2.0 Data Collection and Analytical Methods

This section gives a brief description of the sediment and tissue sample collection and analytical methods for each data type. A full summary can be found in the final report (SAIC 2008) or the SAP (SAIC 2007). A list of the archived samples analyzed for this round of analysis is provided in Tables 1, 2, and 3. In addition, details of the tissue processing are summarized, including: species, number of organisms per replicate, and the final wet weight of each replicate.

2.1 Sampling Platforms

Several sampling vessels were used to meet the multiple data collection objectives. The research vessel (R/V) *Nancy Anne*, owned and operated by Mr. Bill Jaworski of Marine Sampling Systems, was used for the surface sediment collection in DUAs 2, 3, and 4. The R/V *Kittiwake*, owned and operated by Mr. Charlie Eaton of Bio-Marine Enterprises was used for bottom trawling to collect fish tissue in DUAs 2 and 3. The R/V *Growler* and R/V *Schooner*, owned and operated by Science Applications International Corporation (SAIC), were used to sample sediments in DUA 1, as well as crab and clam tissue.

2.2 Surface Sediment Samples

Surface sediment samples (0 to 10 cm) for chemical analysis were collected using a stainless-steel 0.025 m^2 Ekman grab sampling device deployed from a small boat in DUA 1 and a stainless-steel 0.25 m^2 hydraulic grab sampler deployed from the R/V *Nancy Anne* in DUAs 2, 3, and 4. Grab samples were collected and homogenized for each sampling location to provide sufficient volume for chemical analysis and archiving.

Surface sediment samples for archiving and chemical analysis were collected at 128 locations throughout Fidalgo Bay. Samples from 58 of these locations were analyzed in the initial round of analysis. One 8-ounce jar from each of these locations was collected for potential analysis of dioxin/furan congeners. Twenty four of these samples were analyzed for dioxins/furans in the first round of analysis by Axys Analytical Services Ltd. of Sidney, BC. The remaining samples were archived by Axys at -18°C.

At 70 sampling locations, two 16-ounce jars were collected and sent to Columbia Analytical Services (CAS) of Kelso, WA, for archiving. One jar from each location was frozen at -18°C for potential analysis of semi-volatile organic compounds (SVOC) and metals, while the other was archived at 4°C for potential grain size analysis. Holding times have been exceeded for the analysis of grain size, and the sediment samples have been discarded.

For the current round of analyses, 21 surface sediment samples were analyzed for SMS chemistry from three locations in DUA 1, three locations in DUA 2, seven locations in DUA 3, and eight locations in DUA 4. Eleven sediment samples were also analyzed for dioxin/furan congeners, including five locations from DUA 3, and six locations from DUA 4. Table 1 lists the archived sediment samples that have been analyzed in this round of analysis and the reason for analysis. Figure 2 displays the locations of the SMS chemistry and dioxin/furan archive analysis samples.

Table 1. Archived Sediment Samples Submitted for Chemical Analysis

Station Number	Collected	SMS Chemistry	Dioxin/ Furan	Reason for Analysis
A1-09	9/20/2007	X		SQS Toxicity in A1-04 and A1-01
A1-14	9/20/2007	X		CSL toxicity in A1-16
A1-18	9/20/2007	X		CSL toxicity in A1-16
A2-30	8/30/2007	X		Mercury exceeded SQS in A2-23
A2-40	8/30/2007	X		SQS toxicity in A2-38
A2-56	8/30/2007	X		SQS toxicity in A2-38
A3-03	9/4/2007	X	X	CSL toxicity in A3-02 and A3-13; detected dioxin/furan concentrations in A3-02 and A3-22
A3-06	9/4/2007	X	X	CSL toxicity in A3-02 and A3-13; detected dioxin/furan concentrations in A3-02 and A3-22
A3-18	9/5/2007	X	X	CSL toxicity in A3-02 and A3-13; detected dioxin/furan concentrations in A3-02 and A3-22
A3-22b	9/5/2007	X		Mercury exceeded SQS in A2-23
A3-36	9/4/2007	X		SQS toxicity in A2-38
A3-37	9/4/2007	X	X	CSL toxicity and detected dioxin/furan concentrations in A3-32
A3-42	9/4/2007	X	X	CSL toxicity and detected dioxin/furan concentrations in A3-32
A4-03	9/6/2007	X	X	Detected dioxin/furan concentrations in A4-05
A4-04	9/6/2007		X	Detected dioxin/furan concentrations in A4-05
A4-06	9/6/2007	X	X	Detected dioxin/furan concentrations in A4-05
A4-07	9/6/2007		X	Detected dioxin/furan concentrations in A4-05 and A4-09
A4-08	9/6/2007	X	X	CSL chemistry and toxicity in A4-09, A4-10, and A4-12; dioxin/furan congeners in A4-12
A4-11	9/6/2007	X	X	CSL chemistry and toxicity in A4-09, A4-10, and A4-12; dioxin/furan congeners in A4-12
A4-14	9/6/2007	X		SQS chemistry and toxicity in A4-13
A4-28	9/5/2007	X		CSL chemistry at A4-29
A4-29	9/5/2007	X		CSL exceeded for bis(2-ethylhexyl)phthalate, confirmatory analysis
A4-30	9/5/2007	X		CSL chemistry at A4-29

2.3 Fish, Clam, and Crab Tissue

Tissue samples were collected and archived from each of the DUAs (Figure 2). English sole (*Parophrys vetulus*) and starry flounder (*Platichthys stellatus*) were collected using bottom trawls in DUAs 2 and 3 of Fidalgo Bay. Crab pots were deployed for the collection of Dungeness crabs (*Cancer magister*) and red rock crabs (*Cancer productus*) in DUAs 2, 3, and 4. Small shovels and trowels were used to collect Manila (*Ruditapes philippinarum*), bent nose (*Macoma nasuta*), horse (*Tresus* sp.), and *Macoma* sp. clams at low tide from accessible beaches in each of the DUAs. Polychaetes were opportunistically collected from the clamming locations when found in relative abundance. All tissue samples were archived frozen (-25°C) at the SAIC warehouse (Bothell, WA). Tables 2 and 3 present a summary of the sample size and final weight of tissue sent for chemical analysis. Appendix A includes documentation from the tissue processing logbooks.

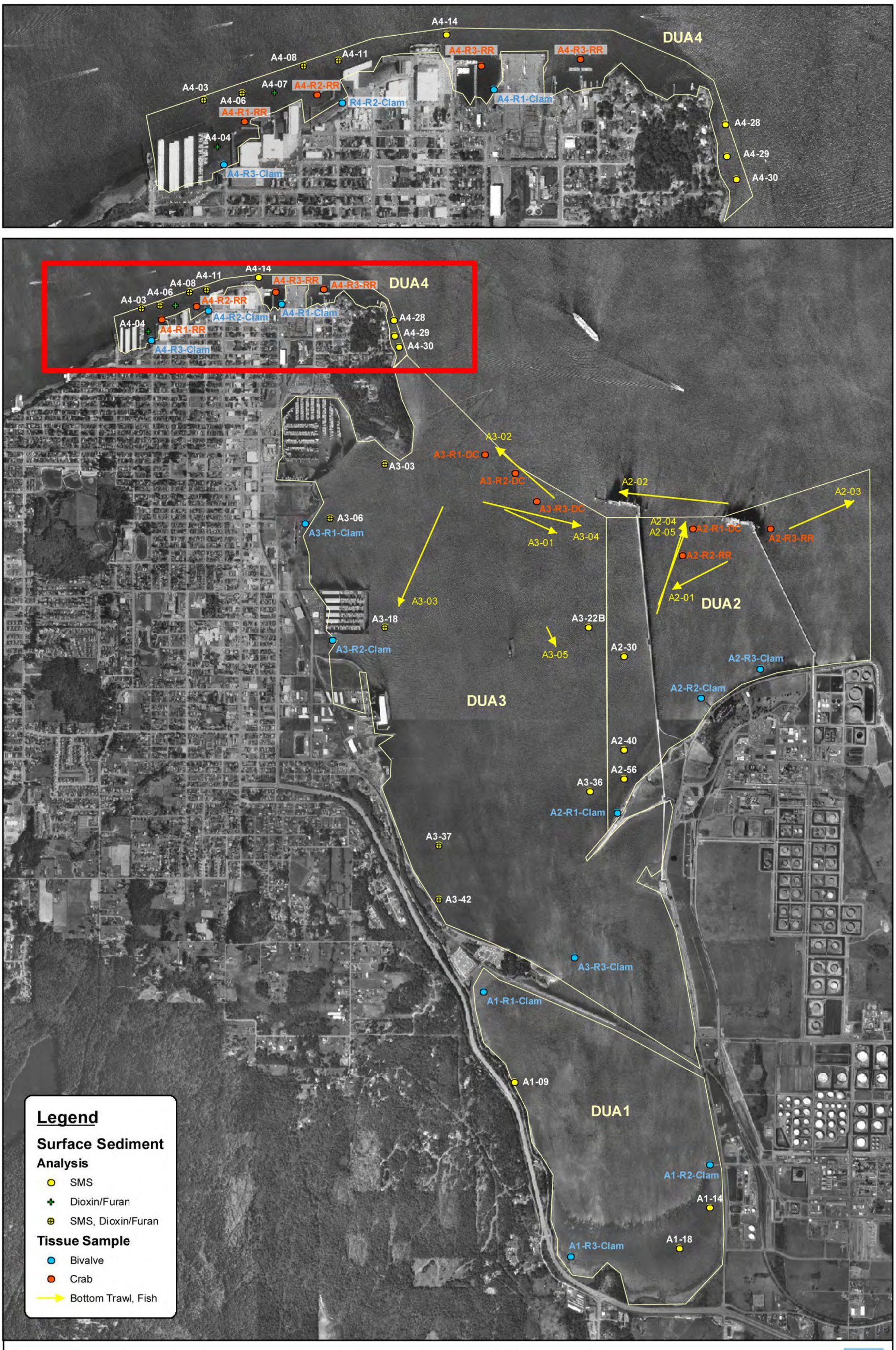

Figure 2. Bottom Trawling, Tissue Sampling, and Archived Sediment Locations

The chemical analysis of the tissue samples included PCB Aroclors and metals by CAS, and dioxin/furan congeners by Axys. To provide adequate sample volumes for both laboratories, all tissue samples were processed and composited at SAIC and separated into two jars. The analytical laboratories were responsible for sample homogenization prior to analysis.

Clams were collected from three different intertidal areas in each DUA. For each tissue sample, individual clams were sorted by length, so that both laboratories received an equal number of similar sized clams. Clams were then shucked and individual weights were recorded in the log book. Table 2 lists the sample size and total weight of each sample, along with the collection and processing dates. Due to insufficient volume of tissue, the A3R2-macoma sample was submitted only to Axys for dioxin/furan congener analysis.

Polychaetes were only analyzed for dioxin/furan congeners. Subsamples of polychaete tissue were taken from the archive jars and sent to Axys (Table 2). Remaining tissue was kept archived at SAIC. A total of six polychaete tissue samples were submitted for dioxin/furan congener analysis.

Table 2. Archived Clam, Polychaete, and Fish Tissue Samples Submitted for Chemical Analysis

Sample ID	Collected	Processed	Axys		CAS	
			N	Total Weight (grams)	N	Total Weight (grams)
A1R1-manilla	10/8/2007	6/17/2008	10	123	10	116
A1R2-manilla	10/8/2007	6/17/2008	10	131	10	130
A1R3-manilla	10/8/2007	6/17/2008	12	158	12	146
A2R1-manila	8/28/2007	6/17/2008	10	75	10	73
A2R2-bentnose	8/28/2007	6/17/2008	15	53	15	49
A2R3-bentnose	9/6/2007	6/17/2008	10	52	10	59
A3R1-horse	8/28/2007	6/17/2008	3	268	3	247
A3R2-macoma	8/28/2007	6/17/2008	7	9	-	-
A3R3-manila	8/28/2007	6/17/2008	6	108	6	128
A4R1-bentnose	9/6/2007	6/17/2008	10	37	10	42
A4R2-bentnose	9/7/2007	6/17/2008	9	63	9	64
A4R3-bentnose	9/7/2007	6/17/2008	10	58	10	57
A2R2-polychaete	8/28/2007	6/18/2008	-	20	-	-
A2R3-polychaete	9/6/2007	6/18/2008	-	47	-	-
A3R1-polychaete	8/28/2007	6/18/2008	-	100	-	-
A3R3-polychaete	8/28/2007	6/18/2008	-	26	-	-
A4R1-polychaete	9/6/2007	6/18/2008	-	102	-	-
A4R2-polychaete	9/7/2007	6/18/2008	-	39	-	-
A2R1-SF	8/23/2007	6/18/2008	5	498	5	496
A2R2-SF	8/23/2007	6/18/2008	5	244	5	245
A2R3-SF	8/23/2007	6/18/2008	5	246	5	246
A3R1-ES	8/23/2007	6/18/2008	5	93	5	96
A3R2-SF	8/23/2007	6/18/2008	5	243	5	243
A3R3-SF	8/23/2007	6/18/2008	5	242	5	241

N = number of individuals

Three replicates of flatfish were collected and processed from DUAs 2 and 3. Starry flounder was the predominant species, comprising five of the six replicates. The remaining replicate was composed of English sole. All fish tissue replicates consisted of five individuals of similar size. Each fish tissue sample consisted of skin-off filets. Equal volumes of tissue were taken from each filet in the replicate and then composited. Table 2 lists the sample volumes submitted to CAS and Axys for chemical analysis.

Dungeness and red rock crab were collected from DUAs 2, 3, and 4. Each sample consisted of a single crab species composed of three to five individuals. All crabs within a given sample were dissected with the tissue separated into three types: muscle, hepatopancreas, and other soft tissue. Equal volumes of tissue from individual crabs were used to produce a sample composite for each tissue type. Aliquots of sample from the composite for each tissue type was submitted to CAS and Axys for chemical analysis, with the exception of the hepatopancreas from A2R1-RR, which was only submitted to Axys due to limited sample volume. Table 3 lists the number of crabs in each sample and the total weight of tissue for each type collected.

Table 3. Archived Crab Tissue Samples Submitted for Chemical Analysis

Sample ID	Collected	Processed	N	Total Weight (grams)
A2R1-RR-hepato*	8/23 - 8/28/2007	6/20/2008	3	24
A2R1-RR-other	8/23 - 8/28/2007	6/20/2008	3	71
A2R1-RR-muscle	8/23 - 8/28/2007	6/20/2008	3	234
A2R2-DC-hepato	8/23 - 8/28/2007	6/20/2008	3	85
A2R2-DC-other	8/23 - 8/28/2007	6/20/2008	3	178
A2R2-DC-muscle	8/23 - 8/28/2007	6/20/2008	3	515
A2R3-DC-hepato	8/23 - 8/28/2007	6/20/2008	4	84
A2R3-DC-other	8/23 - 8/28/2007	6/20/2008	4	221
A2R3-DC-muscle	8/23 - 8/28/2007	6/20/2008	4	570
A3R1-DC-hepato	8/23 - 8/28/2007	6/18/2008	5	114
A3R1-DC-other	8/23 - 8/28/2007	6/18/2008	5	302
A3R1-DC-muscle	8/23 - 8/28/2007	6/18/2008	5	760
A3R2-DC-hepato	8/23 - 8/28/2007	6/18/2008	5	105
A3R2-DC-other	8/23 - 8/28/2007	6/18/2008	5	289
A3R2-DC-muscle	8/23 - 8/28/2007	6/18/2008	5	584
A4R1-RR-hepato	8/23 - 8/28/2007	6/19/2008	5	56
A4R1-RR-other	8/23 - 8/28/2007	6/19/2008	5	95
A4R1-RR-muscle	8/23 - 8/28/2007	6/19/2008	5	215
A4R2-RR-hepato	8/23 - 8/28/2007	6/19/2008	5	38
A4R2-RR-other	8/23 - 8/28/2007	6/19/2008	5	101
A4R2-RR-muscle	8/23 - 8/28/2007	6/19/2008	5	295

* Due to low sample volume, A2R2-RR-hepato was not analyzed by CAS for metals and PCBs.

2.4 Chemical Analysis

The chemical analysis of the archived samples collected in Fidalgo Bay includes the SMS analyte list and dioxin/furan congeners for sediment, and PCB Aroclors, metals, and dioxin/furan congeners for tissue. All of the laboratory analytical methods used in this program were in accordance with the Puget Sound Estuary Program (PSEP) guidelines and any modifications proposed during the Sediment Management Annual Review Meeting (SMARM) (PSEP 1997a,b).

Analysis of SMS chemistry was conducted by CAS. The analyses for SVOCs and PCBs were carried out by U.S. Environmental Protection Agency (USEPA) Methods 8270 and 8082, respectively. The analysis for metals was carried out using USEPA Method 6020, with the exception of mercury, which was analyzed using USEPA Method 7471C (USEPA 2008). The data results are provided in Appendix B, and quality assurance/ quality control (QA/QC) data validation are provided in Appendix C.

Analysis of dioxin/furan congeners was conducted by Axys using USEPA Method 1613B (tetra- through octa-chlorinated dioxins and furans by isotope dilution high resolution gas chromatography/mass spectrophotometry). The concentration of dioxin/furan compounds was normalized to the toxicity of 2,3,7,8-TCDD using toxic equivalent factors (TEFs) updated by the World Health Organization (WHO) in 2005 (Van den Berg et al. 2006).