
E1600745	SJCP006SP1C	E1600745-003	$\checkmark$
E1600745	SJCP009SP1ADUP	E1600745-004	$\checkmark$
E1600745	SJCP009SP1BDUP	E1600745-005	$\checkmark$
E1600745	SJCP009SP1CDUP	E1600745-006	$\checkmark$
E1600745	SJCP007SP1A	E1600745-007	$\checkmark$
E1600745	SJCP007SP1B	E1600745-008	$\checkmark$
E1600745	SJCP007SP1C	E1600745-009	$\checkmark$
E1600745	SJCR002SP2A	E1600745-010	$\checkmark$
E1600745	SJCR002SP2B	E1600745-011	$\checkmark$
E1600745	SJCR002SP2C	E1600745-012	$\checkmark$
E1600745	SJCP004SP1A	E1600745-013	$\checkmark$
E1600745	SJCP004SP1B	E1600745-014	$\checkmark$
E1600745	SJCP004SP1C	E1600745-015	$\checkmark$
E1600745	SJCP012SP1A	E1600745-016	$\checkmark$

## Qualified Data Summary Table San Jacinto River Waste Pits GW - PW SPME 2016

SDG	Sample ID	Laboratory ID	Dioxins
E1600745	SJCP012SP1B	E1600745-017	$\checkmark$
E1600745	SJCP012SP1C	E1600745-018	$\checkmark$
E1600746	SJCR003SP2A	E1600746-001	$\checkmark$
E1600746	SJCR003SP2B	E1600746-002	$\checkmark$
E1600746	SJCR003SP2C	E1600746-003	$\checkmark$
E1600746	SJCP009SP1A	E1600746-004	$\checkmark$
E1600746	SJCP009SP1B	E1600746-005	$\checkmark$
E1600746	SJCP009SP1C	E1600746-006	$\checkmark$
E1600746	SJCR002SP2AW	E1600746-007	$\checkmark$
E1600746	SJCP005SP1A	E1600746-008	$\checkmark$
E1600746	SJCP005SP1B	E1600746-009	$\checkmark$
E1600746	SJCP005SP1C	E1600746-010	$\checkmark$

# DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 SPME Fiber Sampling Dioxin/Furan Compounds by EPA 1613B

This report documents the review of analytical data from the analyses of groundwater and porewater SPME samples and the associated laboratory quality control (QC) samples. ALS Environmental, Houston, Texas, analyzed the samples. Refer to the **Sample Index** for a complete list of samples.

SDG	Number of Samples	VALIDATION LEVEL
E1600282	1 Groundwater SPME Fiber Blank	EPA Stage 2A
E1600308	1 Groundwater SPME Fiber Deployment Blank	EPA Stage 2B
E1600326	5 Groundwater SPME 3 Fiber Prep Process Blank 1 SPME Fiber Blank	EPA Stage 4
E1600426	4 Groundwater SPME 6 Porewater SPME	EPA Stage 4
E1600448	1 Porewater SPME	EPA Stage 2B
E1600542	15 Groundwater SPME 1 SPME Fiber Blank	EPA Stage 2B
E1600554	3 Porewater SPME	EPA Stage 2B
E1600738	9 Porewater SPME	EPA Stage 4
E1600739	1 Groundwater SPME	EPA Stage 2B
E1600740	7 Porewater SPME 1 SPME Fiber Blank	EPA Stage 2B
E1600741	3 Porewater SPME	EPA Stage 2B
E1600745	18 Porewater SPME	EPA Stage 2B
E1600746	10 Porewater SPME	EPA Stage 2B

#### DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

# EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package.

*SDG E1600326*: For Sample 04072016SJGW10, the laboratory reported 13C-1,2,3,7,8,9-HxCDF in the EDD. This was not a target compound and was flagged do-not-report (DNR-11) in the EDD.

For Sample 04072016SJGW12, the laboratory reported 13C-2,3,7,8-TCDD, 13C-2,3,7,8-TCDF, 13C-1,2,3,7,8-PeCDF, and 13C-1,2,3,7,8,9-HxCDF in the EDD. These were not target compounds and were flagged do-not-report (DNR-11) in the EDD.

*SDG E1600426*: The chain-of-custody (COC) did not have Performance Reference Compounds (PRC) requested for all the samples in this SDG. As per the client, PRC were supposed to be requested. The laboratory submitted results for PRC which were included in the EDD. See the **TARGET ANALYTE LIST** section for a list of PRC.

Sample 0515201<u>6</u>-SJPW201 was listed on the COC but the laboratory logged this sample in as 0515201<u>3</u>-SJPW201. No action was taken.

Additionally, the laboratory reported PRC 37CI-2,3,7,8-TCDD for Sample 05152013-SJPW201 and PRC 13C-1,2,3,7,8-PeCDF for Sample 05152016-SJPW206. These were not target compounds and were flagged do-not-report (DNR-11) in the EDD.

*SDG E1600448*: The COC indicated that PRC were requested for the sample in this SDG. As per the client, this was an error. Only native target compounds were required for this sample. No action was taken beyond noting this discrepancy.

*SDG E1600554*: Due to a miscommunication, the laboratory spiked every sample and QC sample with an internal standard solution containing porewater PRC 13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF. Therefore, results for these compounds are not useable. These PRC compounds were not included in the EDD. See the TARGET ANALYTE LIST section for a list of PRC.

*SDG E1600738*: For Sample SJCP013SP1C, the result for 2,3,7,8-TCDD was missing from the EDD. The laboratory resubmitted a corrected EDD.

*SDG E1600740*: Samples SJCP010SP1A, SJCP010SP1B, and SJCP010SP1C were placed on hold at the laboratory per client request. The following samples were cancelled by the client due to an error in sample preparation; the samples were spiked with an internal standard solution containing porewater PRC 13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF:

SJCR004SP2A	SJCPR2SP2A
SJCR004SP2B	SJCPR2SP2B
SJCR004SP2C	SJCPR2SP2C

*SDG E1600745:* For Samples SJCP009SP1BDUP, SJCP004SP1B, and SJCP004SP1C, the result for 2,3,7,8-TCDD was missing from the EDD. The laboratory resubmitted a corrected EDD.

# **TECHNICAL DATA VALIDATION**

1	Sample Receipt, Preservation, and Holding Times	2	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)
$\checkmark$	System Performance and Resolution Checks	1	Matrix Spikes/Matrix Spike Duplicates (MS/MSD)
1	Initial Calibration (ICAL)	1	Field Replicate Samples
$\checkmark$	Calibration Verification	1	Target Analyte List
1	Laboratory Blanks	1	Reported Results
1	Field Blanks	2	Compound Identification
2	Labeled Compound Recovery	1	Calculation Verification

The QC requirements that were reviewed are listed below.

✓ Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

# Sample Receipt, Preservation, and Holding Times

As stated in validation guidance documents, sample coolers should arrive at the laboratory within the advisory temperature range of 2°C to 6°C. Several cooler temperatures were greater than the upper control limit, the highest at 24.3°C. Samples were delivered to the laboratory at the end of the day they were collected. The coolers had insufficient time to cool to 6°C. Dioxin compounds have been found to remain stable at a wide range of temperatures. These temperature outliers did not impact data quality; therefore, no data were qualified.

#### **Initial Calibration**

As stated in the method a calibration curve of five (5) calibration points is employed for native congeners. The Performance Reference Compounds (PRC) are specific labeled compounds treated as target analytes. The method lists the same concentration for all labeled congeners across all the calibration standards. The laboratory employed this same treatment to the PRC standards, using a concentration of 50 ng/mL for each of the five (5) standards, resulting in a one-point calibration. No action was taken beyond noting this discrepancy.

#### Laboratory Blanks

To assess the impact of any blank contaminant on the reported sample results, an action level was established at five times (5x) the concentration reported in the blank. If a contaminant was reported in an associated field sample and the concentration was less than the action level, the result was qualified as not detected (U-7). No action was taken if the sample result was greater than the action level or for non-detected results. The laboratory assigned K-flags to values when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are "estimated maximum possible concentrations". When these occurred in the method blank the results were considered as false positives. No action levels were established for these analytes.

Method blanks were analyzed at the appropriate frequency. Various target analytes were detected in the method blanks, however only the results noted below required qualification; all other associated sample results were either not detected or were detected at concentrations greater than the action levels.

Method blanks were also evaluated for contamination of additional target analytes referred to as performance reference compounds (PRC). PRC were labeled compounds that were listed as target compounds and field spiked into some samples. See the **TARGET ANALYTE LIST** section for a list of PRC.

*SDG E1600426*: Due to a miscommunication, the laboratory spiked the method blank associated with groundwaters with PRC 13C-2,3,4,7,8-PeCDF and the method blank associated with porewaters with PRC 13C-2,3,7,8-TCDD, 13C-2,3,7,8-TCDF, and 13C-2,3,4,7,8-PeCDF; therefore, they were detected in these method blanks. No data were qualified based on positive results of these compounds in method blanks. The groundwater PRC 37CI-2,3,7,8-TCDD and 13C-1,2,3,4-TCDF were not detected in the method blank associated with groundwaters.

*SDG E1600554*: Due to a miscommunication, the laboratory spiked every sample and QC sample with an internal standard solution containing pore water PRC 13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF; therefore, they were detected in the method blank. No data were qualified based on positive results of these compounds in method blanks.

*SDG E1600745:* Porewater PRC were requested for three samples. The laboratory spiked the associated method blank with a labeled standard solution containing pore water PRC 13C-2,3,7,8-TCDD, 13C-2,3,7,8-TCDF, and 13C-2,3,4,7,8-PeCDF; therefore, they were detected in the method blank. No data were qualified based on positive results of these compounds in method blank.

*SDG E1600746:* Porewater PRC were requested for four samples. The laboratory spiked the associated method blank with a labeled standard solution containing pore water PRC 13C-2,3,7,8-TCDD, 13C-2,3,7,8-TCDF, and 13C-2,3,4,7,8-PeCDF; therefore, they were detected in the method blank. No data were qualified based on positive results of these compounds in method blank.

# Field Blanks

The field blanks for this project are SMPE fiber samples. To evaluate the effect of field blank contamination on the sample data, action levels of 5x the blank concentrations were established. If a contaminant was detected in an associated field sample and the concentration was less than the action level, the result was qualified (U-6) at the reported concentration. No action was taken if the sample result was greater than the action level, or for non-detected results.

Only native target compounds (2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF) were evaluated in the field blanks.

*SDG E1600282*: Fiber Sample 04052016SJPW10 was reported in this SDG. No native target analytes were detected in this blank.

*SDG E1600308*: A deployment blank fiber Sample 04132016-SJGW200 was reported in this SDG. No native target analytes were detected in this blank.

*SDG E1600326*: Preparation process fiber Samples 03162016SJGW1, 04072016SJGW1, 04072016SJGW2 were reported with this SDG. No native target analytes were detected in these blanks.

Fiber blank Sample 04072016SJGW10 was reported in this SDG. No native target analytes were detected in this blank.

*SDG E1600542:* Retrieval blank fiber Sample 06142016\_SSGWRB was reported in this SDG. No native target analytes were detected in this blank.

*SDG E1600740*: Retrieval blank fiber Sample 07202016SJPWRB was reported in this SDG. No native target analytes were detected in this blank.

# Labeled Compound Recovery

Labeled compounds were added to all samples. The labeled compound percent recovery (%R) values are evaluated using the laboratory control limits. If the labeled compound recovery outlier value indicates a potential high bias, positive results for the associated compounds are estimated (J-13H); whereas outlier values indicating a potential low bias, positive results and reporting limits for the associated compounds are estimated (J/UJ-13L).

*SDG E1600426*: Labeled standard solution was not spiked into Samples 05152013-SJPW201, 05152016-SJPW202, 05152016-SJPW203, 05152016-SJPW204, 05152016-SJPW205, AND 05152016-SJPW206 because the solution contains porewater PRC compounds. The %R value for the clean-up standard 13C-1,2,3,7,8,9-HxCDF was acceptable.

For the method blank, laboratory control sample and laboratory control sample duplicate (LCS/LCSD) associated with the groundwater samples, the %R values for several labeled compounds were less than the lower control limit. No data were qualified for these QC samples.

*SDG E1600544*: For Sample SJCPRI-PW-2-A, approximately 10 mL of extract was lost during the extraction process resulting in low %R values of labeled compounds, including the PRC which behaved as labeled compounds in this sample. These %R values indicate a potential low bias. All results, including total homolog results, were estimated (J/UJ-13L) in this sample.

*SDG E1600738*: For Sample SJCP001SP1CDUP, the %R value for 13C-2,3,7,8-TCDF was less than the lower control limit, indicating a potential low bias. The reporting limit for 2,3,7,8-TCDF was estimated (UJ-13L) in this sample.

For Samples SJCP013-SP-1B and SJCP013SP1C, the %R values for 13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF were less than the lower control limit, indicating a potential low bias. The reporting limits for 2,3,7,8-TCDD and 2,3,7,8-TCDF was estimated (UJ-13L) in these samples.

For the method blank and the laboratory control sample, the %R values for 13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF were less than the lower control limit. No data were qualified for the QC sample outliers.

*SDG E1600740*: For Sample SJCP011SP1C, the %R value for 13C-2,3,7,8-TCDD was less than the lower control limit, indicating a potential low bias. The reporting limit for 2,3,7,8-TCDD was estimated (UJ-13L) in this sample. In Samples 07202016-SJPWRB, SJCP014SP1A, and SJCP014SP1C, the %R values for 13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF were less than the lower control limit, indicating a potential low bias. The reporting limits for 2,3,7,8-TCDD and 2,3,7,8-TCDF were estimated (UJ-13L) in these samples. In the method blank and the laboratory control sample, the %R values for 13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF were less than the lower control limit. No data were qualified for the QC sample outliers.

*SDG E1600741:* For Sample SJCP008SP1A, the %R value for 13C-2,3,7,8-TCDD was less than the lower control limit, indicating a potential low bias. The reporting limit for 2,3,7,8-TCDD was estimated (UJ-13L) in this sample. The %R value for 13C-2,3,7,8-TCDF was less than 10% in this sample. The reporting limit for 2,3,7,8-TCDF was rejected (R-13L) in this sample.

*SDG E1600745:* Labeled standard solution was not spiked into Samples SJCR002SP2A, SJCR002SP2B, and SJCR002SP2C because the solution contains porewater PRC compounds. The %R value for the clean-up standard 13C-1,2,3,7,8,9-HxCDF was acceptable.

For Samples SJCP012SP1A, and SJCP012SP1C, the %R values for all labeled compounds as well as the cleanup standard, 13C-1,2,3,7,8,9-HxCDF, were less than the lower control limit. All results were estimated (UJ-13L).

*SDG E1600746*: Labeled standard solution was not spiked into Samples SJCR003SP2A, SJCR003SP2B, SJCR003SP2C, and SJCR002SP2AW because the solution contains pore water PRC compounds. The %R values for the clean-up standard 13C-1,2,3,7,8,9-HxCDF were acceptable.

For Samples SJCP009SP1A and SJCP009SP1B, the %R values for all labeled compounds as well as the cleanup standard, 13C-1,2,3,7,8,9-HxCDF, were less than the lower control limit. All results were estimated (UJ-13L).

# Matrix Spike/Matrix Spike Duplicate

Matrix spike/matrix spike duplicate (MS/MSD) samples are not required by the method and were not analyzed. Accuracy and precision were evaluated using the labeled compound and laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results.

# Laboratory Control Sample /Laboratory Control Sample Duplicate

The laboratory control sample/laboratory control sample duplicates (LCS/LCSD) are evaluated using laboratory limits for the native compounds and 30-170% for the performance reference compounds (PRC). If the recovery outlier value indicates a potential high bias, positive results for the associated compounds are estimated (J-10H); whereas outlier values indicating a potential low bias, positive

results and reporting limits for the associated compounds are estimated (J/UJ-10L). The relative percent difference (RPD) control limit for both native compounds and PRC is 50%. If the RPD value is greater than the control limit associated positive results are estimated (J-9).

*SDG E1600282, E1600738, & E1600740:* The %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). The RPD values were acceptable.

*SDG E1600308 & E1600542*: The PRC 13C-1,2,3,4-TCDD, 37CI-2,3,7,8-TCDD, and 13C-2,3,4,7,8-PeCDF were not spiked into the LCS/LCSD. Accuracy for the PRC was evaluated using the labeled compound recovery values, there was no measure of laboratory precision. The %R and RPD values for the native compounds were acceptable.

*SDG E1600326*: The %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). The PRC 13C-1,2,3,4-TCDD, 37CI-2,3,7,8-TCDD, and 13C-2,3,4,7,8-PeCDF were not spiked into the LCS/LCSD. Accuracy for the PRC was evaluated using the labeled compound recovery values, there was no measure of laboratory precision. The %R and RPD values for the native compounds were acceptable.

*SDG E1600426*: For the groundwater and pore water LCS/LCSD samples, the %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). The RPD values were acceptable. The groundwater PRC 37CI-2,3,7,8-TCDD and 13C-1,2,3,4-TCDF were not spiked into the LCS/LCSD. Accuracy for these PRC were evaluated using the labeled compound recovery values; there was no measure of laboratory precision for these PRC. All other groundwater and pore water PRC %R and RPD values were acceptable.

*SDG E1600448*: The %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). All other %R and RPD values were acceptable.

*SDG E1600554*: The PRC 13C-2,3,4,7,8-PeCDF was not spiked into the LCS/LCSD. The %R and RPD values for all other target compounds were acceptable.

*SDG E1600739*: The %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). No PRC were spiked into the LCS/LCSD. Accuracy for the PRC were evaluated using the labeled compound recovery values, there was no measure of laboratory precision. The RPD values for the native compounds were acceptable.

*SDG E1600741:* The %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). No PRC were requested for this data package.

**SDG E1600745:** Two (2) sets of LCS/LCSD were analyzed. The %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). For the LCS/LCSD associated with the samples that included PRC, the %R value for the PRC 13C-2,3,4,7,8-PeCDF was less than the lower control limit, indicating a potential low bias. The associated results were estimated (J-10L).

**SDG E1600746:** Three (3) sets of LCS/LCSD were analyzed. The %R values for 2,3,7,8-TCDD, 2,3,7,8-TCDF, and 2,3,4,7,8-PeCDF were less than the lower control limits, indicating a potential low bias. The associated reporting limits were estimated (UJ-10L). For the LCS/LCSD associated with the samples that included PRC, the %R value for the PRC 13C-2,3,4,7,8-PeCDF was less than the lower control limit, indicating a potential low bias. The associated results were estimated (J-10L).

# **Field Replicates**

The following acceptance criteria were used to evaluate precision: the relative percent difference (RPD) control limit is 50% for results greater than 5x the reporting limit (RL). For results less than 5x the RL, the difference between the sample and replicate must be less than twice the RL. No data were qualified based on field replicate precision outliers. Data users should consider the impact of field precision outliers on the reported results. With the exceptions noted below, field precision was acceptable.

*SDG E1600542*: Two pair of field replicates were submitted with this SDG: SJMW1004 GW77.582.5 & SJMW004D GW77.582.5 and SJWM1012 GW09.814.8 & SJWM012 GW09.814.8. Field precision was acceptable.

*SDG E1600738:* Three pair of field replicates were submitted with this SDG. No target analytes were detected in any of these samples. Field precision was acceptable.

Parent	DUPLICATE
SJCP001SP1A	SJCP001SP1ADUP
SJCP001SP1B	SJCP001SP1BDUP
SJCP001SP1C	SJCP001SP1CDUP

*SDG E1600745 & E1600746:* Three pair of field replicates were submitted with this SDG. No target analytes were detected in any of these samples. Field precision was acceptable.

Parent	DUPLICATE
(from E1600746)	(from 1600745)
SJCP009SP1A	SJCP009SP1ADUP
SJCP009SP1B	SJCP009SP1BDUP
SJCP009SP1C	SJCP009SP1CDUP

# **Target Analyte List**

MATRIX	NATIVE	PRC		
IVIATRIX	INATIVE	(WHEN REQUESTED)		
	2,3,7,8-TCDD	37CI-2,3,7,8-TCDD		
Groundwater Samples	2,3,7,8-TCDF	13C-1,2,3,4-TCDF		
	2,3,4,7,8-PeCDF	13C-2,3,4,7,8-PeCDF		
	2,3,7,8-TCDD	13C-2,3,7,8-TCDD		
Pore Water Samples	2,3,7,8-TCDF	13C-2,3,7,8-TCDF		
	2,3,4,7,8-PeCDF	13C-2,3,4,7,8-PeCDF		

The target analyte list for groundwater and pore water samples was as follows:

# **Reported Results**

All sample results were reported as absolute concentrations, in pg.

The Estimated Detection Limit (EDL) values reported by the laboratory were greater than the Equipment Detection Limit requirements listed in the Sampling Analysis Plan (SAP).

# **Compound Identification**

The laboratory assigned K-flags to results where a peak was detected but did not meet ion ratio quantitation criteria. The reported values cannot be considered as positive identifications for these analytes. These results were considered potential false positives or estimated maximum possible concentrations (EMPC) and were qualified as not detected (U-25) at the reported values. Results were qualified as not detected in SDGs E1600542, E1600745, and E1600746.

The method requires the confirmation of 2,3,7,8-TCDF using an alternate GC column as the DB5 column that is typically used cannot fully separate 2,3,7,8-TCDF from closely eluting non-target TCDF isomers. The laboratory did not perform a second column confirmation; however, the laboratory uses a DB-5MSUI column. This column provides adequate resolution of the TCDF isomers as indicated by the acceptable peak to valley ratios. Since the 2,3,7,8-TCDF resolution was acceptable, no action was necessary.

# **Calculation Verification**

*SDGs E1600326, E1600426, & E1600738:* Several results were verified by recalculation from the raw data.

The recalculations of the 2,3,7,8-TCDD relative response factor (RRF) values from the initial calibration did not match the reported values for two of the SDGs that received a full validation. The laboratory was contacted and they confirmed that the RRF values for 2,3,7,8-TCDD and 2,3,7,8-TCDF had been transposed on the summary form. The same error was found on the ICAL summary forms for SDGs

E1600282, E1600308, E1600448, E1600542, E1600739, E1600740, E1600741, and E1600746. SDGs E1600544, E1600738, and E1600745 contained the corrected initial calibration summary form. Since the same ICAL was used for the analysis of all of the SPME samples, no further action was taken.

#### OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical method. With the exceptions noted above, accuracy was acceptable as demonstrated by labeled compound and LCS/LCSD %R values. Precision was acceptable as demonstrated by the LCS/LCSD and field replicate RPD values.

Detection limits were elevated due to ion ratio outliers and method blank contamination. Data were estimated due to labeled compound and LCS/LCSD accuracy outliers.

Data were rejected due to very low labeled compound recovery outliers. Data were flagged do-not-report (DNR) in the EDD to indicate results that should not be used.

Rejected data and data flagged do-not-report (DNR) are not usable for any reason. All other data, as qualified, are acceptable for use.



# **APPENDIX A**

# DATA QUALIFIER DEFINITIONS REASON CODES AND CRITERIA TABLES

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# DATA VALIDATION QUALIFIER CODES Based on National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents the approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

The following is an EcoChem qualifier that may also be assigned during the data review process:

DNR Do not report; a more appropriate result is reported from another analysis or dilution.

# DATA QUALIFIER REASON CODES

Group	Code	Reason for Qualification
Sample Handling	1	Improper Sample Handling or Sample Preservation (i.e., headspace, cooler temperature, pH, summa canister pressure); Exceeded Holding Times
	24	Instrument Performance (i.e., tune, resolution, retention time window, endrin breakdown, lock-mass)
	5A	Initial Calibration (RF, %RSD, r <sup>2</sup> )
Instrument Performance	5B	Calibration Verification (CCV, CCAL; RF, %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	5C	Initial Calibration Verification (ICV %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	6	Field Blank Contamination (Equipment Rinsate, Trip Blank, etc.)
Blank Contamination	7	Lab Blank Contamination (i.e., method blank, instrument blank, etc.) Use low bias flag (L) <sup>1</sup> for negative instrument blanks
	8	Matrix Spike (MS and/or MSD) Recoveries Use bias flags (H,L) <sup>1</sup> where appropriate
	9	Precision (all replicates: LCS/LCSD, MS/MSD, Lab Replicate, Field Replicate)
Precision and Accuracy	10	Laboratory Control Sample Recoveries (a.k.a. Blank Spikes) Use bias flags (H,L) <sup>1</sup> where appropriate
	12	Reference Material Use bias flags (H,L) <sup>1</sup> where appropriate
	13	Surrogate Spike Recoveries (a.k.a. labeled compounds, recovery standards) Use bias flags (H,L) <sup>1</sup> where appropriate
	16	ICP/ICP-MS Serial Dilution Percent Difference
	17	ICP/ICP-MS Interference Check Standard Recovery Use bias flags (H,L) <sup>1</sup> where appropriate
Interferences	19	Internal Standard Performance (i.e., area, retention time, recovery)
	22	Elevated Detection Limit due to Interference (i.e., chemical and/or matrix)
	23	Bias from Matrix Interference (i.e. diphenyl ether, PCB/pesticides)
	2	Chromatographic pattern in sample does not match pattern of calibration standard
	3	2 <sup>nd</sup> column confirmation (RPD or %D)
Identification and Quantitation	4	Tentatively Identified Compound (TIC) (associated with NJ only)
	20	Calibration Range or Linear Range Exceeded
	25	Compound Identification (i.e., ion ratio, retention time, relative abundance, etc.)
N.C II	11	A more appropriate result is reported (multiple reported analyses i.e., dilutions, re- extractions, etc. Associated with "R" and "DNR" only)
Miscellaneous	14	Other (See DV report for details)
	26	Method QC information not provided

<sup>1</sup>H = high bias indicated

L = low bias indicated

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Waters/Solids $\leq 6^{\circ}C \&$ in the dark Tissues <-10°C & in the dark <b>Preservation Aqueous:</b> If Cl <sub>2</sub> is present Thiosulfate must be added and if pH > 9 it must be adjusted to 7 - 9	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/R(ND) if thiosulfate not added if Cl <sub>2</sub> present; J(pos)/UJ(ND) if pH not adjusted J(pos)/UJ(ND) if temp > 20°C	1	EcoChem PJ, see TM-05
Holding Time	If properly stored, 1 year or: Extraction (all matrices): 30 days from collection Analysis (all matrices): 45 days from extraction	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If not properly stored or HT exceedance: J(pos)/UJ(ND)	1	EcoChem PJ, see TM-05 Gross exceedance = > 1 year 2011 NFG Note: Under CWA, SDWA, and RCRA the HT for H2O is 7 days.
Instrument Performa	nce				
Mass Resolution (Tuning)	PFK (Perfluorokerosene) ≥10,000 resolving power at m/z 304.9824. Exact mass of m/z 380.9760 w/in 5 ppm of theoretical value (380.97410 to 380.97790) . Analyzed prior to ICAL and at the start and end of each 12 hr. shift.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	R(pos/ND) all analytes in all samples associated with the tune	24	Notify PM
Windows Defining Mix	Peaks for first and last eluters must be within established retention time windows for each selector group (chlorination level)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If peaks are not completely within windows (clipped): If natives are ok, J(pos)/UJ(ND) homologs (Totals) If natives are affected, R all results for that selector group	24	Notify PM
Column Performance Mix	Both mixes must be analyzed before ICAL and CCAL Valley < 25% (valley = (x/y)*100%) where x = ht. of TCDD (or TCDF) & y = baseline to bottom of valley For all isomers eluting near the 2378-TCDD (TCDF) peak (TCDD only for 8290)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if valley > 25%	24	EcoChem PJ, see TM-05, Rev. 2; Note: TCDF is evaluated only if second column confirmation is performed
Initial Calibration Sensitivity	S/N ratio > 10 for all native and labeled compounds in CS1 std.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5A	
Initial Calibration <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If 2 or more ion ratios are out for one compound in ICAL, J(pos)	5A	EcoChem PJ, see TM-05, Rev. 2

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments		
Instrument Performa	nce (continued)						
Initial Calibration (Minimum 5 stds.)	%RSD < 20% for native compounds %RSD <30% for labeled compounds (%RSD < 35% for labeled compounds under 1613b)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) natives if %RSD > 20%	5A			
Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD >25 min on DB5 & >15 min on DB-225	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action		EcoChem PJ, see TM-05, Rev. 2		
Continuing Calibration (Prior to each 12 hr. shift) <b>Sensitivity</b>	S/N ratio for CS3 standard > 10	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5B			
Continuing Calibration (Prior to each 12 hr. shift) <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	For congener with ion ratio outlier, J(pos) natives in all samples associated with CCAL. No action for labeled congener ion ratio outliers.	25	EcoChem PJ, see TM-05		
Continuing Calibration (Prior to each 12 hr.	%D+/-20% for native compounds %D +/-30% for labeled compounds (Must meet limits in Table 6, Method 1613B) If %D in the closing CCAL are within 25%/35%, the mean RF from the two CCAL may be used to calculate samples (Section 8.3.2.4 of 8290).	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Labeled compounds: Narrate, no action. Native compounds: 1613: J(pos)/UJ(ND)if %D is outside Table 6 limits J(pos)/R(ND) if %D is +/-75% of Table 6 limits 8290: J(pos)/UJ(ND) if %D = 20% - 75% J(pos)/R(ND) if %D > 75%	5B (H,L) <sup>3</sup>			
shift) Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD and <sup>13</sup> C <sub>12</sub> -123789-HxCDD should be ± 15 seconds of ICAL RRT for all other compounds must meet criteria listed in Table 2 Method 1316.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action	5B	EcoChem PJ, see TM-05		
Blank Contamination	lank Contamination						
Method Blank (MB)	MB: One per matrix per batch of (of ≤ 20 samples) No detected compounds > RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U(pos) if result is < 5X action level.	7	Hierarchy of blank review: #1 - Review MB, qualify as needed		
Field Blank (FB)	FB: frequency as per QAPP No detected compounds > RL	WELTOU	U(pos) if result is < 5X action level.	6	#2 - Review FB , qualify as needed		

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments		
Precision and Accura	recision and Accuracy						
MS/MSD (recovery)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of $\leq$ 20 samples)	EcoChem standard policy	J(pos) if both %R > UCL - high bias J(pos)/UJ(ND) if both %R < LCL - low bias J(pos)/R(ND) if both %R < 10% - very low bias	8 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria. No action if parent concentration is >4x the amount spiked.		
	Use most current laboratory control limits		J(pos)/UJ(ND) if one > UCL & one < LCL, with no bias <b>PJ if only one %R outlier</b>		Qualify parent sample only unless other QC indicates systematic problems.		
MS/MSD (RPD)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) in parent sample if RPD > CL	9	Qualify parent sample only.		
LCS (or OPR)	One per lab batch (of ≤ 20 samples) Use most current laboratory control limits <b>or</b> Limits from Table 6 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	10 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria, when LCSD is analyzed. Qualify all associated samples.		
LCS/LCSD (RPD)	LCSD not typically required for HRMS analyses. One set per matrix and batch of 20 samples RPD < 35%	Method <sup>(2)</sup> Ecochem standard policy	J(pos) assoc. compound in all samples if RPD > CL	9	Qualify all associated samples.		
Lab Duplicate (RPD)	Lab Dup not typically required for HRMS analyses. One per lab batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos)/UJ(ND) if RPD > CL	9			
Labeled Compounds (Internal Standards)	Added to all samples %R = 40% - 135% in all samples 8290 %R must meet limits in Table 7 Method 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	13 (H,L) <sup>3</sup>			
Field Duplicates	Solids: RPD <50% OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Narrate and qualify if required by project	9	Use professional judgment		

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason	Discussion and Comments
Compound ID and Ca	•			Code	
Quantitation/ Identification	All ions for each isomer must maximize within ± 2 seconds. S/N ratio >2.5 Ion ratios must meet criteria listed in Table 8 Method 8290, or Table 9 of 1613B; RRTs w/in limits in Table 2 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate in report; qualify if necessary NJ(pos) for retention time outliers. U(pos) for ion ratio outliers.	25	EcoChem PJ, see TM-05
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If laboratory correctly reported an EMPC value, qualify the native compound U(pos) to indicate that the value is a detection limit and qualify total homolog groups J (pos)	25	Use professional judgment See TM-18
Interferences	Interferences from chlorodiphenyl ether compounds	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if present	23	See TM-16
Interferences	Lock masses must not deviate ± 20% from values in Table 8 of 1613B	Method (2)	J(pos)/UJ(ND) if present	24	See TM-17
Second Column Confirmation	All 2,3,7,8-TCDF hits must be confirmed on a DB-225 (or equiv) column. All QC criteria must also be met for the confirmation analysis.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Report the DB-225 value. If not performed use PJ.	3	DNR-11 DB5 result if both results from both columns are reported. <b>EcoChem PJ, see TM-05</b>
Calculation Check	Check 10% of field & QC sample results	EcoChem standard policy	Contact laboratory for resolution and/or corrective action	na	Full data validation only.
Electronic Data Delive	erable (EDD)				
Verification of EDD to hardcopy data	EcoChem verify @ 10% unless problems noted; then increase level up to 100% for next several packages.		Depending on scope of problem, correct at EcoChem (minor issues) to resubmittal by laboratory (major issues).	na	EcoChem Project Manager and/or Database Administrator will work with lab to provide long-term corrective action.
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	Standard reporting policy	Use "DNR" to flag results that will not be reported.	11	

(pos) - positive (detected) results; (ND) - not detected results

<sup>1</sup> National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) & Chlorinated Dibenzofurans (CDFs) Data Review, September 2011

<sup>2</sup> Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), USEPA SW-846, Method 8290

<sup>2</sup> EPA Method 1613, Rev.B, Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGS/HRMS, October 1994

<sup>3</sup> NFG 2013 suggests using "+ / -" to indicate bias; EcoChem has chosen "H" = high bias indicated; "L" = low bias indicated.



# **APPENDIX B**

# **QUALIFIED DATA SUMMARY TABLE**

SDG	Sample ID	Lab Sample ID	Mathad	Analyte	Result	Units	Lab	DV	DV
300	Sample iD		Wethou	Analyte	Result	Units	Flag	Qualifier	Reason
E1600282	04052016SJPW10	E1600282-006	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600282	04052016SJPW10	E1600282-006	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600282	04052016SJPW10	E1600282-006	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	03162016SJGW1	E1600326-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	03162016SJGW1	E1600326-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	03162016SJGW1	E1600326-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW1	E1600326-002	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW1	E1600326-002	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	04072016SJGW1	E1600326-002	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW2	E1600326-003	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW2	E1600326-003	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	04072016SJGW2	E1600326-003	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW10	E1600326-004	Dioxins	13C12-1,2,3,7,8,9-Hexachlorodibenzofuran	46	percent		DNR	11
E1600326	04072016SJGW10	E1600326-004	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW10	E1600326-004	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	04072016SJGW10	E1600326-004	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW11	E1600326-005	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW11	E1600326-005	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	04072016SJGW11	E1600326-005	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW12	E1600326-006	Dioxins	13C12-1,2,3,7,8,9-Hexachlorodibenzofuran	42	percent		DNR	11
E1600326	04072016SJGW12	E1600326-006	Dioxins	13C12-1,2,3,7,8-Pentachlorodibenzofuran	41	percent		DNR	11
E1600326	04072016SJGW12	E1600326-006	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzofuran	38	percent		DNR	11
E1600326	04072016SJGW12	E1600326-006	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzo-p-dioxin	36	percent		DNR	11
E1600326	04072016SJGW12	E1600326-006	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW12	E1600326-006	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	04072016SJGW12	E1600326-006	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW13	E1600326-007	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW13	E1600326-007	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	04072016SJGW13	E1600326-007	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW14	E1600326-008	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW14	E1600326-008	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L

SDG	Samula ID	Lab Camala ID	Method	Analysis	Result	Units	Lab	DV	DV
SDG	Sample ID	Lab Sample ID	wiethoa	Analyte	Result	Units	Flag	Qualifier	Reason
E1600326	04072016SJGW14	E1600326-008	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600326	04072016SJGW15	E1600326-009	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600326	04072016SJGW15	E1600326-009	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600326	04072016SJGW15	E1600326-009	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05132016-SJGW300	E1600426-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05132016-SJGW300	E1600426-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	622	pg	U	UJ	10L
E1600426	05132016-SJGW300	E1600426-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05132016-SJGW301	E1600426-002	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05132016-SJGW301	E1600426-002	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	909	pg	U	UJ	10L
E1600426	05132016-SJGW301	E1600426-002	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05132016-SJGW302	E1600426-003	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05132016-SJGW302	E1600426-003	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	866	pg	U	UJ	10L
E1600426	05132016-SJGW302	E1600426-003	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05132016-SJGW303	E1600426-004	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05132016-SJGW303	E1600426-004	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600426	05132016-SJGW303	E1600426-004	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05152013-SJPW201	E1600426-005	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05152013-SJPW201	E1600426-005	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600426	05152013-SJPW201	E1600426-005	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05152013-SJPW201	E1600426-005	Dioxins	37Cl4-2,3,7,8-TCDD	985.799	pg		DNR	11
E1600426	05152016-SJPW202	E1600426-006	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05152016-SJPW202	E1600426-006	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600426	05152016-SJPW202	E1600426-006	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05152016-SJPW203	E1600426-007	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05152016-SJPW203	E1600426-007	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600426	05152016-SJPW203	E1600426-007	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05152016-SJPW204	E1600426-008	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05152016-SJPW204	E1600426-008	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600426	05152016-SJPW204	E1600426-008	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05152016-SJPW205	E1600426-009	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05152016-SJPW205	E1600426-009	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab	DV	DV
	Sumple 15		Method		Result	Onits	Flag	Qualifier	Reason
E1600426	05152016-SJPW205	E1600426-009	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600426	05152016-SJPW206	E1600426-010	Dioxins	13C12-1,2,3,7,8-Pentachlorodibenzofuran	4.694	pg		DNR	11
E1600426	05152016-SJPW206	E1600426-010	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600426	05152016-SJPW206	E1600426-010	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600426	05152016-SJPW206	E1600426-010	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600448	05182016_SJPWDP	E1600448-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600448	05182016_SJPWDP	E1600448-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600448	05182016_SJPWDP	E1600448-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600542	SJMW004 SGW1217	E1600542-003	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	5.24	pg	JK	U	25
E1600542	SJMW002 GW07.512.5	E1600542-010	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	34.068	pg	К	U	25
E1600542	SJMW001 GW09.514.5	E1600542-011	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	39.416	pg	К	U	25
E1600542	SJMW1012 GW09.814.8	E1600542-016	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.69	pg	JK	U	25
E1600554	SJCPRI-PW-2-A	E1600554-001	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	28.636	pg		J	13L
E1600554	SJCPRI-PW-2-A	E1600554-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	13L
E1600554	SJCPRI-PW-2-A	E1600554-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	13L
E1600554	SJCPRI-PW-2-A	E1600554-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	13L
E1600554	SJCPRI-PW-2-A	E1600554-001	Dioxins	Pentachlorodibenzofuran (Total)	500	pg	U	UJ	13L
E1600554	SJCPRI-PW-2-A	E1600554-001	Dioxins	Tetrachlorodibenzodioxin (Total)	100	pg	U	UJ	13L
E1600554	SJCPRI-PW-2-A	E1600554-001	Dioxins	Tetrachlorodibenzofuran (Total)	100	pg	U	UJ	13L
E1600738	SJCP001SP1ADUP	E1600738-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.18	pg	U	UJ	10L
E1600738	SJCP001SP1ADUP	E1600738-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	5.38	pg	U	UJ	10L
E1600738	SJCP001SP1ADUP	E1600738-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	2.98	pg	U	UJ	10L
E1600738	SJCP001SP1BDUP	E1600738-002	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	0.957	pg	U	UJ	10L
E1600738	SJCP001SP1BDUP	E1600738-002	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	1.94	pg	U	UJ	10L
E1600738	SJCP001SP1BDUP	E1600738-002	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	2.15	pg	U	UJ	10L
E1600738	SJCP001SP1CDUP	E1600738-003	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.26	pg	U	UJ	10L
E1600738	SJCP001SP1CDUP	E1600738-003	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.92	pg	U	IJ	10L,13L
E1600738	SJCP001SP1CDUP	E1600738-003	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	4.19	pg	U	IJ	10L
E1600738	SJCP001SP1A	E1600738-004	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.72	pg	U	IJ	10L
E1600738	SJCP001SP1A	E1600738-004	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	9.28	pg	U	UJ	10L

SDG	Sample ID	Lab Sample ID	Method	Analyta	Result	Units	Lab	DV	DV
300	Sample ID	Lab Sample ID	wethod	Analyte	Result	Units	Flag	Qualifier	Reason
E1600738	SJCP001SP1A	E1600738-004	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	4.72	pg	U	UJ	10L
E1600738	SJCP001SP1B	E1600738-005	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	3.72	pg	U	UJ	10L
E1600738	SJCP001SP1B	E1600738-005	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	9.94	pg	U	UJ	10L
E1600738	SJCP001SP1B	E1600738-005	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.96	pg	U	UJ	10L
E1600738	SJCP001SP1C	E1600738-006	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.57	pg	U	UJ	10L
E1600738	SJCP001SP1C	E1600738-006	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.11	pg	U	UJ	10L
E1600738	SJCP001SP1C	E1600738-006	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	1.32	pg	U	UJ	10L
E1600738	SJCP013SP1A	E1600738-007	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.26	pg	U	UJ	10L
E1600738	SJCP013SP1A	E1600738-007	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.31	pg	U	UJ	10L
E1600738	SJCP013SP1A	E1600738-007	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	1.24	pg	U	UJ	10L
E1600738	SJCP013SP1B	E1600738-008	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.11	pg	U	UJ	10L
E1600738	SJCP013SP1B	E1600738-008	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	9.65	pg	U	UJ	10L,13L
E1600738	SJCP013SP1B	E1600738-008	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	8.01	pg	U	UJ	10L,13L
E1600738	SJCP013SP1C	E1600738-009	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	3.29	pg	U	UJ	10L
E1600738	SJCP013SP1C	E1600738-009	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	8.33	pg	U	UJ	10L,13L
E1600738	SJCP013SP1C	E1600738-009	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	9.61	pg	U	UJ	10L,13L
E1600739	SJMW013GW50.355.3	E1600739-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600739	SJMW013GW50.355.3	E1600739-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600739	SJMW013GW50.355.3	E1600739-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600740	SJCP011SP1A	E1600740-007	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	SJCP011SP1A	E1600740-007	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600740	SJCP011SP1A	E1600740-007	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600740	SJCP011SP1B	E1600740-008	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	SJCP011SP1B	E1600740-008	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600740	SJCP011SP1B	E1600740-008	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600740	SJCP011SP1C	E1600740-009	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	SJCP011SP1C	E1600740-009	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600740	SJCP011SP1C	E1600740-009	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L,13L
E1600740	07202016-SJPWRB	E1600740-013	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	07202016-SJPWRB	E1600740-013	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	IJ	10L,13L
E1600740	07202016-SJPWRB	E1600740-013	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L,13L

SDG	Sample ID	Lab Sample ID	Method	Analyta	Result	Units	Lab	DV	DV
300	Sample ID		Wethou	Analyte	Result	Units	Flag	Qualifier	Reason
E1600740	SJCP014SP1A	E1600740-014	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	SJCP014SP1A	E1600740-014	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L,13L
E1600740	SJCP014SP1A	E1600740-014	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L,13L
E1600740	SJCP014SP1B	E1600740-015	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	SJCP014SP1B	E1600740-015	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600740	SJCP014SP1B	E1600740-015	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600740	SJCP014SP1C	E1600740-016	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	SJCP014SP1C	E1600740-016	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L,13L
E1600740	SJCP014SP1C	E1600740-016	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L,13L
E1600740	SJCP008SP1AW	E1600740-017	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600740	SJCP008SP1AW	E1600740-017	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600740	SJCP008SP1AW	E1600740-017	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600741	SJCP008SP1A	E1600741-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600741	SJCP008SP1A	E1600741-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	R	10L,13L
E1600741	SJCP008SP1A	E1600741-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L,13L
E1600741	SJCP008SP1B	E1600741-002	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600741	SJCP008SP1B	E1600741-002	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600741	SJCP008SP1B	E1600741-002	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600741	SJCP008SP1C	E1600741-003	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600741	SJCP008SP1C	E1600741-003	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600741	SJCP008SP1C	E1600741-003	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600745	SJCP006SP1A	E1600745-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.91	pg	U	UJ	10L
E1600745	SJCP006SP1A	E1600745-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	3.96	pg	U	UJ	10L
E1600745	SJCP006SP1A	E1600745-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.66	pg	U	UJ	10L
E1600745	SJCP006SP1B	E1600745-002	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.43	pg	U	UJ	10L
E1600745	SJCP006SP1B	E1600745-002	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	3.33	pg	U	UJ	10L
E1600745	SJCP006SP1B	E1600745-002	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	2.91	pg	U	IJ	10L
E1600745	SJCP006SP1C	E1600745-003	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.7	pg	U	UJ	10L
E1600745	SJCP006SP1C	E1600745-003	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.81	pg	U	UJ	10L
E1600745	SJCP006SP1C	E1600745-003	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.99	pg	U	UJ	10L
E1600745	SJCP009SP1ADUP	E1600745-004	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.88	pg	U	UJ	10L

SDG	Samula ID	Lab Comple ID	Method	Analyta	Result	Units	Lab	DV	DV
300	Sample ID	Lab Sample ID	wethod	Analyte	Result	Units	Flag	Qualifier	Reason
E1600745	SJCP009SP1ADUP	E1600745-004	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.16	pg	U	UJ	10L
E1600745	SJCP009SP1ADUP	E1600745-004	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.52	pg	U	UJ	10L
E1600745	SJCP009SP1BDUP	E1600745-005	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.14	pg	U	UJ	10L
E1600745	SJCP009SP1BDUP	E1600745-005	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.2	pg	U	UJ	10L
E1600745	SJCP009SP1BDUP	E1600745-005	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.67	pg	U	UJ	10L
E1600745	SJCP009SP1CDUP	E1600745-006	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.6	pg	U	UJ	10L
E1600745	SJCP009SP1CDUP	E1600745-006	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	5.73	pg	U	UJ	10L
E1600745	SJCP009SP1CDUP	E1600745-006	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	5.05	pg	U	UJ	10L
E1600745	SJCP007SP1A	E1600745-007	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.07	pg	U	UJ	10L
E1600745	SJCP007SP1A	E1600745-007	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	2.27	pg	U	UJ	10L
E1600745	SJCP007SP1A	E1600745-007	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	2.71	pg	U	UJ	10L
E1600745	SJCP007SP1B	E1600745-008	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.35	pg	U	UJ	10L
E1600745	SJCP007SP1B	E1600745-008	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	3.66	pg	U	UJ	10L
E1600745	SJCP007SP1B	E1600745-008	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.12	pg	U	UJ	10L
E1600745	SJCP007SP1C	E1600745-009	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.84	pg	U	UJ	10L
E1600745	SJCP007SP1C	E1600745-009	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.14	pg	U	UJ	10L
E1600745	SJCP007SP1C	E1600745-009	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.07	pg	U	UJ	10L
E1600745	SJCR002SP2A	E1600745-010	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	31.447	pg	Y,*	J	10L
E1600745	SJCR002SP2A	E1600745-010	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	23.1	pg	U	UJ	10L
E1600745	SJCR002SP2A	E1600745-010	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	63.9	pg	U	UJ	10L
E1600745	SJCR002SP2A	E1600745-010	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	46.9	pg	U	UJ	10L
E1600745	SJCR002SP2B	E1600745-011	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	44.063	pg	Y,*	J	10L
E1600745	SJCR002SP2B	E1600745-011	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzo-p-dioxin	27.687	pg	KY,*	U	25
E1600745	SJCR002SP2B	E1600745-011	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	14.4	pg	U	UJ	10L
E1600745	SJCR002SP2B	E1600745-011	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	46.8	pg	U	UJ	10L
E1600745	SJCR002SP2B	E1600745-011	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	27.4	pg	U	UJ	10L
E1600745	SJCR002SP2C	E1600745-012	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	39.567	pg	Y,*	J	10L
E1600745	SJCR002SP2C	E1600745-012	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	17.9	pg	U	UJ	10L
E1600745	SJCR002SP2C	E1600745-012	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	68.1	pg	U	UJ	10L
E1600745	SJCR002SP2C	E1600745-012	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	41.6	pg	U	UJ	10L
E1600745	SJCP004SP1A	E1600745-013	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.15	pg	U	UJ	10L

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab	DV	DV
500	Sample ID		Wiethou		Kesuit	Onits	Flag	Qualifier	Reason
E1600745	SJCP004SP1A	E1600745-013	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	7.52	pg	U	UJ	10L
E1600745	SJCP004SP1A	E1600745-013	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	4.53	pg	U	UJ	10L
E1600745	SJCP004SP1B	E1600745-014	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.37	pg	U	UJ	10L
E1600745	SJCP004SP1B	E1600745-014	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	4.85	pg	U	UJ	10L
E1600745	SJCP004SP1B	E1600745-014	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.44	pg	U	UJ	10L
E1600745	SJCP004SP1C	E1600745-015	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	2.26	pg	U	UJ	10L
E1600745	SJCP004SP1C	E1600745-015	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	2.5	pg	U	UJ	10L
E1600745	SJCP004SP1C	E1600745-015	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	2.52	pg	U	UJ	10L
E1600745	SJCP012SP1A	E1600745-016	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.92	pg	U	UJ	10L,13L
E1600745	SJCP012SP1A	E1600745-016	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	3.97	pg	U	UJ	10L,13L
E1600745	SJCP012SP1A	E1600745-016	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.61	pg	U	UJ	10L,13L
E1600745	SJCP012SP1B	E1600745-017	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.24	pg	U	UJ	10L
E1600745	SJCP012SP1B	E1600745-017	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	1.95	pg	U	UJ	10L
E1600745	SJCP012SP1B	E1600745-017	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	1.61	pg	U	UJ	10L
E1600745	SJCP012SP1C	E1600745-018	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	1.74	pg	U	UJ	10L,13L
E1600745	SJCP012SP1C	E1600745-018	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	3.02	pg	U	UJ	10L,13L
E1600745	SJCP012SP1C	E1600745-018	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.39	pg	U	UJ	10L,13L
E1600746	SJCR003SP2A	E1600746-001	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	11.402	pg	Y,*	J	10L
E1600746	SJCR003SP2A	E1600746-001	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzofuran	5.95	pg	KY,*	U	25
E1600746	SJCR003SP2A	E1600746-001	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzo-p-dioxin	4.701	pg	KY,*	U	25
E1600746	SJCR003SP2A	E1600746-001	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600746	SJCR003SP2A	E1600746-001	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	265	pg	U	UJ	10L
E1600746	SJCR003SP2A	E1600746-001	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	194	pg	U	UJ	10L
E1600746	SJCR003SP2B	E1600746-002	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	11.46	pg	Y,*	J	10L
E1600746	SJCR003SP2B	E1600746-002	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzo-p-dioxin	5.566	pg	KY,*	U	25
E1600746	SJCR003SP2B	E1600746-002	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600746	SJCR003SP2B	E1600746-002	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	141	pg	U	UJ	10L
E1600746	SJCR003SP2B	E1600746-002	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	116	pg	U	UJ	10L
E1600746	SJCR003SP2C	E1600746-003	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	13.202	pg	Y,*	J	10L
E1600746	SJCR003SP2C	E1600746-003	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzo-p-dioxin	8.905	pg	KY,*	U	25
E1600746	SJCR003SP2C	E1600746-003	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L

67 G							Lab	DV	DV
SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Flag	Qualifier	Reason
E1600746	SJCR003SP2C	E1600746-003	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	154	pg	U	UJ	10L
E1600746	SJCR003SP2C	E1600746-003	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	105	pg	U	UJ	10L
E1600746	SJCP009SP1A	E1600746-004	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L,13L
E1600746	SJCP009SP1A	E1600746-004	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L,13L
E1600746	SJCP009SP1A	E1600746-004	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L,13L
E1600746	SJCP009SP1B	E1600746-005	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L,13L
E1600746	SJCP009SP1B	E1600746-005	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L,13L
E1600746	SJCP009SP1B	E1600746-005	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L,13L
E1600746	SJCP009SP1C	E1600746-006	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600746	SJCP009SP1C	E1600746-006	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600746	SJCP009SP1C	E1600746-006	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600746	SJCR002SP2AW	E1600746-007	Dioxins	13C12-2,3,4,7,8-Pentachlorodibenzofuran	5.842	pg	Y,*	J	10L
E1600746	SJCR002SP2AW	E1600746-007	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzofuran	2.74	pg	KY,*	U	25
E1600746	SJCR002SP2AW	E1600746-007	Dioxins	13C12-2,3,7,8-Tetrachlorodibenzo-p-dioxin	3.68	pg	KY,*	U	25
E1600746	SJCR002SP2AW	E1600746-007	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600746	SJCR002SP2AW	E1600746-007	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	285	pg	U	UJ	10L
E1600746	SJCR002SP2AW	E1600746-007	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	165	pg	U	UJ	10L
E1600746	SJCP005SP1A	E1600746-008	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600746	SJCP005SP1A	E1600746-008	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600746	SJCP005SP1A	E1600746-008	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600746	SJCP005SP1B	E1600746-009	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600746	SJCP005SP1B	E1600746-009	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600746	SJCP005SP1B	E1600746-009	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L
E1600746	SJCP005SP1C	E1600746-010	Dioxins	2,3,4,7,8-Pentachlorodibenzofuran	500	pg	U	UJ	10L
E1600746	SJCP005SP1C	E1600746-010	Dioxins	2,3,7,8-Tetrachlorodibenzofuran	100	pg	U	UJ	10L
E1600746	SJCP005SP1C	E1600746-010	Dioxins	2,3,7,8-Tetrachlorodibenzo-p-dioxin	100	pg	U	UJ	10L



# DATA VALIDATION REPORT

# SAN JACINTO RIVER WASTE PITS 2016 SURFACE WATER SAMPLING

#### **Prepared for:**

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EcoChem Project: C22130-24

September 13, 2016

**Approved for Release:** 

Alisa S. Bodkin

Alison Bodkin Project Chemist **EcoChem, Inc.** 

# **PROJECT NARRATIVE**

#### **Basis for Data Validation**

This report summarizes the results of summary and full validation (EPA Stage 2B & 4) performed on surface water sample data for the San Jacinto River 2016 SPME Sampling Study. A complete list of samples is provided in the **Sample Index**.

Samples were analyzed for conventional parameters by ALS Environmental, Kelso, Washington. Maxxam Analytics International, Mississaugua, Ontario, Canada performed the dioxin analyses. The analytical methods and EcoChem project chemists are listed below.

ANALYSIS	METHOD	PRIMARY REVIEW	SECONDARY REVIEW
Dioxin/Furan Compounds	1613B	M. Swanson/A. Bodkin	C. Ransom
Total and Dissolved Organic Carbon	SM5310C		
Total Dissolved Solids	SM2540C	E. Clayton	A. Bodkin
Total Suspended Solids	SM2540D		

The data were reviewed using guidance and quality control criteria documented in the analytical methods and the following project and guidance documents:

- Sampling and Analysis Plan: Surface Water Study San Jacinto River Waste Pits Superfund Site (Integral/Anchor QEA, March 2016).
- USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review (USEPA 2011).
- USEPA National Functional Guidelines for Inorganic Data Review (USEPA October 2004).

EcoChem's goal in assigning data assessment qualifiers is to assist in proper data interpretation. If values are estimated (J or UJ), data may be used for site evaluation and risk assessment purposes but reasons for data qualification should be taken into consideration when interpreting sample concentrations. If values are assigned an R, the data are to be rejected and should not be used for any site evaluation purposes. If values have no data qualifier assigned, then the data meet the data quality objectives as stated in the documents and methods referenced above.

Data qualifier definitions, reason codes, and validation criteria are included as **APPENDIX A**. A Qualified Data Summary Table is included in **APPENDIX B**. Data Validation Worksheets and project associated communications will be kept on file at EcoChem, Inc. A qualified laboratory electronic data deliverable (EDD) is also submitted with this report.

# Sample Index San Jacinto River Waste Pits SW 2016

SDG	Sample ID	Lab Sample ID	Sample Type	Dioxins	тос	DOC	TDS/TSS
K1607776	SW003	K1607776-002			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW006	K1607776-001			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW009	K1607776-007			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW012	K1607776-004			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW015	K1607776-005			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW018	K1607776-006			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW021	K1607776-003			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW024	K1607776-008			$\checkmark$	$\checkmark$	$\checkmark$
K1607776	SW027	K1607776-009				$\checkmark$	
K1608119	SW032	K1608119-002			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW035	K1608119-001			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW038	K1608119-006			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW041	K1608119-004			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW044	K1608119-009			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW047	K1608119-008			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW050	K1608119-003			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW053	K1608119-005			$\checkmark$	$\checkmark$	$\checkmark$
K1608119	SW056	K1608119-007				$\checkmark$	
K1608356	SW086	K1608356-003			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW089	K1608356-002			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW092	K1608356-008			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW095	K1608356-009			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW098	K1608356-005			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW101	K1608356-006			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW104	K1608356-007			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW107	K1608356-004			$\checkmark$	$\checkmark$	$\checkmark$
K1608356	SW110	K1608356-001				$\checkmark$	
B6E4851	SW001	CRT810	Column	$\checkmark$			
B6E4851	SW002	CRT811	Filter	$\checkmark$			
B6E4851	SW004	CRT812	Column	$\checkmark$			
B6E4851	SW005	CRT813	Filter	$\checkmark$			
B6E4851	SW010	CRT814	Column	$\checkmark$			1
B6E4851	SW011	CRT815	Filter	$\checkmark$			
B6E4851	SW019	CRT816	Column	$\checkmark$			1
B6E4851	SW020	CRT817	Filter	$\checkmark$			
B6E4851	SW013	CRT818	Column	$\checkmark$			
B6E4851	SW014	CRT819	Filter	$\checkmark$			
B6E4851	SW016	CRT869	Column	$\checkmark$			
B6E4851	SW017	CRT871	Filter	$\checkmark$			

# Sample Index San Jacinto River Waste Pits SW 2016

SDG	Sample ID	Lab Sample ID	Sample Type	Dioxins	тос	DOC	TDS/TSS
B6E4851	SW025	CRT872	Column	$\checkmark$			
B6E4851	SW026	CRT873	Filter	$\checkmark$			
B6E4851	SW007	CRT874	Column	$\checkmark$			
B6E4851	SW008	CRT877	Filter	$\checkmark$			
B6E4851	SW022	CRT878	Column	$\checkmark$			
B6E4851	SW023	CRT879	Filter	$\checkmark$			
B6F0192	SW033	CSR059	Column	$\checkmark$			
B6F0192	SW034	CSR060	Filter	$\checkmark$			
B6F0192	SW030	CSR061	Column	$\checkmark$			
B6F0192	SW031	CSR062	Filter	$\checkmark$			
B6F0192	SW039	CSR063	Column	$\checkmark$			
B6F0192	SW040	CSR064	Filter	$\checkmark$			
B6F0192	SW048	CSR065	Column	$\checkmark$			
B6F0192	SW049	CSR066	Filter	$\checkmark$			
B6F0192	SW051	CSR067	Column	$\checkmark$			
B6F0192	SW052	CSR068	Filter	$\checkmark$			
B6F0192	SW036	CSR069	Column	$\checkmark$			
B6F0192	SW037	CSR070	Filter	$\checkmark$			
B6F0192	SW045	CSR071	Column	$\checkmark$			
B6F0192	SW046	CSR072	Filter	$\checkmark$			
B6F0192	SW054	CSR073	Column	$\checkmark$			
B6F0192	SW055	CSR074	Filter	$\checkmark$			
B6F0192	SW042	CSR075	Column	$\checkmark$			
B6F0192	SW043	CSR076	Filter	$\checkmark$			
B6F5904	SW108	CTR999	Column	$\checkmark$			
B6F5904	SW109	CTS000	Filter	$\checkmark$			
B6F5904	SW087	CTS001	Column	$\checkmark$			
B6F5904	SW084	CTS002	Column	$\checkmark$			
B6F5904	SW088	CTS003	Filter	$\checkmark$			
B6F5904	SW085	CTS004	Filter	$\checkmark$			
B6F5904	SW105	CTS005	Column	$\checkmark$			
B6F5904	SW106	CTS006	Filter	$\checkmark$			
B6F5904	SW096	CTS007	Column	$\checkmark$			
B6F5904	SW097	CTS008	Filter	$\checkmark$			
B6F5904	SW099	CTS009	Column	$\checkmark$			
B6F5904	SW100	CTS010	Filter	$\checkmark$			
B6F5904	SW102	CTS011	Column	$\checkmark$			
B6F5904	SW103	CTS012	Filter	$\checkmark$			
B6F5904	SW090	CTS013	Column	$\checkmark$			

# Sample Index San Jacinto River Waste Pits SW 2016

SDG	Sample ID	Lab Sample ID	Sample Type	Dioxins	тос	DOC	TDS/TSS
B6F5904	SW091	CTS014	Filter	$\checkmark$			
B6F5904	SW093	CTS015	Column	$\checkmark$			
B6F5904	SW094	CTS016	Filter	$\checkmark$			
B6F5904	SW028	CTS139	Column	$\checkmark$			
B6F5904	SW029	CTS140	Filter	$\checkmark$			

# DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 Surface Water Sampling Dioxin/Furan Compounds by Method 1613B

This report documents the review of analytical data from the analysis of treated surface water samples and the associated laboratory and field quality control (QC) samples. Samples were analyzed by Maxxam Analytics International, Mississaugua, Ontario, Canada. See the **Sample Index** for a complete list of samples.

SDG	Number of Samples	Validation Level	
B6E4851	8 Surface Water (XAD) 8 Surface Water (Filter) 1 Equipment Blank (XAD) 1 Equipment Blank (Filter)	EPA Stage 4	
B6F0192	8 Surface Water (XAD) 8 Surface Water (Filter) 1 Equipment Blank (XAD) 1 Equipment Blank (Filter)	Stage 2B	
B6F5904	8 Surface Water (XAD) 8 Surface Water (Filter) 1 Equipment Blank (XAD) 1 Equipment Blank (Filter) 1 Field Blank (XAD) 1 Field Blank (Filter)	Stage 2B	

# DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

*SDG B6F0192:* The filter sample result for Sample SW042 was incorrect in the original submission. The laboratory was contacted and submitted a revised hardcopy and EDD.

*SDG B6E4851:* The filter sample results were incorrect in the original submission. The laboratory was contacted and submitted a revised hardcopy and EDD.

# EDD TO HARDCOPY VERIFICATION

The electronic data deliverable (EDD) was verified against the laboratory portable document format (PDF) data package. No errors were found.

# **TECHNICAL DATA VALIDATION**

The QC requirements that were reviewed are listed below.

1	Sample Receipt, Preservation, and Holding Times	2	Laboratory Control Samples
$\checkmark$	System Performance and Resolution Checks	1	Matrix Spike/Matrix Spike Duplicates (MS/MSD)
$\checkmark$	Initial Calibration	1	Laboratory Duplicates
2	Calibration Verification	1	Field Duplicates
2	Method Blanks	1	Reported Results
2	Field Blanks	2	Compound Identification
2	Labeled Compounds	1	Calculation Verification

 $\checkmark$  Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

#### Sample Receipt, Preservation, and Holding Times

The sample coolers were received at temperatures greater than the upper advisory limit of 6°C, ranging from 7.1°C to 13.2°C. These temperature outliers did not impact data quality; no action was taken.

#### **Calibration Verification**

The control limit for continuing calibration (CCAL) percent difference (%D) is 20% for native compounds (30% for labeled compounds). For continuing CCAL %D outliers, positive results associated with a potential high bias were estimated (J-5BH). For %D values indicative of a potential low bias, positive results and non-detects were estimated (J/UJ-5BL). The following outliers resulted in qualification of data:

SDG	CCAL Analysis Date Compound		%D	Qualifier	
	M4160812B15	08/12/16	1,2,3,6,7,8,-HxCDF	22	J-5BH
B6E4851	M4160815C01	14160815C01 08/15/16 OCD		-21	J-5BL
	M4160815C13 08/15/16 2,3,7,8-TCDF		-21	J-5BL	
	M3160819C01	08/19/16	1,2,3,4,6,7,8-HpCDD	22	J-5BH
	M3160821B02	08/21/16	OCDF	21	J-5BH
	M3160821B08	08/21/16	OCDF	23	J-5BH
B.650100	M3160821C15	08/21/16	OCDF	23	J-5BH
B6F0192 &			1,2,3,7,8,9-HxCDD	21	J-5BH
∝ B6F5904			1,2,3,6,7,8-HxCDD	21	J-5BH
2013301	M3160823B15	08/23/16	OCDF	24	J-5BH
			2,3,7,8-TCDD	25	J-5BH
			1,2,3,7,8,9-HxCDD	22	J-5BH
			1,2,3,6,7,8-HxCDD	24	J-5BH

# Method Blanks

In order to assess the impact of blank contamination on the reported sample results, action levels were established at five times the blank concentrations. If the concentrations in the associated field samples were less than the action levels, the results were qualified as not detected (U-7) at the reported concentrations. Method Blanks were reported using a sample volume of 1 L. Action levels were adjusted to account for field sample volumes of approximately 200 L and equipment blank volumes of approximately 10 - 20 L.

The laboratory assigned an "EMPC" flag to an analyte result when a peak was detected but did not meet identification criteria. These values cannot be considered as positive identifications, but are "estimated maximum possible concentrations". When a result in the method blank had an "EMPC" flag, the result was treated as not-detected at an elevated detection limit; therefore, no action level was established for these analytes. Blank qualifiers are not assigned to homolog groups.

Although several target analytes were detected in the method blanks, only the following results required qualified based on method blank contamination:

SDG	Analyte	Samples	Qualifier
B6E4851	OCDD	SW026	U-7
	1,2,3,7,8,9-HxCDF	SW002, SW020, SW008, SW023	U-7
B6F0192	OCDD	SW054, SW055	U-7
	1,2,3,4,6,7,8-HpCDF	SW030, SW039, SW048, SW051, SW036, SW045, SW042	U-7
B6F5904	OCDD	SW109, SW108, SW028	U-7
	OCDF	SW029	U-7
	1,2,3,4,6,7,8-HpCDF	SW096, SW099, SW102, SW090, SW093	U-7

# Field Blanks

Field blanks were submitted for both the filter and XAD fractions. After qualification based on method blank contamination, any remaining positive results were used to evaluate the effects on the field samples. Action levels were established at five times the blank concentration and were adjusted for the sample volume of ~200 L. Results in the associated samples that were less than the action levels were qualified as not-detected (U-6). No action levels were established for EMPC flagged results in the blanks or for total homolog groups.

*SDG B6E4851:* Two equipment blanks were submitted: SW025 (XAD) and SW026 (filter). After qualification based on the method blanks, a positive result remained for OCDD in Sample SW025. The OCDD result for Sample SW004 was qualified as not-detected (U-6).

*SDG B6F0192:* Two equipment blanks were submitted: SW054 (XAD) and SW055 (filter). After qualification based on the method blanks, no target analytes were detected in these blanks.

*SDG B6F5904*: Two equipment blanks were submitted: SW108 (XAD) and SW109 (filter). Two field blanks were also submitted: SW028 (XAD) and SW029 (filter). After qualification based on the

method blanks, there were positive results for several target analytes in Sample SW029. All associated sample results were greater than the action levels; no qualification of data was necessary.

# Labeled Compounds

For labeled compound recoveries greater than the laboratory upper control limit, associated positive results were estimated (J-13H). No action was taken for non-detects. For labeled compound recoveries that were less than the laboratory lower control limit, associated results were estimated (J/UJ-13L). If the recovery was less than 10%, non-detects were rejected (R-13L) and positive results were estimated (J-13L).

For the filters, each labeled compound was associated with one or more native compounds. For the column samples, each native compound had its own unique labeled compound.

SDG	Sample	Labeled Compound	Associated Native Compound	%R	Qualifier
B6E4851	SW002	13C-1,2,3,6,7,8-HxCDF	1,2,3,4,7,8-HxCDF		UJ-13L
			1,2,3,6,7,8-HxCDF	26	J-13L
			2,3,4,6,7,8-HxCDF		UJ-13L
	SW004	13C-1,2,3,6,7,8-HxCDF	1,2,3,6,7,8-HxCDF	3	R-13L
			1,2,3,4,7,8-HxCDF		UJ-13L
	SW005	13C-1,2,3,6,7,8-HxCDF	1,2,3,6,7,8-HxCDF	13	UJ-13L
			2,3,4,6,7,8-HxCDF		UJ-13L
	SW020	13C-1,2,3,6,7,8-HxCDF	1,2,3,4,7,8-HxCDF	22	J-13L
			1,2,3,6,7,8-HxCDF		UJ-13L
			2,3,4,6,7,8-HxCDF		J-13L
	SW014	13C-1,2,3,6,7,8-HxCDF	1,2,3,4,7,8-HxCDF	38	J-13L
			1,2,3,6,7,8-HxCDF		J-13L
			2,3,4,6,7,8-HxCDF		J-13L
	SW026	13C-1,2,3,6,7,8-HxCDF	1,2,3,4,7,8-HxCDF		UJ-13L
			1,2,3,6,7,8-HxCDF	35	UJ-13L
			2,3,4,6,7,8-HxCDF		UJ-13L
	SW008	13C-1,2,3,6,7,8-HxCDF	1,2,3,4,7,8-HxCDF	30	J-13L
			1,2,3,6,7,8-HxCDF		J-13L
			2,3,4,6,7,8-HxCDF		UJ-13L
			1,2,3,4,7,8-HxCDF		J-13L
	SW023	13C-1,2,3,6,7,8-HxCDF	1,2,3,6,7,8-HxCDF	11	J-13L
			2,3,4,6,7,8-HxCDF		UJ-13L

The following labeled compound recovery outliers resulted in qualification of data:

#### Laboratory Control Samples

The laboratory analyzed laboratory control sample/laboratory control sample duplicates (LCS/LCSD) with each batch. For recoveries less than the laboratory lower control limit, associated results were estimated (J/UJ-10L) to indicate a potential low bias. For recoveries greater than the laboratory upper control limit only positive results in the associated samples were estimated (J-10L) to indicate a potential high bias. No action was taken if only one of the LCS or LCSD recoveries was outside of the control limits.

The laboratory control limit for the LCS/LCSD relative percent difference (RPD) is 35%. If the RPD value was greater than the control limit, associated positive results were estimated (J-9). For each LCS/LCSD, there were a subset of congeners where the lab reported value as "NC" (not calculable as the results were <5x RL). This is not applicable to the LCS/LCSD samples. During validation, the RPD values for the "NC" flagged congeners were calculated from the raw data.

The following LCS/LCSD recovery and RPD outliers resulted in qualification of data:

*SDG B6E4851:* For the filter samples, the %R values for 2,3,4,6,7,8-HxCDF were greater than the upper control limit. Associated positive results were estimated (J-10H).

For the XAD samples, the RPD values for 1,2,3,7,8,9-HxCDF, OCDF, and 1,2,3,7,8,9-HxCDD were greater that the control limit. Associated positive results were estimated (J-9).

#### Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicates were not analyzed and are not required by the method. Laboratory precision and accuracy were evaluated using the labeled compound recoveries and the LCS/LCSD recovery and RPD values.

#### Laboratory Duplicate

Laboratory duplicates were not analyzed. Precision was evaluated from the LCS/LCSD and field duplicate RPD values.

#### **Field Duplicates**

For results greater than 5x the detection limit, the RPD control limit is 35%. For results less than 5x the detection limit, the difference between result should be less than the detection limit.

No data were qualified based on field duplicate outliers; however, data users should take field precision into account when interpreting sample data. Field duplicates and any outliers are noted below.

*SDG B6E4851*: One field duplicate pair was submitted for the XAD samples, SW0007 and SW022, and one field duplicate pair was submitted for the filter samples, SW008 and SW023.

For samples SW008 and SW023, the RPD values for 1,2,3,4,7,8,9-HpCDF, , 2,3,4,6,7,8-HxCDF, Total HxCDFs and Total PeCDDs, and the difference value for 1,2,3,7,8,9-HxCDF did not meet the precision criteria.

*SDG B6F0192:* One field duplicate pair was submitted for the XAD samples, SW036 and SW051, and one field duplicate pair was submitted for the filter samples, SW037 and SW052.

For samples SW036 and SW051, the difference values for 1,2,3,4,6,7,8-HpCDF and Total TCDD were greater than the control limit.

For Samples SW037 and SW052, the RPD values for 1,2,3,6,7,8-HxCDD and Total PeCDF, and the difference values for 1,2,3,4,7,8,9-HpCDF and 1,2,3,4,7,8-HxCDD were greater than the control limits.

*SDG B6F5904*: One field duplicate pair was submitted for the XAD samples, SW090 and SW093, and one field duplicate pair was submitted for the filter samples, SW091 and SW094.

For samples SW090 and SW093, the RPD values for 1,2,3,4,6,7,8-HpCDD, OCDD, OCDF, total HpCDD, total HpCDF, and total HxCDD, did not meet precision criteria.

#### **Reported Results**

The detection limits did not meet the target detection limits specified in the sampling and analysis plan (SAP).

#### **Compound Identification**

For estimated maximum potential concentration (EMPC) values, the lab footnoted the cause for the ion ration not meeting criteria. Some of these results were reported by the laboratory as not-detected at an elevated detection limit and some were reported as and estimated positive result. Because the ion abundance ratio is the primary identification criterion for high resolution mass spectroscopy, an outlier indicates that the reported result may be a false positive. When ion ratios did not meet the acceptance criteria and the lab reported the results as hits, the results were qualified as not detected (U-25) at the reported concentration. No further action was taken for results that the lab reported as not-detected.

The method requires the confirmation of 2,3,7,8-TCDF using an alternate GC column as the DB5 column that is typically used cannot fully separate 2,3,7,8-TCDF from closely eluting non-target TCDF isomers. The laboratory performed confirmation on a DB-225 column as required; both sets of result were reported. When confirmation was done, results from the DB-5 column were flagged do-not-report (DNR-11).

### **Calculation Verification**

*SDG B6E4851:* Several results were verified by recalculation from the raw data. There was a sample result calculation error found for the filter samples. The laboratory corrected and resubmitted the PDF and the EDD. The laboratory also checked for the calculation error in the other two SDGs. One

error was found for SDG B6F1992. The PDF and EDD were resubmitted for this SDG. No other calculation or transcription errors were found.

#### OVERALL ASSESSMENT

As was determined by this evaluation, the laboratory followed the specified analytical method. With the exceptions noted above, accuracy was acceptable as demonstrated by the labeled compound and LCS/LCSD recoveries and precision was acceptable as demonstrated by the LCS/LCSD and field duplicate RPD values.

Detection limits were elevated based on ion ratio outliers, method blank contamination, and field blank contamination. Data were estimated due to CCAL %D, labeled compound %R, LCS/LCSD %R, and LCS/LCSD RPD outliers.

Where 2,3,7,8-TCDF was reported form both columns, the results from the DB5 column were flagged do-not-report (DNR-11). One result was rejected due to a labeled compound %R value that was less than 10%. Results that have been flagged do-not-report or that have been rejected should not be used for any purpose.

All other data, as qualified, are acceptable for use.

## DATA VALIDATION REPORT San Jacinto River Waste Pits 2016 Surface Water Sampling Conventional Parameters

This report documents the review of analytical data from the analyses of surface water samples and the associated field and laboratory quality control (QC) samples. ALS Environmental, Kelso, Washington, analyzed the samples. Refer to the **Sample Index** for a complete list of samples.

SDG	Number of Samples	VALIDATION LEVEL
K1607776	7 Water & 1 Field Dup & 1 Rinse Blank	EPA Stage 3
K1608119	7 Water & 1 Field Dup & 1 Rinse Blank	EPA Stage 2B
K1608356	7 Water & 1 Field Dup & 1 Rinse Blank	EPA Stage 2B

The analytical tests that were performed are summarized below.

Parameter	Method
Total Dissolved Solids	SM2540C
Total Suspended Solids	SM2540D
Total & Dissolved Organic Carbon	SM5310C

#### DATA PACKAGE COMPLETENESS

The laboratory submitted all required deliverables. The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative.

#### EDD TO HARDCOPY VERIFICATION

Ten percent (10%) of the results in the laboratory EDD were verified by comparison to the laboratory data package. No errors were noted.

#### TECHNICAL DATA VALIDATION

The QC requirements that were reviewed are listed below:

1	Sample Receipt, Preservation, and Holding Times	$\checkmark$	Matrix Spikes (MS)
$\checkmark$	Initial Calibration	$\checkmark$	Laboratory Replicates
$\checkmark$	Calibration Verification	1	Field Replicates
$\checkmark$	Laboratory Blanks	1	Reporting Limits
2	Field Blanks	2	Reported Results
$\checkmark$	Laboratory Control Samples (LCS)	1	Calculation Verification

 $\checkmark$  Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed below.

#### Sample Receipt, Preservation, and Holding Times

The validation guidance documents state that the cooler temperatures should be within an advisory temperature range of 2° to 6°C. With the following exception noted below, the laboratory received the sample coolers within the advisory temperature range.

*SDG K1608356*: One sample cooler temperature was less than the lower control limit at 0.0°C. This outlier did not impact data quality; no data were qualified.

For Sample SW089, the container for dissolved organic carbon did not arrive at the laboratory. The laboratory filtered an aliquot from another container for that analysis.

#### Field Blanks

**SDG K1607776:** One rinse blank, SW027, was included with this data set. Dissolved organic carbon (DOC) was detected. All sample results less than the 5x action level were qualified as not detected (U-6).

*SDG K1608119:* One rinse blank, SW056, was included with this data set. DOC was detected. All sample results less than the 5x action level were qualified as not detected (U-6).

*SDG K1608356*: One rinse blank, SW110, was included with this data set. DOC was detected. All sample results less than the 5x action level were qualified as not detected (U-6).

#### **Field Replicates**

*SDG K1607776*: Two samples were identified as field duplicates, SW009 & SW024. All acceptance criteria were met.

*SDG K1608119*: Two samples were identified as field duplicates, SW038 & SW053. All acceptance criteria were met.

*SDG K1608356*: Two samples were identified as field duplicates, SW092 & SW095. All acceptance criteria were met.

#### **Reporting limits**

*All SDGs:* Most dissolved organic carbon (DOC) results were qualified as not detected (U-6) because of field blank contamination. The resulting elevated reporting limits are greater than the QAPP required reporting limit of 0.5 µg/L.

#### **Reported Results**

*SDG K1607776*: For Sample SW006, the positive result for dissolved organic carbon was greater than positive result for total organic carbon. The RPD value was greater than the acceptance criteria of 20%. Both results for this sample were estimated (J-14).

#### **Calculation Verification**

*SDG K1607776*: Several results were verified by recalculation from the raw data. No calculation or transcription errors were noted.

#### OVERALL ASSESSMENT

As determined by this evaluation, the laboratory followed the specified analytical methods. Accuracy was acceptable as demonstrated by the laboratory control sample percent recovery values. With the exceptions noted above, precision was acceptable as demonstrated by the laboratory and field replicate %RSD values.

Detection limits for DOC were elevated due to field blank contamination. Total and Dissolved Organic Carbon results were estimated due to total and dissolved value precision outliers.

All data, as qualified, are acceptable for use.



## **APPENDIX A**

# DATA QUALIFIER DEFINITIONS REASON CODES AND CRITERIA TABLES

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## DATA VALIDATION QUALIFIER CODES Based on National Functional Guidelines

The following definitions provide brief explanations of the qualifiers assigned to results in the data review process.

U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents the approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

The following is an EcoChem qualifier that may also be assigned during the data review process:

DNR Do not report; a more appropriate result is reported from another analysis or dilution.

### DATA QUALIFIER REASON CODES

Group	Code	Reason for Qualification
Sample Handling	1	Improper Sample Handling or Sample Preservation (i.e., headspace, cooler temperature, pH, summa canister pressure); Exceeded Holding Times
	24	Instrument Performance (i.e., tune, resolution, retention time window, endrin breakdown, lock-mass)
	5A	Initial Calibration (RF, %RSD, r <sup>2</sup> )
Instrument Performance	5B	Calibration Verification (CCV, CCAL; RF, %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	5C	Initial Calibration Verification (ICV %D, %R) Use bias flags (H,L) <sup>1</sup> where appropriate
	6	Field Blank Contamination (Equipment Rinsate, Trip Blank, etc.)
Blank Contamination	7	Lab Blank Contamination (i.e., method blank, instrument blank, etc.) Use low bias flag (L) <sup>1</sup> for negative instrument blanks
	8	Matrix Spike (MS and/or MSD) Recoveries Use bias flags (H,L) <sup>1</sup> where appropriate
	9	Precision (all replicates: LCS/LCSD, MS/MSD, Lab Replicate, Field Replicate)
Precision and Accuracy	10	Laboratory Control Sample Recoveries (a.k.a. Blank Spikes) Use bias flags (H,L) <sup>1</sup> where appropriate
	12	Reference Material Use bias flags (H,L) <sup>1</sup> where appropriate
	13	Surrogate Spike Recoveries (a.k.a. labeled compounds, recovery standards) Use bias flags (H,L) <sup>1</sup> where appropriate
	16	ICP/ICP-MS Serial Dilution Percent Difference
	17	ICP/ICP-MS Interference Check Standard Recovery Use bias flags (H,L) <sup>1</sup> where appropriate
Interferences	19	Internal Standard Performance (i.e., area, retention time, recovery)
	22	Elevated Detection Limit due to Interference (i.e., chemical and/or matrix)
	23	Bias from Matrix Interference (i.e. diphenyl ether, PCB/pesticides)
	2	Chromatographic pattern in sample does not match pattern of calibration standard
	3	2 <sup>nd</sup> column confirmation (RPD or %D)
Identification and Quantitation	4	Tentatively Identified Compound (TIC) (associated with NJ only)
	20	Calibration Range or Linear Range Exceeded
	25	Compound Identification (i.e., ion ratio, retention time, relative abundance, etc.)
N.C II	11	A more appropriate result is reported (multiple reported analyses i.e., dilutions, re- extractions, etc. Associated with "R" and "DNR" only)
Miscellaneous	14	Other (See DV report for details)
	26	Method QC information not provided

<sup>1</sup>H = high bias indicated

L = low bias indicated

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Waters/Solids $\leq 6^{\circ}C \&$ in the dark Tissues <-10°C & in the dark <b>Preservation Aqueous:</b> If Cl <sub>2</sub> is present Thiosulfate must be added and if pH > 9 it must be adjusted to 7 - 9	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/R(ND) if thiosulfate not added if Cl <sub>2</sub> present; J(pos)/UJ(ND) if pH not adjusted J(pos)/UJ(ND) if temp > 20°C	1	EcoChem PJ, see TM-05
Holding Time	If properly stored, 1 year or: Extraction (all matrices): 30 days from collection Analysis (all matrices): 45 days from extraction	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If not properly stored or HT exceedance: J(pos)/UJ(ND)	1	EcoChem PJ, see TM-05 Gross exceedance = > 1 year 2011 NFG Note: Under CWA, SDWA, and RCRA the HT for H2O is 7 days.
Instrument Performa	nce				
Mass Resolution (Tuning)	PFK (Perfluorokerosene) ≥10,000 resolving power at m/z 304.9824. Exact mass of m/z 380.9760 w/in 5 ppm of theoretical value (380.97410 to 380.97790) . Analyzed prior to ICAL and at the start and end of each 12 hr. shift.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	R(pos/ND) all analytes in all samples associated with the tune	24	Notify PM
Windows Defining Mix	Peaks for first and last eluters must be within established retention time windows for each selector group (chlorination level)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If peaks are not completely within windows (clipped): If natives are ok, J(pos)/UJ(ND) homologs (Totals) If natives are affected, R all results for that selector group	24	Notify PM
Column Performance Mix	Both mixes must be analyzed before ICAL and CCAL Valley < 25% (valley = (x/y)*100%) where x = ht. of TCDD (or TCDF) & y = baseline to bottom of valley For all isomers eluting near the 2378-TCDD (TCDF) peak (TCDD only for 8290)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if valley > 25%	24	EcoChem PJ, see TM-05, Rev. 2; Note: TCDF is evaluated only if second column confirmation is performed
Initial Calibration Sensitivity	S/N ratio > 10 for all native and labeled compounds in CS1 std.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5A	
Initial Calibration <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If 2 or more ion ratios are out for one compound in ICAL, J(pos)	5A	EcoChem PJ, see TM-05, Rev. 2

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Instrument Performa	nce (continued)				
Initial Calibration (Minimum 5 stds.)	%RSD < 20% for native compounds %RSD <30% for labeled compounds (%RSD < 35% for labeled compounds under 1613b)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) natives if %RSD > 20%	5A	
Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD >25 min on DB5 & >15 min on DB-225	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action		EcoChem PJ, see TM-05, Rev. 2
Continuing Calibration (Prior to each 12 hr. shift) <b>Sensitivity</b>	S/N ratio for CS3 standard > 10	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If <10, elevate Det. Limit or R(ND)	5B	
Continuing Calibration (Prior to each 12 hr. shift) <b>Selectivity</b>	Ion Abundance ratios within QC limits (Table 8 of method 8290) (Table 9 of method 1613B)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	For congener with ion ratio outlier, J(pos) natives in all samples associated with CCAL. No action for labeled congener ion ratio outliers.	25	EcoChem PJ, see TM-05
Continuing Calibration (Prior to each 12 hr.	%D+/-20% for native compounds %D +/-30% for labeled compounds (Must meet limits in Table 6, Method 1613B) If %D in the closing CCAL are within 25%/35%, the mean RF from the two CCAL may be used to calculate samples (Section 8.3.2.4 of 8290).	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Labeled compounds: Narrate, no action. Native compounds: 1613: J(pos)/UJ(ND)if %D is outside Table 6 limits J(pos)/R(ND) if %D is +/-75% of Table 6 limits 8290: J(pos)/UJ(ND) if %D = 20% - 75% J(pos)/R(ND) if %D > 75%	5B (H,L) <sup>3</sup>	
shift) Stability	Absolute RT of <sup>13</sup> C <sub>12</sub> -1234-TCDD and <sup>13</sup> C <sub>12</sub> -123789-HxCDD should be ± 15 seconds of ICAL RRT for all other compounds must meet criteria listed in Table 2 Method 1316.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate, no action	5B	EcoChem PJ, see TM-05
Blank Contamination					
Method Blank (MB)	MB: One per matrix per batch of (of ≤ 20 samples) No detected compounds > RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U(pos) if result is < 5X action level.	7	Hierarchy of blank review: #1 - Review MB, qualify as needed
Field Blank (FB)	FB: frequency as per QAPP No detected compounds > RL	WELTOU	U(pos) if result is < 5X action level.	6	#2 - Review FB , qualify as needed

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Precision and Accura	cy				
MS/MSD (recovery)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of $\leq$ 20 samples)	EcoChem standard policy	J(pos) if both %R > UCL - high bias J(pos)/UJ(ND) if both %R < LCL - low bias J(pos)/R(ND) if both %R < 10% - very low bias	8 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria. No action if parent concentration is >4x the amount spiked.
	Use most current laboratory control limits		J(pos)/UJ(ND) if one > UCL & one < LCL, with no bias <b>PJ if only one %R outlier</b>		Qualify parent sample only unless other QC indicates systematic problems.
MS/MSD (RPD)	MS/MSD not typically required for HRMS analyses. If lab analyzes MS/MSD then one set per matrix per batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos) in parent sample if RPD > CL	9	Qualify parent sample only.
LCS (or OPR)	One per lab batch (of ≤ 20 samples) Use most current laboratory control limits <b>or</b> Limits from Table 6 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	10 (H,L) <sup>3</sup>	No action if only one spike %R is outside criteria, when LCSD is analyzed. Qualify all associated samples.
LCS/LCSD (RPD)	LCSD not typically required for HRMS analyses. One set per matrix and batch of 20 samples RPD < 35%	Method <sup>(2)</sup> Ecochem standard policy	J(pos) assoc. compound in all samples if RPD > CL	9	Qualify all associated samples.
Lab Duplicate (RPD)	Lab Dup not typically required for HRMS analyses. One per lab batch (of ≤ 20 samples) Use most current laboratory control limits	EcoChem standard policy	J(pos)/UJ(ND) if RPD > CL	9	
Labeled Compounds (Internal Standards)	Added to all samples %R = 40% - 135% in all samples 8290 %R must meet limits in Table 7 Method 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos) if %R > UCL - high bias J(pos)/UJ(ND) if %R < LCL - low bias J(pos)/R(ND) if %R < 10% - very low bias	13 (H,L) <sup>3</sup>	
Field Duplicates	Solids: RPD <50% OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Narrate and qualify if required by project	9	Use professional judgment

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason	Discussion and Comments
Compound ID and Ca	•			Code	
Quantitation/ Identification	All ions for each isomer must maximize within ± 2 seconds. S/N ratio >2.5 Ion ratios must meet criteria listed in Table 8 Method 8290, or Table 9 of 1613B; RRTs w/in limits in Table 2 of 1613B	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Narrate in report; qualify if necessary NJ(pos) for retention time outliers. U(pos) for ion ratio outliers.	25	EcoChem PJ, see TM-05
EMPC (estimated maximum possible concentration)	If quantitation identification criteria are not met, laboratory should report an EMPC value.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If laboratory correctly reported an EMPC value, qualify the native compound U(pos) to indicate that the value is a detection limit and qualify total homolog groups J (pos)	25	Use professional judgment See TM-18
Interferences	Interferences from chlorodiphenyl ether compounds	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if present	23	See TM-16
Interferences	Lock masses must not deviate ± 20% from values in Table 8 of 1613B	Method (2)	J(pos)/UJ(ND) if present	24	See TM-17
Second Column Confirmation	All 2,3,7,8-TCDF hits must be confirmed on a DB-225 (or equiv) column. All QC criteria must also be met for the confirmation analysis.	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Report the DB-225 value. If not performed use PJ.	3	DNR-11 DB5 result if both results from both columns are reported. <b>EcoChem PJ, see TM-05</b>
Calculation Check	Check 10% of field & QC sample results	EcoChem standard policy	Contact laboratory for resolution and/or corrective action	na	Full data validation only.
Electronic Data Delive	erable (EDD)				
Verification of EDD to hardcopy data	EcoChem verify @ 10% unless problems noted; then increase level up to 100% for next several packages.		Depending on scope of problem, correct at EcoChem (minor issues) to resubmittal by laboratory (major issues).	na	EcoChem Project Manager and/or Database Administrator will work with lab to provide long-term corrective action.
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	Standard reporting policy	Use "DNR" to flag results that will not be reported.	11	

(pos) - positive (detected) results; (ND) - not detected results

<sup>1</sup> National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) & Chlorinated Dibenzofurans (CDFs) Data Review, September 2011

<sup>2</sup> Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), USEPA SW-846, Method 8290

<sup>2</sup> EPA Method 1613, Rev.B, Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGS/HRMS, October 1994

<sup>3</sup> NFG 2013 suggests using "+ / -" to indicate bias; EcoChem has chosen "H" = high bias indicated; "L" = low bias indicated.

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments	
Sample Handling						
Cooler/Storage Temperature Preservation	Cooler temperature: 4°C±2°C Preservation: Analyte/Method Specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if preservation requirements not met	1	Use <b>PJ</b> to qualify for cooler temp outliers.	
Holding Time	Analyte/Method Specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if holding time exceeded	1		
Instrument Performa	nce	•				
Initial Calibration (ICAL)	blank + multiple standards as per method requirements r ≥ 0.995	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) for r < 0.995	5A		
Initial Calibration Verification (ICV)	Independent source analyzed immediately after calibration %R method specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < lower control limit (LCL) J (pos) if %R > upper control limit (UCL)	5A (H,L) <sup>3</sup>	Qualify all samples in run	
Continuing Calibration Verification (CCV)	Immediately following ICV, every 10 samples, and end of run %R method specific	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J(pos)/UJ(ND) if %R < LCL J(pos) if %R > UCL	5B (H,L) <sup>3</sup>	Qualify samples bracketed by CCV outliers	
Blank Contamination	Jank Contamination					
Method Blank (MB)	One per matrix per batch of (of $\leq$ 20 samples) Blank conc < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U (pos) if result is < 5X method blank concentration	7	Refer to <b>TM-02</b> for additional information. Blank Evaluation based on NFG 1994	

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Instrument Blanks (ICB/CCB)	After each ICV & CCV   blank concentration   < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	Action level is 5x absolute value of blank conc. For positive blanks: U (pos) results < action level For negative blanks: J (pos)/UJ (ND) results < action level	Pos Blanks: 7 Neg Blanks: 7L <sup>3</sup>	Use blanks bracketing samples for Qualification Refer to <b>TM-02</b> for additional information. <b>Hierarchy of blank review:</b> <b>#1 - Review MB, qualify as needed</b> <b>#2 - Review IB , qualify as needed</b> <b>#3 - Review FB , qualify as needed</b>
Field Blank (FB)	Blank conc < MDL	EcoChem standard policy	U (pos) if result is < 5x action level, as per analyte.	6	Qualify in associated field samples only. Refer to <b>TM-02</b> for additional information.
Precision and Accura	су				
Laboratory Control Sample (LCS)	One per matrix per batch (of ≤ 20 samples) %R within Method control limits (or Laboratory control limtis if none specified in method)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < LCL J (pos) if %R > UCL	10 (H,L) <sup>3</sup>	Qualify all samples in batch QAPP may have overriding accuracy limits.
Reference Materials (RM, CRM, SRM)	Result $\pm 20\%$ of the 95% confidence interval of the true value for analytes	EcoChem standard policy	J (pos)/UJ (ND) if < LCL J (pos) if > UCL	12 (H,L) <sup>3</sup>	QAPP may have overriding accuracy limits. Some manufacturers may have different RM control limits

#### Conventional Methods with Instrument Calibrations (i.e., Ion Chromatography, Total Organic Carbon) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	Acceptance Criteria	Source of Criteria	Action for Non-Conformance	Reason Code	Discussion and Comments
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Where applicable to method; MSD may not be required One per matrix per batch (of ≤ 20 samples) For samples <4x spike level, %R within method control limits (or Laboratory control limits if none specified in method)	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if %R < LCL J (pos) if %R > UCL	8 (H,L)3	Qualify all samples in batch No action if native analyte concentration ≥ 4x spike added. Qualify all samples in batch. QAPP may have overriding accuracy limits.
Laboratory Duplicate (or MS/MSD)	One per matrix per batch (of ≤ 20 samples) RPD ≤ 20% for results ≥ 5x RL Solids: difference < 2X RL for results < 5X RL Aqueous: difference < 1X RL for results < 5X RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if RPD > 20% or if difference > control limit	9	Qualify all samples in batch. QAPP may have overriding precision limits.
Field Duplicate	Solids: RPD <50% (for results ≥ 5x RL) OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% (for results ≥ 5x RL) OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Qualify only parent and field duplicate samples J (pos)/UJ (ND)	9	QAPP may have overriding precision limits. Client/QAPP may not require qualification based on field precision.
Compound Quantita	tion				
Linear Range	Sample concentrations less than highest calibration standard	NFG <sup>(1)</sup> Method <sup>(2)</sup>	If result exceeds linear range & sample was not diluted J (pos)	20	
Dilutions, Re- extractions and/or Reanalyses	Report only one result per analyte	EcoChem standard policy	Use "DNR" to flag results that will not be reported.	11	TM-04 EcoChem Policy for Rejection/Selection Process for Multiple Results

<sup>1</sup> National Functional Guidelines for Inorganic Superfund Data Review, January 2010.

<sup>2</sup> SW846 or EPA Standard Methods

<sup>3</sup> "H" = high bias indicated; "L" = low bias indicated

(pos): Positive Result (ND): Not Detected

#### Conventional Methods by Gravimetric Analysis (i.e., Total Solids, Total Dissolved Solids, Total Suspended Solids, Grain Size) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	EcoChem Acceptance Criteria	Source of Criteria	EcoChem Action for Non- Conformance	Reason Code	Discussion and Comments
Sample Handling					
Cooler/Storage Temperature Preservation	Cooler temperature: 4°C±2°C Preservation: Analyte/Method Specific	Method <sup>(1)</sup> NFG <sup>(2)</sup>	J (pos)/UJ (ND) if preservation requirements not met	1	Use <b>PJ</b> to qualify for cooler temp outliers.
Holding Time	Analyte/Method Specific	Method NFG <sup>(2)</sup>	J (pos)/UJ (ND) if holding time exceeded	1	
<b>Blank Contamination</b>					
Method Blank (MB)	If required by method,one per matrix per batch of (of ≤ 20 samples) Blank conc < MDL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	U (pos) if result is < 5X method blank concentration	7	Refer to <b>TM-02</b> for additional information. Blank Evaluation based on NFG 1994
Precision and Accurat	cy			•	
LCS (If appropriate to method)	One per matrix per batch (of ≤ 20 samples) %R between 80-120%	Method <sup>(2)</sup>	J (pos)/R (ND) if %R <50% J (pos)/UJ (ND) if %R 50% - 79% J (pos) if %R > 120%	10 (H,L) <sup>3</sup>	Qualify all samples in batch QAPP may have overriding accuracy limits.
Reference Material (RM, SRM, or CRM)	Result $\pm 20\%$ of the 95% confidence interval of the true value for analytes	EcoChem standard policy	J (pos)/UJ (ND) if < LCL J (pos) if > UCL	12 (H,L) <sup>3</sup>	QAPP may have overriding accuracy limits. Some manufacturers may have different RM control limits

#### **Conventional Methods by Gravimetric Analysis** (i.e., Total Solids, Total Dissolved Solids, Total Suspended Solids, Grain Size) (Based on Inorganic NFG 2010 and EPA methods)

QC Element	EcoChem Acceptance Criteria	Source of Criteria	EcoChem Action for Non- Conformance	Reason Code	Discussion and Comments
Laboratory Duplicate	One per matrix per batch (of $\leq 20$ samples) RPD $\leq 20\%$ for results $\geq 5x$ RL Solids: difference $< 2X$ RL for results $< 5X$ RL Aqueous: difference $< 1X$ RL for results $< 5X$ RL	NFG <sup>(1)</sup> Method <sup>(2)</sup>	J (pos)/UJ (ND) if RPD > 20% For Grain Size, no action if results for fraction are less than 5%	9	Qualify all samples in batch, except Grain Size - qualify parent only. QAPP may have overriding precision limits.
Field Duplicate	Solids: RPD <50% (for results $\ge$ 5x RL) OR difference < 2X RL (for results < 5X RL) Aqueous: RPD <35% (for results $\ge$ 5x RL) OR difference < 1X RL (for results < 5X RL)	EcoChem standard policy	Qualify only parent and field duplicate samples J (pos)/UJ (ND)	9	QAPP may have overriding precision limits. Client/QAPP may not require qualification based on field precision.
Compound Quantitati	on	•			
Dilutions, Re-extractions and/or Reanalyses	Dilutions, extractions and/or Report only one result per analyte per sample		Use "DNR" to flag results that will not be reported.	11	

<sup>1</sup> National Functional Guidelines for Inorganic Superfund Data Review, January 2010.
 <sup>2</sup> SW846 or EPA Standard Methods

(pos): Positive Result (ND): Not Detected

<sup>3</sup> "H" = high bias indicated; "L" = low bias indicated



## **APPENDIX B**

## **QUALIFIED DATA SUMMARY TABLE**

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
K1607776	SW003	K1607776-002	SM5310C	Organic carbon	10.1	mg/L		U	6
K1607776	SW021	K1607776-003	SM5310C	Organic carbon	7.4	mg/L		U	6
K1607776	SW012	K1607776-004	SM5310C	Organic carbon	6.55	mg/L		U	6
K1607776	SW015	K1607776-005	SM5310C	Organic carbon	2.92	mg/L		U	6
K1607776	SW018	K1607776-006	SM5310C	Organic carbon	3.18	mg/L		U	6
K1607776	SW009	K1607776-007	SM5310C	Organic carbon	5.11	mg/L		U	6
K1607776	SW024	K1607776-008	SM5310C	Organic carbon	6.39	mg/L		U	6
K1607776	SW006	K1607776-001	SM5310C	Organic carbon	7.54	mg/L		J	14
K1607776	SW006	K1607776-001	SM5310C	Organic carbon	15.3	mg/L		J	14
K1608119	SW035	K1608119-001	SM5310C	Organic carbon	5.99	mg/L		U	6
K1608119	SW050	K1608119-003	SM5310C	Organic carbon	5.6	mg/L		U	6
K1608119	SW041	K1608119-004	SM5310C	Organic carbon	5.6	mg/L		U	6
K1608119	SW053	K1608119-005	SM5310C	Organic carbon	4.83	mg/L		U	6
K1608119	SW038	K1608119-006	SM5310C	Organic carbon	4.79	mg/L		U	6
K1608119	SW047	K1608119-008	SM5310C	Organic carbon	2.09	mg/L		U	6
K1608119	SW044	K1608119-009	SM5310C	Organic carbon	2.15	mg/L		U	6
K1608356	SW089	K1608356-002	SM5310C	Organic carbon	5.1	mg/L		U	6
K1608356	SW086	K1608356-003	SM5310C	Organic carbon	8	mg/L		U	6
K1608356	SW107	K1608356-004	SM5310C	Organic carbon	2.8	mg/L		U	6
K1608356	SW098	K1608356-005	SM5310C	Organic carbon	2.33	mg/L		U	6
K1608356	SW101	K1608356-006	SM5310C	Organic carbon	1.9	mg/L		U	6
K1608356	SW104	K1608356-007	SM5310C	Organic carbon	2.1	mg/L		U	6
K1608356	SW092	K1608356-008	SM5310C	Organic carbon	2.62	mg/L		U	6
K1608356	SW095	K1608356-009	SM5310C	Organic carbon	2.8	mg/L		U	6
B6E4851	SW001	CRT810	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.02	pg/L	J	U	25
B6E4851	SW001	CRT810	EPA1613B	Octachlorodibenzofuran	0.051	pg/L	J	J	9
B6E4851	SW002	CRT811	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.0218	pg/L	J	U	25
B6E4851	SW002	CRT811	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.044	pg/L	J	UJ	13L,25
B6E4851	SW002	CRT811	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.018	pg/L	J	J	5BH,13L
B6E4851	SW002	CRT811	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.022	pg/L	J	U	7
B6E4851	SW002	CRT811	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.0094	pg/L	J	U	25

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
B6E4851	SW002	CRT811	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.02	pg/L	J	UJ	13L,25
B6E4851	SW002	CRT811	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.175	pg/L		DNR	11
B6E4851	SW004	CRT812	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0099	pg/L	J	U	25
B6E4851	SW004	CRT812	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.03	pg/L	U	R	13L
B6E4851	SW004	CRT812	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.0086	pg/L	J	J	9
B6E4851	SW004	CRT812	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.104	pg/L		J	9
B6E4851	SW004	CRT812	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.0181	pg/L	J	U	25
B6E4851	SW004	CRT812	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.0594	pg/L	J	J	5BL
B6E4851	SW004	CRT812	EPA1613B	Octachlorodibenzo-p-dioxin	3.36	pg/L	-	U	6
B6E4851	SW004	CRT812	EPA1613B	Octachlorodibenzofuran	0.033	pg/L	J	J	5BL,9
B6E4851	SW004	CRT812	EPA1613B	Tetrachlorodibenzodioxin (Total)	0.0181	pg/L	J	U	25
B6E4851	SW005	CRT813	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.015	pg/L	J	U	25
B6E4851	SW005	CRT813	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.032	pg/L	U	UJ	13L
B6E4851	SW005	CRT813	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.03	pg/L	U	UJ	13L
B6E4851	SW005	CRT813	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.0565	pg/L	J	U	25
B6E4851	SW005	CRT813	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.032	pg/L	U	UJ	13L
B6E4851	SW005	CRT813	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.124	pg/L		DNR	11
B6E4851	SW010	CRT814	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.035	pg/L	J	U	25
B6E4851	SW010	CRT814	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.112	pg/L		DNR	11
B6E4851	SW010	CRT814	EPA1613B	Octachlorodibenzofuran	0.152	pg/L	J	J	9
B6E4851	SW011	CRT815	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.0288	pg/L	J	U	25
B6E4851	SW011	CRT815	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.017	pg/L	J	U	25
B6E4851	SW011	CRT815	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.029	pg/L	J	J	10H
B6E4851	SW011	CRT815	EPA1613B	2,3,4,7,8-Pentachlorodibenzofuran	0.02	pg/L	J	U	25
B6E4851	SW011	CRT815	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.466	pg/L		DNR	11
B6E4851	SW019	CRT816	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.046	pg/L	J	U	25
B6E4851	SW019	CRT816	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.131	pg/L		DNR	11
B6E4851	SW019	CRT816	EPA1613B	Octachlorodibenzofuran	0.148	pg/L	J	J	9
B6E4851	SW020	CRT817	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.125	pg/L		J	13L
B6E4851	SW020	CRT817	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.04	pg/L	J	UJ	13L,25
B6E4851	SW020	CRT817	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.047	pg/L	J	U	7

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
B6E4851	SW020	CRT817	EPA1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin	0.0215	pg/L	J	U	25
B6E4851	SW020	CRT817	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.093	pg/L	J	J	10H,13L
B6E4851	SW020	CRT817	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.627	pg/L		DNR	11
B6E4851	SW013	CRT818	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.014	pg/L	J	J	9
B6E4851	SW013	CRT818	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.05	pg/L	J	U	25
B6E4851	SW013	CRT818	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.139	pg/L		DNR	11
B6E4851	SW013	CRT818	EPA1613B	Octachlorodibenzofuran	0.304	pg/L	J	J	9
B6E4851	SW014	CRT819	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.091	pg/L	J	J	13L
B6E4851	SW014	CRT819	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.04	pg/L	J	J	5BH,13L
B6E4851	SW014	CRT819	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.016	pg/L	J	U	25
B6E4851	SW014	CRT819	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.032	pg/L	J	J	10H,13L
B6E4851	SW014	CRT819	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.365	pg/L		DNR	11
B6E4851	SW016	CRT869	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.061	pg/L	J	U	25
B6E4851	SW016	CRT869	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.205	pg/L		DNR	11
B6E4851	SW016	CRT869	EPA1613B	Octachlorodibenzofuran	0.32	pg/L	J	J	9
B6E4851	SW017	CRT871	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.028	pg/L	J	U	25
B6E4851	SW017	CRT871	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.04	pg/L	J	J	5BH
B6E4851	SW017	CRT871	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.02	pg/L	J	U	25
B6E4851	SW017	CRT871	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.455	pg/L		DNR	11
B6E4851	SW026	CRT873	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.27	pg/L	J	U	25
B6E4851	SW026	CRT873	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.18	pg/L	U	UJ	13L
B6E4851	SW026	CRT873	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.16	pg/L	U	UJ	13L
B6E4851	SW026	CRT873	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.18	pg/L	U	UJ	13L
B6E4851	SW026	CRT873	EPA1613B	Heptachlorodibenzofuran (Total)	0.27	pg/L	J	J	25
B6E4851	SW026	CRT873	EPA1613B	Octachlorodibenzo-p-dioxin	1.76	pg/L	J	U	7
B6E4851	SW007	CRT874	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.0132	pg/L	J	J	9
B6E4851	SW007	CRT874	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.0613	pg/L	J	U	25
B6E4851	SW007	CRT874	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.185	pg/L		DNR	11
B6E4851	SW007	CRT874	EPA1613B	Octachlorodibenzofuran	0.193	pg/L	J	J	9
B6E4851	SW008	CRT877	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.0329	pg/L	J	U	25
B6E4851	SW008	CRT877	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.153	pg/L		J	13L

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV	DV
	-	•					g	Qualifier	Reason
B6E4851	SW008	CRT877	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.039	pg/L	J	J	5BH,13L
B6E4851	SW008	CRT877	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.027	pg/L	J	U	7
B6E4851	SW008	CRT877	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.037	pg/L	J	UJ	13L,25
B6E4851	SW008	CRT877	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.975	pg/L		DNR	11
B6E4851	SW022	CRT878	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.0176	pg/L	J	J	9
B6E4851	SW022	CRT878	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.062	pg/L	J	U	25
B6E4851	SW022	CRT878	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.195	pg/L		DNR	11
B6E4851	SW022	CRT878	EPA1613B	Octachlorodibenzofuran	0.211	pg/L	J	J	9
B6E4851	SW023	CRT879	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzofuran	0.188	pg/L		J	13L
B6E4851	SW023	CRT879	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzofuran	0.042	pg/L	J	J	5BH,13L
B6E4851	SW023	CRT879	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzofuran	0.054	pg/L	J	U	7
B6E4851	SW023	CRT879	EPA1613B	2,3,4,6,7,8-Hexachlorodibenzofuran	0.093	pg/L	J	UJ	13L,25
B6E4851	SW023	CRT879	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.871	pg/L		DNR	11
B6F0192	SW033	CSR059	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0176	pg/L	J	U	25
B6F0192	SW033	CSR059	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.041	pg/L	J	U	25
B6F0192	SW033	CSR059	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.14	pg/L		DNR	11
B6F0192	SW033	CSR059	EPA1613B	Heptachlorodibenzofuran (Total)	0.0176	pg/L	J	U	25
B6F0192	SW033	CSR059	EPA1613B	Octachlorodibenzofuran	0.125	pg/L	J	U	25
B6F0192	SW034	CSR060	EPA1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	0.036	pg/L	J	U	25
B6F0192	SW034	CSR060	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.082	pg/L	J	J	5BH
B6F0192	SW034	CSR060	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.165	pg/L		J	5BH
B6F0192	SW034	CSR060	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.153	pg/L		J	5BH
B6F0192	SW034	CSR060	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.367	pg/L		DNR	11
B6F0192	SW034	CSR060	EPA1613B	Octachlorodibenzofuran	2.21	pg/L		J	5BH
B6F0192	SW030	CSR061	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0171	pg/L	J	U	7
B6F0192	SW030	CSR061	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.014	pg/L	J	J	5BH
B6F0192	SW030	CSR061	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.076	pg/L	J	DNR	11
B6F0192	SW030	CSR061	EPA1613B	Octachlorodibenzofuran	0.095	pg/L	J	J	5BH
B6F0192	SW031	CSR062	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.094	pg/L	J	J	5BH
B6F0192	SW031	CSR062	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.194	pg/L		J	5BH
B6F0192	SW031	CSR062	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.134	pg/L		J	5BH

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
B6F0192	SW031	CSR062	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.378	pg/L		DNR	11
B6F0192	SW031	CSR062	EPA1613B	Octachlorodibenzofuran	1.71	pg/L		J	5BH
B6F0192	SW039	CSR063	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0313	pg/L	J	U	7
B6F0192	SW039	CSR063	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.01	pg/L	J	J	5BH
B6F0192	SW039	CSR063	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.012	pg/L	J	J	5BH
B6F0192	SW039	CSR063	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.055	pg/L	J	U	25
B6F0192	SW039	CSR063	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.203	pg/L		DNR	11
B6F0192	SW039	CSR063	EPA1613B	Octachlorodibenzofuran	0.287	pg/L	J	J	5BH
B6F0192	SW040	CSR064	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	4.94	pg/L		J	5BH
B6F0192	SW040	CSR064	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.578	pg/L		DNR	11
B6F0192	SW048	CSR065	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0341	pg/L	J	U	7
B6F0192	SW048	CSR065	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.018	pg/L	J	J	5BH
B6F0192	SW048	CSR065	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.055	pg/L	J	U	25
B6F0192	SW048	CSR065	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.194	pg/L		DNR	11
B6F0192	SW048	CSR065	EPA1613B	Octachlorodibenzofuran	0.243	pg/L	J	J	5BH
B6F0192	SW049	CSR066	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	4.84	pg/L		J	5BH
B6F0192	SW049	CSR066	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.729	pg/L		DNR	11
B6F0192	SW051	CSR067	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.046	pg/L	J	U	7
B6F0192	SW051	CSR067	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.075	pg/L	J	U	25
B6F0192	SW051	CSR067	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.232	pg/L		DNR	11
B6F0192	SW051	CSR067	EPA1613B	Octachlorodibenzofuran	0.262	pg/L	J	J	5BH
B6F0192	SW052	CSR068	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	3.3	pg/L		J	5BH
B6F0192	SW052	CSR068	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.896	pg/L		DNR	11
B6F0192	SW036	CSR069	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.03	pg/L	J	U	7
B6F0192	SW036	CSR069	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.064	pg/L	J	U	25
B6F0192	SW036	CSR069	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.226	pg/L		DNR	11
B6F0192	SW036	CSR069	EPA1613B	Octachlorodibenzofuran	0.212	pg/L	J	J	5BH
B6F0192	SW036	CSR069	EPA1613B	Tetrachlorodibenzodioxin (Total)	0.064	pg/L	J	U	25
B6F0192	SW037	CSR070	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	4.5	pg/L		J	5BH
B6F0192	SW037	CSR070	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	1.04	pg/L		DNR	11
B6F0192	SW045	CSR071	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.065	pg/L	J	U	7

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
B6F0192	SW045	CSR071	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.058	pg/L	J	U	25
B6F0192	SW045	CSR071	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.257	pg/L		DNR	11
B6F0192	SW046	CSR072	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	2.93	pg/L		J	5BH
B6F0192	SW046	CSR072	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.371	pg/L		DNR	11
B6F0192	SW054	CSR073	EPA1613B	Octachlorodibenzo-p-dioxin	1.05	pg/L	J	U	7
B6F0192	SW055	CSR074	EPA1613B	Octachlorodibenzo-p-dioxin	1.62	pg/L	J	U	7
B6F0192	SW042	CSR075	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.054	pg/L	J	U	7
B6F0192	SW042	CSR075	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.024	pg/L	J	U	25
B6F0192	SW042	CSR075	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.057	pg/L	J	U	25
B6F0192	SW042	CSR075	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.206	pg/L		DNR	11
B6F0192	SW043	CSR076	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.316	pg/L		DNR	11
B6F0192	SW043	CSR076	EPA1613B	Octachlorodibenzofuran	2.92	pg/L		J	5BH
B6F5904	SW108	CTR999	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.19	pg/L	U	DNR	11
B6F5904	SW108	CTR999	EPA1613B	Octachlorodibenzo-p-dioxin	1.18	pg/L	J	U	7
B6F5904	SW109	CTS000	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.16	pg/L	U	DNR	11
B6F5904	SW109	CTS000	EPA1613B	Octachlorodibenzo-p-dioxin	1.9	pg/L	J	U	7
B6F5904	SW109	CTS000	EPA1613B	Octachlorodibenzofuran	0.39	pg/L	J	U	25
B6F5904	SW087	CTS001	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.018	pg/L	J	U	25
B6F5904	SW087	CTS001	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.027	pg/L	J	U	25
B6F5904	SW087	CTS001	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.087	pg/L	J	DNR	11
B6F5904	SW087	CTS001	EPA1613B	Heptachlorodibenzofuran (Total)	0.018	pg/L	J	U	25
B6F5904	SW087	CTS001	EPA1613B	Tetrachlorodibenzodioxin (Total)	0.027	pg/L	J	U	25
B6F5904	SW084	CTS002	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.031	pg/L	J	U	25
B6F5904	SW084	CTS002	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.036	pg/L	J	U	25
B6F5904	SW084	CTS002	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.133	pg/L		DNR	11
B6F5904	SW088	CTS003	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.11	pg/L		U	25
B6F5904	SW088	CTS003	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.264	pg/L		DNR	11
B6F5904	SW088	CTS003	EPA1613B	Octachlorodibenzofuran	1.74	pg/L		J	5BH
B6F5904	SW085	CTS004	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.0811	pg/L	J	J	5BH
B6F5904	SW085	CTS004	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.104	pg/L		J	5BH
B6F5904	SW085	CTS004	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.451	pg/L		DNR	11

SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	DV Qualifier	DV Reason
B6F5904	SW085	CTS004	EPA1613B	Octachlorodibenzofuran	2.34	pg/L		J	5BH
B6F5904	SW105	CTS005	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0362	pg/L	J	U	25
B6F5904	SW105	CTS005	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.011	pg/L	J	U	25
B6F5904	SW105	CTS005	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.022	pg/L	J	U	25
B6F5904	SW105	CTS005	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.0591	pg/L	J	U	25
B6F5904	SW105	CTS005	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.179	pg/L		DNR	11
B6F5904	SW106	CTS006	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.079	pg/L	J	J	5BH
B6F5904	SW106	CTS006	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.115	pg/L		J	5BH
B6F5904	SW106	CTS006	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.559	pg/L		DNR	11
B6F5904	SW106	CTS006	EPA1613B	Octachlorodibenzofuran	2.94	pg/L		J	5BH
B6F5904	SW096	CTS007	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.043	pg/L	J	U	7
B6F5904	SW096	CTS007	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.061	pg/L	J	U	25
B6F5904	SW096	CTS007	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.193	pg/L		DNR	11
B6F5904	SW097	CTS008	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.053	pg/L	J	U	25
B6F5904	SW097	CTS008	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.087	pg/L	J	J	5BH
B6F5904	SW097	CTS008	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.387	pg/L		DNR	11
B6F5904	SW097	CTS008	EPA1613B	Octachlorodibenzofuran	2.41	pg/L		J	5BH
B6F5904	SW099	CTS009	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0505	pg/L	J	U	7
B6F5904	SW099	CTS009	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.055	pg/L	J	U	25
B6F5904	SW099	CTS009	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.207	pg/L		DNR	11
B6F5904	SW100	CTS010	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.068	pg/L	J	J	5BH
B6F5904	SW100	CTS010	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.091	pg/L	J	J	5BH
B6F5904	SW100	CTS010	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.336	pg/L		DNR	11
B6F5904	SW100	CTS010	EPA1613B	Octachlorodibenzofuran	3.56	pg/L		J	5BH
B6F5904	SW102	CTS011	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0523	pg/L	J	U	7
B6F5904	SW102	CTS011	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.062	pg/L	J	U	25
B6F5904	SW102	CTS011	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.232	pg/L		DNR	11
B6F5904	SW103	CTS012	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.0701	pg/L	J	J	5BH
B6F5904	SW103	CTS012	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.0808	pg/L	J	J	5BH
B6F5904	SW103	CTS012	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.347	pg/L		DNR	11
B6F5904	SW103	CTS012	EPA1613B	Octachlorodibenzofuran	3.86	pg/L		J	5BH

CDC	Committee ID	Lab Commis ID		Anglata	Desult	Unite	Lab Elan	DV	DV
SDG	Sample ID	Lab Sample ID	Method	Analyte	Result	Units	Lab Flag	Qualifier	Reason
B6F5904	SW090	CTS013	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.035	pg/L	J	U	7
B6F5904	SW090	CTS013	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.077	pg/L	J	U	25
B6F5904	SW090	CTS013	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.249	pg/L		DNR	11
B6F5904	SW091	CTS014	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.087	pg/L	J	J	5BH
B6F5904	SW091	CTS014	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.172	pg/L		J	5BH
B6F5904	SW091	CTS014	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	1.52	pg/L		DNR	11
B6F5904	SW091	CTS014	EPA1613B	Octachlorodibenzofuran	3.08	pg/L		J	5BH
B6F5904	SW093	CTS015	EPA1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran	0.0204	pg/L	J	U	7
B6F5904	SW093	CTS015	EPA1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin	0.0478	pg/L	J	U	25
B6F5904	SW093	CTS015	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	0.187	pg/L		DNR	11
B6F5904	SW094	CTS016	EPA1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	0.0927	pg/L	J	J	5BH
B6F5904	SW094	CTS016	EPA1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	0.17	pg/L		U	25
B6F5904	SW094	CTS016	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	1.53	pg/L		DNR	11
B6F5904	SW094	CTS016	EPA1613B	Octachlorodibenzofuran	3.31	pg/L		J	5BH
B6F5904	SW028	CTS139	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	2.2	pg/L	U	DNR	11
B6F5904	SW028	CTS139	EPA1613B	Octachlorodibenzo-p-dioxin	9.1	pg/L	J	U	7
B6F5904	SW029	CTS140	EPA1613B	2,3,7,8-Tetrachlorodibenzofuran	2.4	pg/L	J	DNR	11
B6F5904	SW029	CTS140	EPA1613B	Octachlorodibenzofuran	3.5	pg/L	J	U	7

# APPENDIX C PRC FIBERS FOR THE 2016 ASSESSMENT OF POREWATER WITHIN THE TCRA ARMORED CAP

## **1 INTRODUCTION**

Two errors occurred with the management of the solid phase-microextraction (SPME) fibers spiked with performance reference compounds (PRCs) for the 2016 TCRA armored cap porewater study. The PRC-spiked fibers are used to evaluate whether the SPME samplers have reached equilibrium with the porewater environment by comparing the mass of each PRC at the time of deployment to the mass of each PRC at retrieval. For the TCRA cap porewater study, the SPME samplers were deployed in May 2016, and six fibers spiked with PRCs were deployed at the same time. Figure 1 shows the locations of the sampling and PRC fibers.

## 2 SUMMARY

Of the six PRC fibers deployed with the porewater samplers:

- One PRC fiber (SJCPR1) was retrieved mid-deployment (June 2016).
- One PRC fiber (SJCR001) could not be located at the end of the deployment period (July 2016)
- The remaining four PRC fibers were retrieved as planned.

Because of an error in communication with the laboratory, the PRC fiber retrieved middeployment (SJCPR1) and two of the PRC fibers retrieved at the end of deployment (approximately July 19–22) were inadvertently spiked with PRC compounds following retrieval. As a result, the information generated by these PRC fibers is limited.

In addition, a portion of the extract from the upper interval of the fiber retrieved middeployment was spilled at the laboratory. The extract was recovered, but use of the results may also be limited.

Two of the PRC fibers were deployed, retrieved and analyzed consistent with the sampling and analysis plan (SAP; Integral and Anchor QEA 2016), and results can be used as planned.

## **3 PRC FIBER RETRIEVED MID-DEPLOYMENT**

The PRC fiber at station SJCPR1 (Figure 1) was retrieved mid-deployment to evaluate the equilibrium status of the samplers. Results were not used (and this fiber was not planned for

use) to perform calculations related to conclusions of the 2016 study. Per the SAP, the fiber was sectioned into three depth intervals and extracted. Because of an error in communication with the analytical laboratory, extracts for all three intervals were spiked with two of the three PRCs (13C-2,3,7,8-TCDD and 13C-2,3,7,8-TCDF), precluding the evaluation of equilibrium for these compounds in these samples. For a typical study involving measurement of dioxins and furans in environmental media, the carbon-13 labeled dioxins are employed by the analytical method to monitor instrument conditions and measure the efficiency of the extraction. It is standard practice for all samples to be spiked with the carbon-13 labeled compounds when analyzing for dioxins and furans. For the porewater study, these PRC fibers were not supposed to be spiked with the carbon-13 labeled compounds. This unusual requirement was not correctly communicated to the laboratory, leading to this error.

The third PRC compound (13C-2,3,4,7,8-PeCDF) was not spiked and the concentrations and equilibrium status for this compound can still be evaluated. The resulting data can be used to check the equilibrium status of the fibers; but for this location, evaluation of equilibrium status will rely on results for 13C-2,3,4,7,8-PeCDF only. This limitation will introduce some uncertainty if results are used to estimate the equilibrium status of other congeners in the related sample, underestimating the equilibrium status of other, less-chlorinated congeners.

In addition, a portion of the extract for the upper interval of the SJCPR1 fiber was spilled by the laboratory. The recoveries of all compounds spiked prior to extraction (13C-2,3,7,8-TCDD; 13C-2,3,7,8-TCDF; and 13C-1,2,3,7,8-PeCDF) were approximately half of the recoveries for these compounds from the extracts for the other two intervals. The recoveries of the clean-up standard (13C-1,2,3,7,8,9-HxCDF), which is spiked following extraction, but prior to any clean-up procedures, are comparable in all three extracts. The low recoveries of the compounds spiked prior to extraction introduces additional uncertainty in evaluating the equilibrium of the upper interval at station SJCPR1.

## 4 PRC FIBERS RETRIEVED AT END OF DEPLOYMENT

The four PRC fibers retrieved at the end of deployment (SJCR002, SJCR003, SJCR004, and SJCRP2; Figure 1) were also each sectioned into three depth intervals and extracted. For the fibers from two of these stations (SJCR004 and SJCPR2), all three intervals were spiked with

all three PRC compounds, precluding the evaluation of equilibrium at these stations. This error occurred for the same reason as for the fiber retrieved mid-deployment: it is standard laboratory procedure to spike the carbon-13 labeled compounds and the spiking requirements were not properly communicated to the laboratory.

The PRC fibers from stations SJCR002 and SJCR003 are unaffected and can be used to provide an assessment of the equilibrium status of sample fibers at the time of retrieval.

## 5 CONCLUSION

The data quality objectives of the TCRA cap porewater study are threefold:

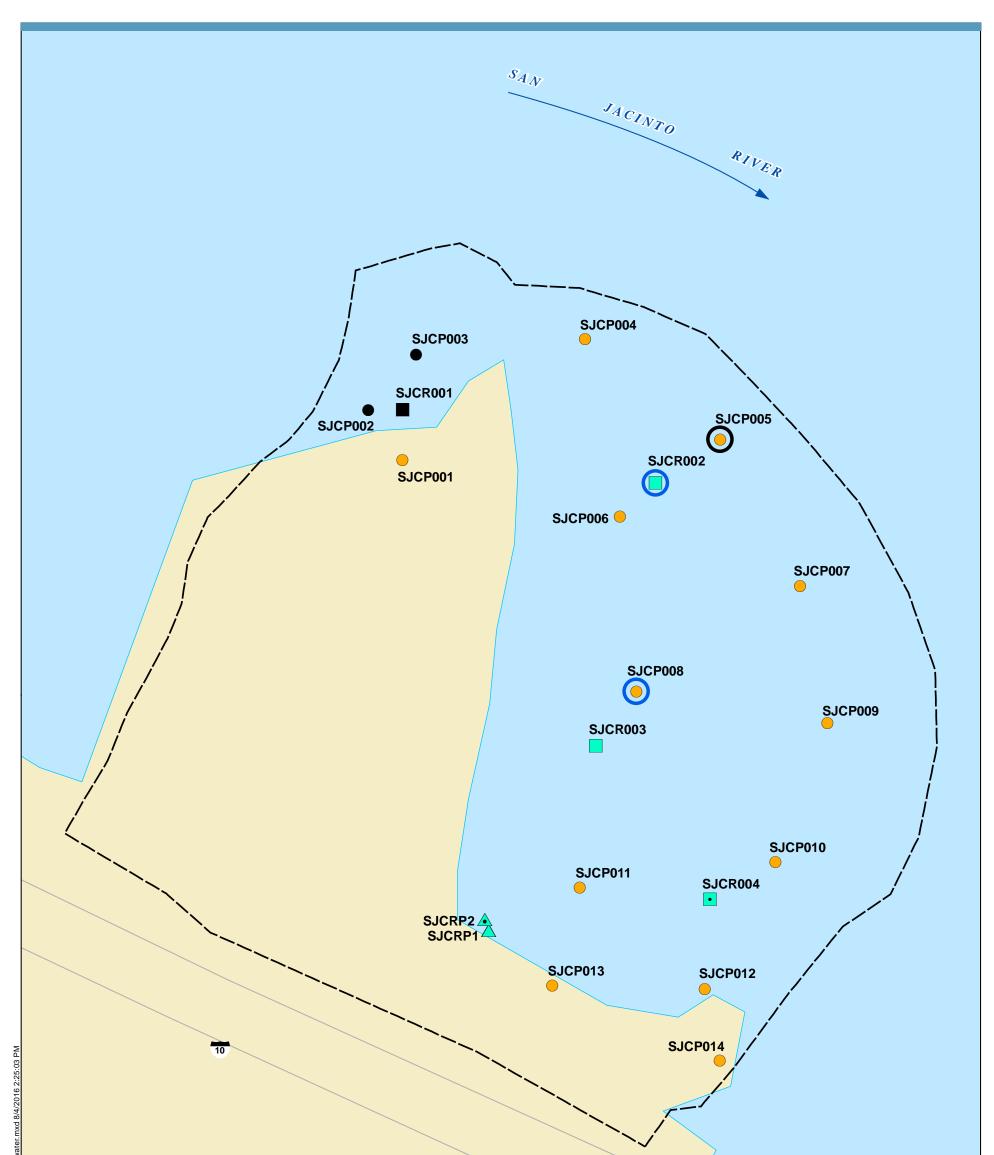
- To determine whether dissolved dioxins and furans are present in porewater of the TCRA armored cap
- To determine whether vertical gradients in concentrations of dioxins and furans in the porewater of the cap are present
- To determine whether porewater concentrations in the cap differ from concentrations in surface water above the cap.

The errors described above will not compromise achievement of these DQOs. The study objectives will be met using the two remaining PRC fibers. Samplers were placed in areas of the cap with design thicknesses of 12 and 18 in. The 12-in. thickness is represented by SJCR003 and the 18-in. cap thickness by SJCR002. Both fibers were separated into three segments allowing assessment of the equilibrium status at all three depth intervals. SJCR002 includes an ambient water segment to be used to assess equilibrium status of the fiber that was in the water above the cap.

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and Anchor QEA, LLC, Ocean Springs, MS. February 2016.



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Porewater Sampling Station

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Porewater Sampling Station (Lost Sampler)

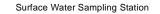
Performance Reference Compound-loaded Fiber Location

Performance Reference Compound-loaded Fiber Location (Samples Affected by Error)

Performance Reference Compound-loaded Fiber Location (Lost Sampler)

Extra Performance Reference Compound-loaded Fiber Location

Extra Performance Reference Compound-loaded Fiber Location (Samples Affected by Error)



Surface Water Sampling Station (Lost Sampler)

Original (1966) Perimeter of the Northern Impoundments

Figure 1 Porewater Sampling Locations SJRWP Superfund/MIMC and IPC