

Exponent<sup>®</sup>

**Chukchi Sea  
Environmental Studies  
Baseline Program  
2009 Fish Sampling –  
Chemistry Results**





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2009 Fish Sampling –  
Chemistry Results**

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## Acronyms and Abbreviations

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COC	chain-of-custody
COP	ConocoPhillips
CRM	certified reference material
CSF	cancer slope factor
CVAAS	or cold-vapor atomic absorption spectrometry
DQO	data quality objective
DUP	laboratory duplicate
FAAS	flame atomic absorption spectrometry
FIT	Florida Institute of Technology
ft <sup>3</sup>	cubic feet
g	grams
GC	gas chromatograph
GC/MS	gas chromatography/mass spectrometry
GPS	global positioning system
H <sub>2</sub> O <sub>2</sub>	hydrogen peroxide
H <sub>2</sub> SO <sub>4</sub>	sulfuric acid
HNO <sub>3</sub>	nitric acid
HPAH	high molecular weight PAH
ICP-MS	inductively coupled - plasma mass spectrometry
IHA	Incidental Harassment Authorization
LCS	laboratory control sample
LOA	letters of authorization
LPAH	low molecular weight PAH
M	meters
MDL	method detection limits
mL	milliliters
MMS	Minerals Management Service
MS	matrix spike
MSD	mass spectral detector
NEPA	National Environmental Policy Act
NIST	U.S. National Institute of Standards and Technology
nm	nautical mile
NPDES	National Pollution Discharge Elimination System
NRC	National Research Council of Canada
NSC	North Slope Crude
PAH	polycyclic aromatic hydrocarbons
PB	procedure blank
PFTBA	perfluorotributylamine
POC	particulate organic carbon
QA	quality assurance
QA/QC	quality assurance/quality control
QAM	quality assurance manual
QAPP	quality assurance project plans

QAU	quality assurance unit
QC	quality control
QMP	quality management plan
RF	response factors
RL	reporting limit
RSD	relative standard deviation
Shell E&P	Shell Exploration and Production
SIM	selected ion monitoring
SIS	surrogate internal standard
SOP	standard operating procedures
SRM	standard reference material
TEO	total extractable organics
TPAH	total PAH
USEPA	United States Environmental Protection Agency
WBM	water based drilling muds



## Executive Summary

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The Chukchi Sea Planning Area is located off the northwest coast of Alaska. The planning area extends from near Point Barrow (156°W longitude) in the east to the boundary with Russian waters at 169°W longitude and from near Point Hope (68°20'N latitude) in the south, northward to 75°N latitude and lies entirely above the Arctic Circle (66°33' 39"N) in the Arctic geographic zone. The Chukchi Sea Planning Area is predominantly a shallow embayment of the Arctic with water depths within the Lease Sale Area ranging from 30 meters (m) to approximately 3000 m. The Burger and Klondike prospect areas are located about 120 km northwest of Wainright, Alaska, in 40 to 50 m of water (Figure 1-1). ConocoPhillips (COP) and Shell Exploration and Production (Shell E&P) submitted bids for lease blocks in the Burger and Klondike Prospect areas in the February 6, 2008 Chukchi Sea Lease Sale 193. COP is managing a scientific field survey program in these two prospect areas on behalf of both COP and Shell E&P. The field program is generating baseline (pre-drilling) information on the physical, chemical, biological, and oceanographic environment, and distribution and concentrations of chemicals associated with offshore oil and gas operations (metals and hydrocarbons) in the Klondike and Burger prospect areas.

The objective of the COP Chukchi Sea Environmental Studies Program is to develop baseline information about the marine environment in the Burger and Klondike Prospect areas for submission to the Minerals Management Service (MMS). The objective of the Chemical Characterization component of the COP Chukchi Sea Environmental Studies Program is to determine baseline (pre-exploration and development) concentrations of metals and hydrocarbons in sediments and tissues of zooplankton, benthic invertebrates, and fish in the study area and how distribution and concentrations of these chemicals are being influenced by inter-annual environmental changes. The assessment of potential impact to subsistence or commercial fishing was not a focus of this investigation since no subsistence or commercial fishing is performed in the Burger and Klondike prospect areas. This report presents the results of analyses of metals and hydrocarbons in fish samples collected during the September/October 2009 field survey event.

The fish tissue samples collected have very low concentrations of Total PAHs and metals that are within the range of values reported from previous studies of other Alaskan coastal and continental shelf areas and are representative of background tissue concentrations. The Total PAH values are also low as compared to the United States Environmental Protection Agency (USEPA) (2000) fish advisory consumption limits for PAH and pose no human health risk if consumed. Since PAH, and other hydrocarbons, do not biomagnify in the marine food chains (Neff, 2002), trophic transfer is inefficient and the measured hydrocarbon concentrations are unlikely to reach high concentrations in upper trophic level animals, such as birds, whales, seals, and polar bears. Fish tissues samples collected from the Chukchi Sea for this study have very low concentrations of Ag, Cd, Hg and Pb (acronyms for metals are provided in Table 2-3). Copper, Se and Zn are regulated by fish and the values for fish from this study are within 10% of concentrations reported for Arctic cod from the Beaufort Sea and most likely represent background conditions. Concentrations of Ba and Cr were low, but enhanced by the presence of sediment in the composite fish samples. Even though the samples for this study were made up of mixed fish species, the data show that the fish tissue is essentially free of metal contamination and comparable with clean fish samples from the Beaufort Sea.

# 1 Introduction

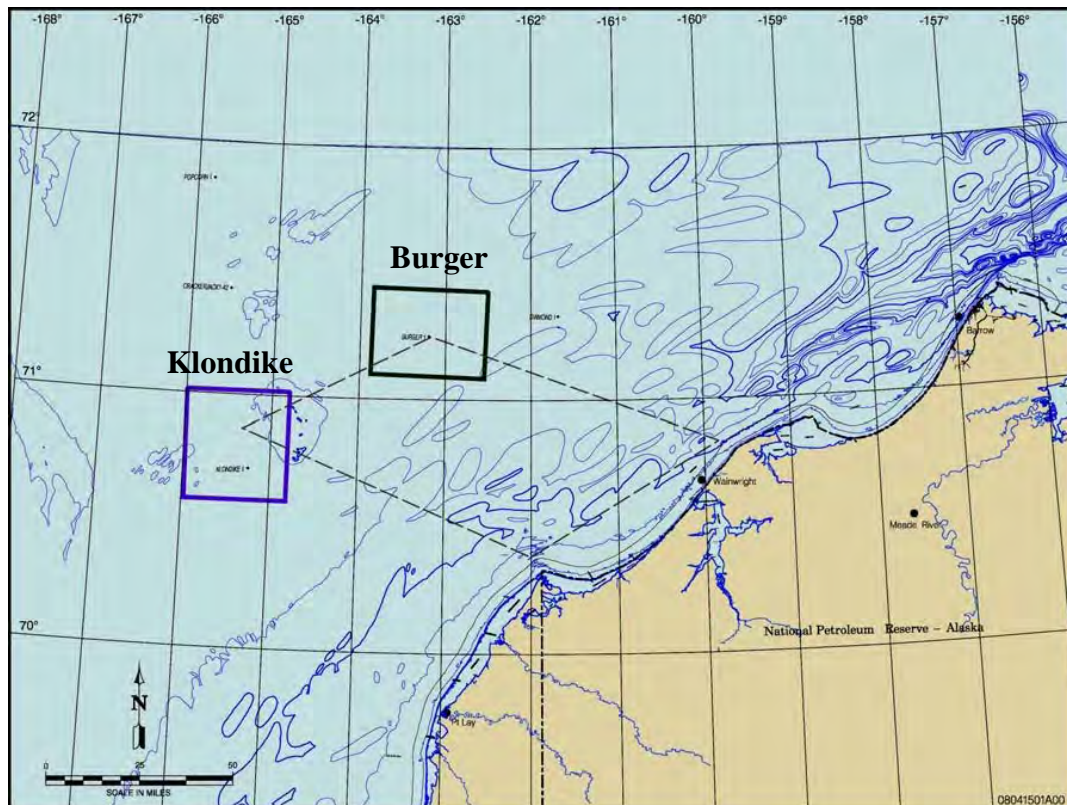
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## 1.1 Study Area

The Chukchi Sea Planning Area is located off the northwest coast of Alaska. The planning area extends from near Point Barrow (156°W longitude) in the east to the boundary with Russian waters at 169°W longitude and from near Point Hope (68°20'N latitude) in the south, northward to 75°N latitude and lies entirely above the Arctic Circle (66°33' 39"N) in the Arctic geographic zone.

The Chukchi Sea Planning Area is a shallow embayment of the Arctic with water depths within the Lease Sale Area ranging from 30 meters (m) to approximately 3000 m. About 80% of the planning area lies between the 30 and 90 m isobaths. The Burger and Klondike prospect areas are located about 120 km northwest of Wainwright, Alaska, in 40 to 50 m of water (Figure 1-1).

**Figure 1-1. Chukchi Sea Study Planning Area**



The Chukchi Sea outer continental shelf where the Klondike and Burger prospects are located is a highly productive marine ecosystem (Dunton et al., 1989; Feder et al., 1989; Grebmeier and Dunton, 2000; Moran et al., 2005). Much of the particulate organic carbon (POC), produced primarily by phytoplankton photosynthesis or in runoff from land, is exported to the sediments or off the shelf. The POC also supports blooms of zooplankton communities, dominated by calanoid copepods and euphausiids, which are consumed by bowhead whales. POC in sediments support development of a rich benthic fauna that supports benthic feeders, such as walrus and many species of demersal fish, and results in strong benthic/pelagic coupling of nutrients. Sediments in the northeast Chukchi Sea contain high concentrations of organic matter from marine and terrigenous sources (Yunker et al., 2005). This organic matter tends to sequester metals and hydrocarbons and is ingested by benthic fauna. However, the highly humic fraction of POC derived from diagenesis of terrestrial plant carbon, such as peat, kerogens, immature bitumen, and lignite (Steinhauer and Boehm, 1992; Yunker et al., 2005), tightly binds metals and hydrocarbons, lowering their bioavailability to benthic fauna, even those that ingest peat carbon, limiting bioaccumulation and trophic transfer in the Chukchi Sea food web.

## **1.2 Project Background**

Four lease sales were held for different parts of the Chukchi Sea shelf in 1988 and 1991 (Sales 97, 109, 124, and 126). Five exploratory wells were drilled in blocks leased in sales 97 and 109 (Table 1-1). Shell Western E&P, Inc. drilled exploratory wells in the Klondike and Burger Prospects during the summer and fall of 1989. The other three wells were drilled between October 1989 and September 1991. The Burger well discovered gas and condensate resources estimate at 7.6 to 27.5 trillion cubic feet (ft<sup>3</sup>) and 393 to 1,404 million barrels, respectively (Craig and Sherwood, 2004). The Klondike well discovered small oil plays at several depths. Two of the other three wells also discovered small plays of oil. The discoveries were not considered economic at the time and the wells were plugged and abandoned.

**Table 1-1. Exploratory wells drilled in the Chukchi Sea Planning Area, ordered by spud date**

Prospect Area	Latitude	Longitude	Spud Date	Water Depth (m)
Klondike	70° 42' 39.171"N	165° 14' 59.107"W	7/15/89	43
Burger	71° 15' 0.4995"N	163° 11' 40.499"W	9/22/89	45
Popcorn	71° 51' 16.385"N	165° 48' 24.893"W	10/14/89	44
Crackerjack	71° 25' 7.665"N	165° 32' 29.253"W	9/23/90	42
Diamond	71° 19' 48.34"N	161° 40' 48.01"W	9/7/91	46

Source: [http://www.mms.gov/alaska/fo/wellhistory/CK\\_WELLS.HTM](http://www.mms.gov/alaska/fo/wellhistory/CK_WELLS.HTM)

ConocoPhillips (COP) and Shell Exploration and Production (Shell E&P) submitted bids for lease blocks in the Burger and Klondike Prospect areas in the February 6, 2008 Chukchi Sea Lease Sale 193. COP is managing a scientific field survey program in these two prospect areas on behalf of both COP and Shell E&P. The field program is generating baseline (pre-drilling) information on the physical, chemical, biological, and oceanographic environment, and distribution and concentrations of chemicals associated with offshore oil and gas operations (metals and hydrocarbons) in the Klondike and Burger prospect areas.

Chemicals that might be introduced into the Chukchi Sea by exploratory drilling activities will tend to accumulate in sediments, from which they may pass through the local food web to valued ecosystem components, such as marine fish, mammals and birds. The major permitted discharges associated with offshore exploratory drilling are drilling muds and drill cuttings (Neff, 2005). The only permitted discharges anticipated for development of Chukchi Sea fossil fuel resources are water based drilling muds (WBM) and associated drill cuttings generated during drilling of exploratory wells. The contaminants of greatest concern associated with discharge of WBM and cuttings are metals (particularly barium, cadmium, copper, chromium, lead, mercury, and zinc) and petroleum hydrocarbons. Small amounts of petroleum hydrocarbons may be discharged in WBM and cuttings. The hydrocarbons of major environmental concern are polycyclic aromatic hydrocarbons (PAH). PAH may get into WBM and cuttings from organic rich shales and kerogens in geologic formations penetrated by the drill bit. Petroleum also may enter the Chukchi Sea in accidental discharges or spills during exploration, development, and production.

### **1.3 Objective and Scope of the Chemistry Program**

The objective of the COP Chukchi Sea Environmental Studies Program is to develop baseline information about the marine environment in the Burger and Klondike Prospect areas for submission to the MMS. The objective of the Chemical Characterization component of the COP Chukchi Sea Environmental Studies Program is to determine baseline (pre-exploration and development) concentrations of metals and hydrocarbons in sediments and tissues of zooplankton, benthic invertebrates, and fish in the study area and how distribution and concentrations of these chemicals are being influenced by inter-annual environmental changes. The pre-drilling baseline information will be used as part of an analysis of potential effects of offshore oil and gas activities on the Chukchi Sea marine environment and its resources, particularly valued resource species such as bowhead whales, gray whales, walrus, some ice seals, seabirds, and fishery resources. This analysis will be used in preparation of several regulatory documents, including National Pollution Discharge Elimination System (NPDES) permits, National Environmental Policy Act (NEPA) documents, and Incidental Harassment Authorization (IHA) and Letters of Authorization (LOA) for incidental, unintentional takes of marine mammals. This report presents the results of analyses of metals and hydrocarbons in fish samples collected during the September/October 2009 field survey event.

## **2 Methods**

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This section summarizes the methods used in the field sampling and laboratory sample analyses, and the quality assurance/quality control (QA/QC) measures that were employed to ensure data quality. The field and laboratory work, including the technical procedures, are described in more detail in the original Study Plan (Battelle, 2009, Appendix D) and the 2009 field report for the ConocoPhillips environmental studies program (Norcross, et al. 2009). The 2009 fish field sampling program was conducted under the direction of and performed by University of Alaska Fairbanks fisheries scientists.

### **2.1 Field Methods**

#### **2.1.1 Sampling Design**

The sampling design is described in detail in the 2008 Field Survey Report (Exponent, 2009). The overall design of the Chemical Characterization Study field sampling is based on a stratified-random strategy, in which each of the two prospect areas was gridded for random sampling stations. The historic drill sites in each prospect were considered a central location for site-specific sampling stations, along with other fixed locations based on oceanographic and biological features (e.g., depositional basins, productive shoals, whale and walrus feeding areas, etc). Sampling stations were divided into three categories: fixed stations, site-specific historic drill site stations, and primary and secondary random stations. For the fish sampling program, fish were collected at the odd-numbered fixed stations with the exception of BF005 and KF021 (Figures 2-1 and 2-2). In total, 25 fish samples were selected for metals and PAH analyses from 12 Burger and 12 Klondike fixed stations with one duplicate sample set collected at station KF009 (Table 2-1).

**Table 2-1. Summary of fish samples collected for chemical analysis.**

Station ID	Sample ID	Date	Species Identified
<b><i>Klondike Fixed Stations</i></b>			
KF001	09-04-KF001-FC-01	9/26/2009	Arctic cod, Sculpin
KF003	09-04-KF003-FC-01	9/27/2009	Arctic cod, Sculpin
KF005	09-04-KF005-FC-01	9/29/2009	Arctic cod, Sculpin, Sand lance, Eelpout, Eel blenny
KF007	09-04-KF007-FC-01	9/27/2009	Arctic cod, Sculpin, Eelpout, Arctic flounder
KF009	09-04-KF009-FC-01	9/28/2009	Arctic cod, Sculpin, Eelpout, Sand lances, Snailfish
KF011	09-04-KF011-FC-01	9/26/2009	Arctic cod, Sculpin, Arctic flounder
KF013	09-04-KF013-FC-01	9/28/2009	Arctic cod, Sculpin, Eelpout, Snailfish
KF015	09-04-KF015-FC-01	9/29/2009	Arctic cod, Eelpout, Sculpin, Sand lance
KF017	09-04-KF017-FC-01	9/26/2009	Arctic cod, Sculpin, Eelpout
KF019	09-04-KF019-FC-01	9/28/2009	Arctic cod, Sculpin, Eelpout, Snailfish
KF023	09-04-KF023-FC-01	9/27/2009	Eelpout, Arctic cod, Sculpin
KF025	09-04-KF025-FC-01	9/29/2009	Eelpout, Arctic cod, Sculpin, Sand lance
<b><i>Burger - Fixed Station</i></b>			
BF001	09-04-BF001-FC-01	10/1/2009	Arctic cod, Sculpin, Eelpout
BF003	09-04-BF003-FC-01	10/10/2009	not identified
BF007	09-04-BF007-FC-01	10/6/2009	Eelpout, Sculpin, Arctic cod, Alligator fish
BF009	09-04-BF009-FC-01	10/9/2009	not identified
BF011	09-04-BF011-FC-01	10/1/2009	Arctic cod, Eelpout, Sculpin
BF013	09-04-BF013-FC-01	10/6/2009	Eelpout, Arctic cod, Sculpin
BF015	09-04-BF015-FC-01	10/7/2009	Eelpout, Arctic cod
BF017	09-04-BF017-FC-01	10/6/2009	Eelpout, Arctic cod
BF019	09-04-BF019-FC-01	10/7/2009	Eelpout
BF021	09-04-BF021-FC-01	10/1/2009	Arctic cod, Eelpout, Sculpin
BF023	09-04-BF023-FC-01	10/6/2009	Arctic cod, Sculpin, Eelpout
BF025	09-04-BF025-FC-01	10/7/2009	not identified



Figure 2-1. Burger Prospect Sampling Station Locations

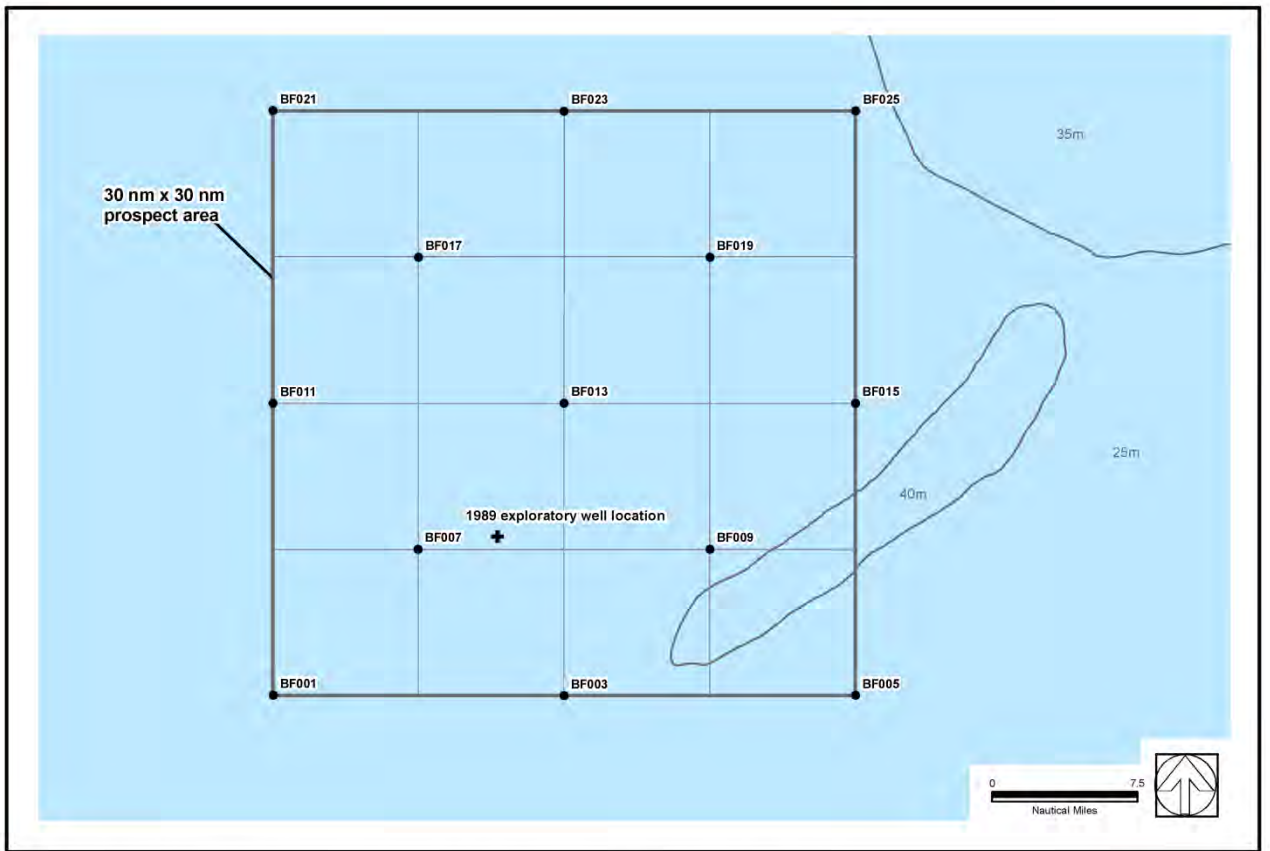


Figure 2-1. Contaminants program fixed sampling station locations in Burger



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Figure 2-2. Klondike Prospect Sampling Station Locations

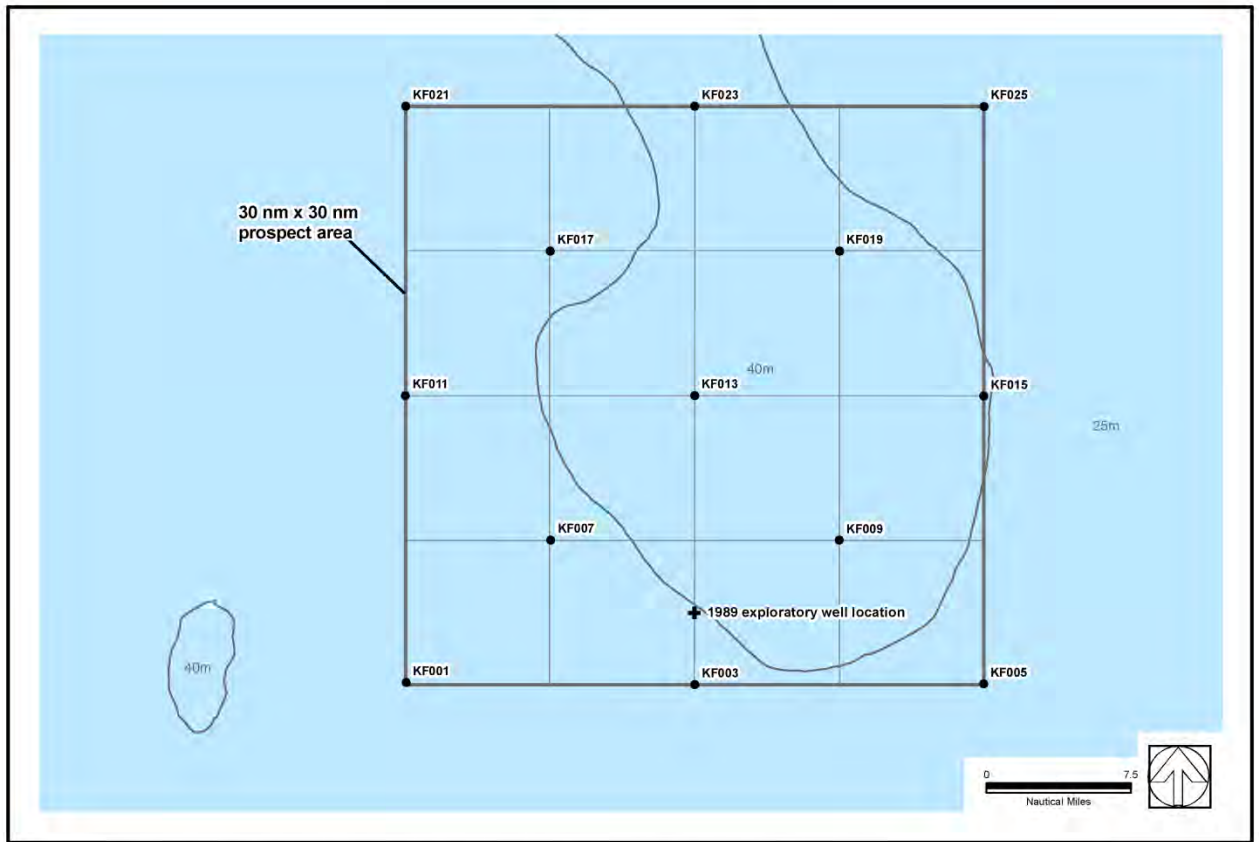


Figure 2-2. Contaminants program fixed sampling station locations in Klondike



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## **2.1.2 Field Sampling Procedures**

### **2.1.2.1 Navigation**

Each “station” was defined as a 0.2 nautical mile (nm) radius around the target station position. The actual latitude and longitude of the stations were recorded from satellite transmissions using a global positioning system (GPS). The coordinates for each sampling station are the GPS latitude and longitude values for the location where the sample was collected.

### **2.1.2.2 Equipment Decontamination**

Equipment decontamination procedures were followed at all times during sampling activities. The plastic buckets used to hold the fish were decontaminated between each sampling station, and always prior to the first sampling in each shift period. The decontamination procedure included a site-water rinse and physical removal of visible sediment debris, followed by a Liquinox™-water rinse and cleaning with scrub brushes, an additional site-water rinse, a distilled water rinse, and a wipe-down with acetone wipes. To assess potential sample contamination, QA/QC samples were collected periodically from cleaned equipment. Section 2.3.2 contains a summary of the QA/QC samples collected in this study.

### **2.1.2.3 Fish Collections**

Fish samples were collected for chemical analysis at all odd numbered fixed station locations using a beam trawl net. Each fish sample was a composite of multiple fish and fish species collected at each site. Compositing was necessary to obtain sufficient sample mass for the chemical analyses. No fish were collected at Station BF005 or KF021.

## **2.1.3 Handling of Samples**

All fish and quality control samples for chemical analysis were inventoried, recorded in a field log book and on chain of custody (COC) forms (Appendix C), and stored in secure areas on the vessel immediately after collection. Inventory included counting all of the samples to ensure that all samples were collected and safely returned to the custody area on board, documenting all samples, and preparing a COC form. Sample identification numbers (ID's) were cross-

checked against the COC logs prior to packaging samples in coolers for shipment to the analytical laboratories. Fish samples for organics and metals analysis were frozen immediately in on-board scientific freezers after collection to ensure their integrity and temperature. Sample integrity and custody was maintained at all times.

Every effort was made to deliver the samples to the analytical laboratories in a timely manner that maintained sample temperatures below 4 to 6° C. Coolers containing samples were custody sealed and samples were shipped on blue ice by priority overnight shipment. Fish samples were shipped to Battelle (Duxbury, MA). Battelle homogenized whole body fish samples by maceration with a Tissuemizer™ or blender equipped with Teflon™ gaskets and titanium probes. Battelle shipped frozen aliquots of each homogenized fish sample to Florida Institute of Technology (FIT) in Melbourne, FL for metals analyses. Battelle performed the PAH analyses on the fish tissue samples.

#### **2.1.4 Shipping of Samples**

The samples were collected in late September-October 2009 and were stored in a secured freezer for approximately three months. Samples were shipped priority overnight from Anchorage, AK to Battelle with delivery at the laboratory on January 27, 2010. All COC and custody procedures were followed and maintained throughout the collection, packaging, and shipping process. Fully executed COCs with receipt conditions reported by Battelle are presented in Appendix C. The shipping carrier was Federal Express. Samples were shipped frozen with frozen gel ice with two custody seals on the outside of each cooler and COC forms inside each cooler. No hazardous materials were included in the shipping.

## **2.2 Analytical Methods**

The fish tissue samples were analyzed for PAHs and trace metals. The analytical methods employed were originally developed, refined, and validated specifically for trace-level analysis of marine sediment and biological tissue. The analyte list includes selected hydrocarbons and metals (Tables 2-2 and 2-3) that may be present in permitted or accidental discharges during

exploratory and development activities. Additionally, these analyses may be useful in identifying potential sources of these chemicals in fish tissues. Analysis of PAH in fish tissue samples was performed at Battelle in Duxbury, MA. Trace metal analyses were performed by FIT in Melbourne, FL. Both laboratories also performed percent moisture and percent lipid determinations on these samples.

### **2.2.1 Preliminary Sample Processing**

Fish samples for the Contaminants Project were homogenized at Battelle and split for PAH and metals analyses. Sample splits were shipped to FIT for metals analysis and lipid content determination. The procedures listed below describe sample homogenization and splitting of the fish samples.

Frozen whole fish samples were completely thawed. The overlying water was poured off and the sample was homogenized in a glass jar by maceration with a Tissuemizer™ or blender equipped with Teflon™ gaskets and titanium probes as described in Battelle Standard Operating Procedure (SOP) 5-190, *Tissue Extraction for Trace Level Semi-Volatile Organic Contaminant Analysis*. An aliquot of the homogenized tissue samples was placed in a certified clean glass jars with a Teflon-lined lids, packed in a cooler with ice, and shipped to FIT via Federal Express.

### **2.2.2 Analysis of Polycyclic Aromatic Hydrocarbon (PAH)**

The fish tissue samples were analyzed for a large suite of parent and alkylated PAH (Table 2-2). The laboratory sample analysis procedures are summarized below.

**Table 2-2. Target parent and alkylated polycyclic aromatic hydrocarbons (PAH) and method detection limits.**

Compound	Reporting Code	MDL (ng/g dry wt.)	Compound	Reporting Code	MDL (ng/g dry wt.)
Naphthalene	C0N	3.60	Benzo[a]anthracene	BAA	1.32
C1-Naphthalenes	C1N	1.52	Chrysene	C0C	0.26
C2-Naphthalenes	C2N	1.52	C1-Chrysenes	C1C	0.26
C3-Naphthalenes	C3N	1.52	C2-Chrysenes	C2C	0.26
C4-Naphthalenes	C4N	1.52	C3-Chrysenes	C3C	0.26
Acenaphthylene	ACEY	0.36	C4-Chrysenes	C4C	0.26
Acenaphthene	ACE	0.45	Benzo[b]fluoranthene	BBF	0.44
Biphenyl	BIP	0.81	Benzo[k]fluoranthene	BKF	0.27
Dibenzofuran	DBF	0.68	Benzo[e]pyrene	BEP	0.20
Fluorene	C0F	1.04	Benzo[a]pyrene	BAP	0.39
C1-Fluorenes	C1F	1.04	Perylene	PER	0.62
C2-Fluorenes	C2F	1.04	Indeno[1,2,3-c,d]pyrene	IND	0.64
C3-Fluorenes	C3F	1.04	Dibenzo[a,h]anthracene	DAH	0.57
Anthracene	C0A	0.63	Benzo[g,h,i]perylene	BGP	0.99
Phenanthrene	C0P	0.66	Total PAH	TPAH	
C1-Phenanthrenes/Anthracenes	C1P/A	0.66			
C2-Phenanthrenes/Anthracenes	C2P/A	0.66			
C3-Phenanthrenes/Anthracenes	C3P/A	0.66			
C4-Phenanthrenes/Anthracenes	C4P/A	0.66			
Dibenzothiophene	C0D	0.75	<i>Surrogate Compounds</i>		
C1-Dibenzothiophenes	C1D	0.75	Naphthalene-d8	D8N	
C2-Dibenzothiophenes	C2D	0.75	Acenaphthene-d10	D10ACE	
C3-Dibenzothiophenes	C3D	0.75	Phenanthrene-d10	D10PH	
Fluoranthene	FLANT	0.52	Benzo(a)pyrene-d12	D12BAP	
Pyrene	PYR	0.58			
C1-Fluoranthenes/Pyrenes	C1F/P	0.58	<i>Recovery Internal Standard</i>		
C2-Fluoranthenes/Pyrenes	C2F/P	0.58	Fluorene-d10	D10F	
C3-Fluoranthenes/Pyrenes	C3F/P	0.58	Chrysene-d12	D12C	

### **2.2.2.1 Tissue Sample Preparation for Analysis**

Tissue samples were stored frozen at approximately -20°C until laboratory processing could begin. The 25 fish tissue samples were processed in one batch. This batch included a set of QC samples that included a procedure blank (PB), laboratory control sample (LCS), standard reference material (SRM), matrix spike (MS), and laboratory duplicate (DUP). In addition, a reference crude oil was analyzed with each batch to monitor instrument performance. Fish tissue samples were extracted as described in Battelle SOP 5-190.

Approximately 20 grams (g) of homogenized tissue was spiked with the appropriate amount of PAH surrogate internal standard (SIS) and serially extracted three times with dichloromethane by Tissuemizer™ and orbital shaker table techniques. Between extractions, the samples were centrifuged to facilitate solvent removal. The combined extract was dried over anhydrous sodium sulfate and concentrated by Kuderna-Danish and nitrogen evaporation techniques. A portion of the extract was removed prior to the concentration step to determine the total extractable organics (TEO), or total lipid weight, by a gravimetric analysis. The extracts were processed through alumina columns and fractionated on silica gel columns to isolate the hydrocarbon fraction of interest. The F2 fraction was collected, concentrated, and spiked with internal standard and analyzed for PAH by gas chromatography /mass spectrometry (GC/MS).

### **2.2.2.2 Instrumental Analysis of PAH in Tissues**

Fish tissue samples were analyzed for PAH as described in Battelle SOP 5-157, *Identification and Quantification of Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry*. The method described in the SOP is a modification of EPA SW-846 Method 8270 (USEPA, 2007), modified to include additional target compounds (e.g., alkyl PAHs), and to obtain lower detection limits and better specificity by operating the MS detector in the selected ion monitoring (SIM) mode. The target parent and alkylated PAHs are summarized in Table 2-2.

The analysis was performed with an Agilent 6890 Gas Chromatograph (GC) with an Agilent 5973 mass spectral detector (MSD). The GC is equipped with a 60-m DB-5 column (0.25-mm ID, 0.25- $\mu$ m film thickness) and a split/splitless injector, operating in the splitless mode. A data system interfaced to the GC/MS is used to control the acquisition and to store, retrieve, and manipulate mass spectral data.

Prior to the analysis of analytical standards and samples, the mass spectrometer was tuned with perfluorotributylamine (PFTBA) to maximize the sensitivity of the instrument. The GC/MS was calibrated with a 5-point calibration consisting of target compounds to demonstrate the linear range of the analysis. Typically, the calibration for this method ranges from 0.010 ng/ $\mu$ L to 10 ng/ $\mu$ L. The concentration of the low standard is 2 to 3 times the method detection limits (MDL). Calibration verification was performed at the beginning and end of each 24 hour period in which samples were analyzed. Concentrations of the individual PAH were calculated by the internal standard method. Target PAH concentrations were quantified using average response factors (RF) generated from the five-point linear calibration. Alkyl homologue PAH series concentrations were determined using the average RF for the corresponding parent compound. Well established alkyl homologue pattern recognition and integration techniques were used to identify alkyl homologues. Final concentrations were adjusted based on the recovery of the associated surrogate compound (i.e., surrogate corrected).

### **2.2.3 Analysis of Metals**

The fish tissue samples were analyzed for twelve metals (Table 2-3) that may be present in permitted or accidental discharges during exploration and development activities or that are of environmental concern because of toxicity. The laboratory sample analysis procedures are summarized below.

#### **2.2.3.1 Tissue Sample Preparation for Analysis**

The homogenized tissue samples were thawed and re-mixed with a Teflon stirring rod prior to acid digestion. The samples were then split into two portions; one subsample to be wet digested for mercury (Hg) analysis and the other to be freeze-dried and digested for analysis of 11 other



metals (Ag, As, Ba, Cd, Cr, Cu, Fe, Mn, Pb, Se and Zn: symbols defined in Table 2-3). The freeze-dried subsamples also were used to obtain data for percent water content.

Concentrations of all metals (except Hg) were determined using 5 to 7 g of wet weight tissue aliquots that were weighed in 100-mL glass digestion flasks. These subsamples were freeze-dried, reweighed for percent water content, and then digested by sequential addition of concentrated, high-purity nitric acid (HNO<sub>3</sub>) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) with gentle refluxing. In addition to the experimental fish tissue, samples of reference materials also were digested for analysis. Once the fish tissue samples and reference materials were completely digested, the clear solutions were transferred to graduated cylinders, diluted to 20 milliliters (mL) with reagent water (18-megohm resistivity) rinses of the digestion flasks, and then stored in labeled 30-mL polyethylene screw-cap bottles for metal analysis.

Mercury analyses were carried out using 1.5 to 2.5 g of wet fish tissue and dry reference materials that were weighed into 50-mL glass digestion tubes. These subsamples were digested with concentrated, high-purity HNO<sub>3</sub> and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) by refluxing at 90°C for 2 hours in the sealed tubes. The digested samples were transferred to graduated cylinders, diluted to 20 mL with reagent water (18-megohm resistivity) rinses of the digestion tubes, and then stored in labeled 30-mL polyethylene screw-cap bottles for Hg analysis.

Labware used in both the tissue digestion procedures and subsequent analyses was acid-washed with hot HNO<sub>3</sub> and rinsed three times with reagent water prior to use.

#### **2.2.3.2 Instrumental Analysis of Metals in Tissues**

Metal concentrations in the digested tissue samples, reference materials and blanks were determined by flame atomic absorption spectrometry (FAAS), inductively coupled - plasma mass spectrometry (ICP-MS) or cold-vapor atomic absorption spectrometry (CVAAS) as specified in Table 2-3. Concentrations of Cu, Fe, Mn and Zn were measured by FAAS using a Perkin-Elmer Model 4000 AAS. Concentrations of Ag, As, Ba, Cd, Cr, Pb and Se were determined by ICP-MS using a Varian Model 820-MS with a Collision Reaction Interface and a SPS-3 Sample Preparation System. Mercury concentrations were determined by CVAAS using a Laboratory

Data Control Model 1235 Mercury Monitor. In all cases, the manufacturers' specifications were followed and adherence to QA/QC requirements was maintained.

**Table 2-3. Methods, method detection limits (MDLs) and minimum concentrations of metals in fish tissue samples from this study.**

Metal	Organisms		
	Method	MDLs (µg/g dry weight)	Minimum Sample Concentrations (this study) (µg/g dry weight)
Ag – silver	ICP-MS	0.001	0.060
As – arsenic	ICP-MS	0.012	7.1
Ba – barium	ICP-MS	0.006	1.3
Cd – cadmium	ICP-MS	0.001	0.12
Cr – chromium	ICP-MS	0.003	0.25
Cu – copper	FAAS	0.7	2.9
Fe – iron	FAAS	2.5	124
Hg – mercury	CVAAS	0.001	0.023
Mn – manganese	FAAS	1.1	6.0
Pb – lead	ICP-MS	0.001	0.40
Se – selenium	ICP-MS	0.02	2.7
Zn – zinc	FAAS	0.4	71

ICP/MS = Inductively Coupled Plasma/Mass Spectrometry

FAAS = Flame Atomic Absorption Spectrometry

CVAAS = Cold Vapor Atomic Absorption Spectrometry

## 2.3 Quality Assurance/Quality Control

A quality assurance (QA) plan, which included all specific quality control (QC) measures, was employed for this program. Laboratory QA procedures were documented in the Survey Plan (Battelle, 2009) and Field Survey Report (Exponent, 2009), and laboratory procedures were documented in project-specific quality assurance project plans (QAPPs) and/or the laboratory's SOPs. The following sections present key elements of the plan.

### **2.3.1 Quality Assurance**

All project activities conducted by Battelle followed a Quality System described in Battelle's Quality Management Plan (QMP). Battelle's Quality Assurance Manual (QAM) details the application of the Quality System specifically to Battelle's Analytical and Environmental Chemistry Laboratory and other operations. Similar QA/QC procedures were in place at Florida Institute of Technology, where key project data also were generated.

Specific project activities were defined in a laboratory QAPP that was prepared by the Project's Task Leader and reviewed by the Project Manager. The Quality Assurance Unit (QAU) at Battelle monitored the analytical components of the project according to existing Battelle SOPs to ensure the accuracy, integrity, and completeness of the data. All sample receipt, storage, preparation, analysis, and reporting procedures followed written SOPs. Project staff members were responsible for following these procedures and ensuring that data quality objectives (DQOs) were achieved. In the event that DQOs were not met, the analytical staff documented all corrective actions taken related to the exceedances. The task leader reviewed and approved corrective actions. An independent QC Chemist reviewed all sample preparation and analytical documentation for completeness and accuracy and conducted full error checking of reported project data. The task leader was responsible for ensuring that project objectives were met and that the data were traceable and defensible.

### **2.3.2 Field Quality Control**

QA/QC samples were collected as part of the sampling program to assess data quality related to field activities. All field personnel (including boat crew members) were briefed on the potential for contamination and cross-contamination of samples and were given guidance on techniques to avoid such problems (e.g., cigarette smoking). This included the use of pre-cleaned sample containers; the use of clean sampling equipment; the use of the decontamination protocol described above; and good laboratory practices in general. It also included following specified sampling procedures and protocols in accordance with Exponent SOPs. Several types of field quality control samples were collected during the survey, including equipment blanks, field blanks, and replicate (duplicate) samples.

### **2.3.2.1 Replicate Samples**

Duplicate fish samples were collected at one sampling location in Klondike (KF009) to assess the sample heterogeneity and sample collection reproducibility.

### **2.3.2.2 Field and Equipment Blank Samples**

Equipment blank samples were collected as a distilled water rinse of a decontaminated bucket used during the field investigation. Field blank samples consisted of blank sample jars and site-seawater pumped through the pump hoses. The equipment blanks and field blank samples were not analyzed. These samples were held in frozen archive awaiting analysis in case contamination issues were suspected with the samples.

### **2.3.3 Laboratory Quality Control for Hydrocarbon Analysis**

Quality control (QC) is an integral part of the laboratory activities. It demonstrates the quality of operations and analyses, provides analysts with metrics about method performance, and aids project managers in identifying and correcting systematic and random problems that can plague field and laboratory operations, and in interpreting the results. QC procedures to assure analytical integrity included the following:

- Documentation of method detection limits
- Documentation of analytical accuracy
- Documentation of analytical precision
- Documentation of potential background laboratory interference/contamination

#### **2.3.3.1 Quality Control Samples**

A routine set of QC samples accompanied the batch of fish samples were processed and analyzed for PAH analysis. The following QC samples were analyzed with the batch of fish samples:

- Procedural Blank (PB) - A procedural blank is combination of solvents, surrogates, and all reagents used during sample processing, processed concurrently with the field

samples. It is intended to monitor purity of reagents and potential laboratory background contamination.

- Laboratory Control Sample (LCS) - An LCS sample is a contaminant-free matrix-specific sample (e.g., clean *Tilapia* tissue) that is prepared with each processing batch. It is spiked with the analytes of interest and processed identically to the field samples to assess the analyte recovery and method accuracy in the absence of a field sample matrix.
- Matrix spike (MS) - A matrix spike is a field sample spiked with the analytes of interest at approximately  $10 \times$  the MDL, processed concurrently with the field samples. It is intended to monitor the analyte recovery and method accuracy in the presence of a field sample matrix.
- Sample duplicate (DUP) - A duplicate is a second aliquot of a field sample processed and analyzed to monitor analytical precision. The duplicate may be a second matrix spike sample.
- Standard reference material (SRM) - A standard reference material is a field sample with certified and naturally incurred analyte concentrations. NIST SRM 2977 (mussel tissue) was prepared and analyzed to assess the accuracy of the analytical procedures.
- North Slope Crude (NSC) Reference Oil - A NSC oil sample is used to evaluate the instrumental accuracy and also provides petroleum pattern information, aiding in the qualitative identification of target analytes. The NSC is only prepared for organic compound analysis.
- Surrogate Internal Standards (SIS) - Four SIS compounds are spiked into each field and quality control sample prior to organic compound extraction and analysis. The surrogate recoveries provides a measure of the overall sample extraction and processing efficiency. SIS compounds are only added for organic compound analysis.

A set of DQOs was established for the program to ensure that the analytical data would be of the quality necessary to achieve the project objectives. The DQOs were included in the laboratory QAPPs specific for the project. The DQO for each QC parameter listed above is presented in Table 2-4.

**Table 2-4. Data quality objectives for hydrocarbon analysis.**

<b>QC Sample Type</b>	<b>Data Quality Objective</b>	<b>Corrective Action</b>
Procedural Blank	Hydrocarbons: < 5xMDL, or field sample concentration >5xMDL.	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented
Laboratory Control Sample	Hydrocarbons: 70 – 130% Recovery	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented
Matrix Spike	Hydrocarbons: 70 – 130% Recovery Spike levels >5x unspiked field sample concentration for DQO to apply.	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented
Duplicate	Hydrocarbons: RPD < 30% Field sample concentration >5x MDL for DQO to apply.	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented
Standard Reference Material	Hydrocarbons: Values must be within 30% of the certified value on average for all compounds, not to exceed 35% of the certified value for more than 30% of the compounds. Target concentration > 5x MDL for DQO to apply.	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented
Control Oil	Hydrocarbons: < 30% Difference from control values for 90% of the analytes. Concentration > 5x MDL for DQO to apply.	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented
SIS Recovery	Hydrocarbons: 40 – 120% recovery	Results examined by PM or task leader. Corrective action (re-extraction, re-analysis) or justification documented.
Initial Calibration	Hydrocarbons: < 25% RSD	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented
Continuing Calibration	Hydrocarbons: < 25% PD	Re-extraction, re-analysis, and/or document and justify – determined by PM; all corrective actions documented

### **2.3.3.2 Method Detection Limits (MDL)**

The MDL is defined as the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. Reporting limits (RL) are defined by the sample concentration of a compound that is equivalent to the final extract concentration based on the low calibration standard concentration. Target compounds confidently detected below the reporting limit (RL; typically down to a concentration using a signal-to-noise ratio criteria of approximately 3:1) will be reported and qualified appropriately, regardless of how it

compares to the calculated MDL. The approximate MDLs and RLs for PAHs in fish tissue samples are 0.20-3.6 ppb and 5.0 ppb, respectively, based on a sample mass of 2.0 grams dry weight. Sample specific MDLs and RLs are determined based on adjustments for sample preparation factors including mass extracted, final extract volume, dilution, and percent moisture.

### **2.3.4 Laboratory Quality Control for Metals Analysis**

Each tissue sample received by the Marine & Environmental Chemistry Laboratories at FIT was carefully inspected to ensure that it was intact and that the identification number on the sample container matched that found on the custody sheet. All tissue samples for metal analysis were kept frozen (-20°C) until processed for analysis.

Electronic balances used for weighing samples and reagents were calibrated prior to each use with certified (NIST traceable) standard weights. All pipettes (electronic or manual) were calibrated prior to use. Each of the spectrometers used for metal analysis was initially standardized with a three- to five-point calibration with a linear correlation coefficient of  $r \geq 0.999$  required before experimental samples could be analyzed. Analysis of complete three- to five-point calibrations and/or single standard checks alternated every 5 to 10 samples until all the analyses were complete. The relative standard deviation (RSD) between complete calibration and standard check was required to be <15% or recalibration and reanalysis of the affected samples were performed.

#### **2.3.4.1 Quality Control Samples**

For this project, QC measures included balance calibration, instrument calibration (FAAS, CVAAS, ICP-MS, TOC analyzer), matrix spike analysis for each metal, duplicate sample analysis, reference material analysis, procedural blank analysis and standard checks. With each batch of up to 40 samples, two procedural blanks, two reference materials, two duplicate samples and two matrix-spiked samples were analyzed. Analytical QC samples include:

- Procedural Blank (PB) - Two procedural blanks were prepared with each set of up to 40 samples to monitor potential contamination resulting from laboratory reagents, glassware and processing procedures. These blanks were processed using the same analytical scheme, reagents and handling techniques as used for the experimental samples.
- Matrix Spike (MS) – Matrix spikes were prepared for a minimum of 5% of the total number of samples analyzed and included each metal to be determined. Results from matrix spike analysis using the method of standard additions provided information on the extent of any signal suppression or enhancement due to the sample matrix. If necessary (i.e., spike results outside 80-120% limit), spiking frequency was increased to 20% and a correction applied to the metal concentrations of the experimental samples.
- Duplicates (DUP) – Duplicate samples from homogenized field samples (as distinct from field replicates) were prepared in the laboratory for a minimum of 5% of the total samples. These laboratory duplicates were included as part of each set of sample digestions and analyses and provide a measure of analytical precision.
- Standard Reference Materials (SRM) – A common method used to evaluate the accuracy of environmental data is to analyze CRMs and SRMs, samples for which consensus or "accepted" analyte concentrations exist. The following reference materials were used: Marine Sediments, MESS-3 (NRC); Soil (SRM 2709 with certified value for Ba), Mussel Tissue 2976 (NIST); Oyster Tissue 1566b (NIST). Metal concentrations obtained for the reference materials were required to be within  $\pm 20\%$  of accepted values for  $>85\%$  of other certified analyses. When no certified values existed for a metal, matrix spikes were used to evaluate analytical accuracy.

The DQOs for these QC measurements are provided below in Table 2-5.

#### **2.3.4.2 Method Detection Limits**

The MDL is defined as the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The RL for metals is defined as 5 times the MDL. MDLs for metals in tissues are summarized in Table 2-3.



**Table 2-5. Data quality objectives for metal analysis.**

<b>Parameter</b>	<b>Minimum Frequency</b>	<b>Data Quality Objective</b>
Initial Calibration	Prior to every batch of samples	3- to 5-point curve depending on the element and a blank. Standard curve correlation coefficient $r > 0.999$ for all analytes.
Continuing Calibration	Must end every analytical sequence; for FAAS and CVAAS, repeat all standards every 5 samples; for ICP/MS recheck standard after every 8 to 10 samples.	RSD <15% for all analytes.
Standard Reference Materials	One per batch of 20 samples	Values must be within 20% of accepted values for >85% of the certified analytes and within 25% for Hg.
Method Blank	One per batch of 20 samples	No more than 2 analytes to exceed 5 times MDL unless analyte not detected in associated samples.
Matrix Spike and Spike Method Blank	One per batch of 20 samples	RSD 80 to 120%
Laboratory Duplicate	One per batch of 20 samples	RSD <25% for 65% of the analytes.

## 3 Results

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This section summarizes the results of the PAH and trace metals analyses in fish tissues samples collected from the Burger and Klondike prospect areas.

The detailed fish tissue chemistry results for each site and parameter are summarized in Appendices A and B. All PAH and metal data in this report are presented on a dry weight basis, unless otherwise noted. The use of dry weight to report chemical concentrations reduces data variability caused by variations in the amounts of water retained by the tissues, and provides for a more reliable data comparison. All PAH data were adjusted based on the recovery of the associated surrogate compound in the sample (i.e., surrogate corrected). The main purpose of the correction is to account for sample loss that may have occurred during sample processing, and more accurately represent the native sample concentration.

All of the fish samples processed for PAH and trace metal analysis contained a composite of different fish species. A composite was used because individual fish specimens were generally small (<10 cm) in length and several fish were needed to obtain sufficient mass for analysis of both metals and PAHs. Furthermore, sufficient amounts of the same fish species were not found at each station. The species collected were primarily Arctic cod, Sculpin and Eelpout. Whole body fish samples were processed for this study. Due to the mixture of fish species per sample, no interspecies comparisons could be performed as part of this study and variability between samples may be related to differences in the mixture of fish species.

### 3.1 Polycyclic Aromatic Hydrocarbons

Concentrations of individual and total PAH were measured in fish samples collected in the Burger and Klondike prospect areas. Table 3-1 presents the individual sample results for select PAH parameters. Table 3-2 presents the concentration ranges and summary statistics of several PAH parameters for the Klondike and Burger prospects. All sample and quality control results are presented in Appendix A.

### **3.1.1 PAH Concentrations in Fish Tissue**

Total PAH (TPAH) in the Burger and Klondike fish tissue samples were detected at very low concentrations ranging from 15 to 28 ug/Kg, with all TPAH results reported at levels less than the MDLs. These results showed low between sample variability with a relative standard deviation for TPAH that was <18% for all samples [ $RSD = (\text{standard deviation}/\text{mean}) \times 100\%$ ]. Figure 3-1 graphically presents the TPAH concentrations for the Burger and Klondike samples.

**Table 3-1. PAH results in fish samples in fish tissue samples collected at fixed stations in the Burger and Klondike prospect areas.**

Sample Identification	% Moisture	% Lipid	Total PAHs (ug/Kg)	Pyrogenic PAH (ug/Kg)	Petrogenic PAH (ug/Kg)	% Petrogenic	LPAH (ug/Kg)	HPAH (ug/Kg)	% LPAH
<b>Burger</b>									
09-04-BF001-FC-01	80.79	2.52	21.3	3.3	16.7	78%	17.1	4.2	80%
09-04-BF003-FC-01	81.94	2.94	28.0	5.0	21.2	76%	18.5	9.6	66%
09-04-BF007-FC-01	81.97	2.98	22.2	3.1	17.5	79%	17.0	5.1	77%
09-04-BF009-FC-01	82.62	2.43	18.0	2.8	14.8	82%	15.1	2.9	84%
09-04-BF011-FC-01	82.40	2.38	20.7	3.3	16.5	79%	15.9	4.9	77%
09-04-BF013-FC-01	83.50	2.53	22.6	3.6	17.4	77%	17.0	5.6	75%
09-04-BF015-FC-01	79.34	4.17	19.2	3.2	15.5	81%	14.8	4.3	77%
09-04-BF017-FC-01	81.69	3.59	22.3	3.3	18.4	83%	18.1	4.2	81%
09-04-BF019-FC-01	80.77	3.52	17.4	2.4	14.4	83%	15.0	2.4	86%
09-04-BF021-FC-01	81.70	3.48	19.8	2.7	16.5	84%	15.5	4.3	78%
09-04-BF023-FC-01	82.76	2.84	17.3	2.5	14.3	82%	15.2	2.1	88%
09-04-BF025-FC-01	83.99	3.03	15.6	2.2	13.0	83%	13.7	1.9	88%
<b>Klondike</b>									
09-04-KF001-FC-01	82.18	3.29	17.8	1.8	15.4	87%	16.3	1.5	92%
09-04-KF003-FC-01	82.35	3.37	17.7	1.9	15.2	86%	16.3	1.4	92%
09-04-KF005-FC-01	81.14	3.39	14.4	1.7	12.2	84%	13.1	1.3	91%
09-04-KF007-FC-01	82.87	3.03	19.2	2.3	16.4	86%	17.3	1.9	90%
09-04-KF009-FC-01	82.39	2.97	15.1	1.8	12.8	85%	13.7	1.4	91%
09-04-KF009-FC-02	83.46	2.34	14.3	1.9	11.9	83%	12.8	1.5	90%
09-04-KF011-FC-01	82.87	3.32	19.6	2.9	16.0	82%	17.5	2.1	90%
09-04-KF013-FC-01	83.27	2.85	15.2	1.6	13.1	86%	14.0	1.2	92%
09-04-KF015-FC-01	81.84	2.45	13.5	1.6	11.4	85%	12.3	1.2	91%
09-04-KF017-FC-01	81.22	3.83	17.2	1.6	15.0	87%	16.0	1.2	93%
09-04-KF019-FC-01	84.89	2.15	14.4	1.6	12.3	85%	13.2	1.2	92%
09-04-KF023-FC-01	82.39	3.39	20.9	2.8	17.5	84%	18.6	2.2	89%
09-04-KF025-FC-01	80.94	3.13	21.6	2.2	18.8	87%	18.6	2.9	86%

**Table 3-2. Summary statistics of PAHs parameters in fish tissue samples collected at fixed stations in the Burger and Klondike prospect areas.**

Hydrocarbon Type	Burger				Klondike			
	mean	SD	min	max	mean	SD	min	max
Total PAHs (ug/Kg)	20.37	3.29	15.64	28.00	17.16	2.73	13.50	21.56
Pyrogenic PAH (ug/Kg)	3.12	0.72	2.25	4.99	1.98	0.46	1.58	2.91
Petrogenic PAH (ug/Kg)	16.34	2.19	12.98	21.18	14.63	2.35	11.45	18.83
Pyro:Petro PAH	0.19	0.02	0.16	0.24	0.14	0.02	0.11	0.18
Total PAH less Perylene(ug/Kg)	19.97	2.88	15.64	26.64	17.16	2.73	13.50	21.56
Perylene (ug/Kg)	0.41	0.53	0.00	1.36	0.00	0.00	0.00	0.00
LPAH (ug/Kg)	16.08	1.45	13.73	18.45	15.54	2.29	12.27	18.64
HPAH (ug/Kg)	4.30	2.05	1.91	9.55	1.62	0.54	1.15	2.92
LPAH/TPAH	0.79	0.06	0.66	0.88	0.91	0.018	0.86	0.93

The summary results listed above represent the following PAH parameters:

- Total PAH (TPAH) – the sum of the 42 parent and alkylated PAH isomer groups (Table 2-2)
  - Low molecular weight PAH (LPAH) – the sum of 2- and 3-ring PAH; LPAH, particularly the alkyl homologues, are frequently associated with crude oil and refined and residual petroleum products (petrogenic source)
  - High molecular weight PAH (HPAH) – the sum of 4-, 5-, and 6-ring PAH; parent (unalkylated) HPAH are primarily derived from the combustion of fossil fuels (pyrogenic source). Alkyl chrysenes are diagnostic of a crude or heavy oil source
  - Pyrogenic PAH – the sum of combustion related PAHs including: anthracene, phenanthrene/2, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(e)pyrene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenz(a,h)anthracene, and benzo(g,h,i)perylene
- Petrogenic PAH – the sum of petrogenic related PAHs including: C0-C4 naphthalenes, acenaphthylene, acenaphthene, C0-C3 fluorene, phenanthrene/2, C1-C4 phenanthrenes/anthracenes, C0-C4 dibenzothiophenes, C1-C3 fluoranthenes/pyrenes, C1-C4 chrysenes

- Pyrogenic:Petrogenic PAH – the ratio of pyrogenic to petrogenic PAH useful in differentiating hydrocarbon source type
- Total PAH less Perylene – Total PAH less the concentration of the biogenic PAH perylene
- Perylene – the only biogenic PAH included in the target PAH compound list, formed during the early diagenesis in marine sediments and may be associated with terrestrial plant sources

### 3.1.2 PAH Composition

Figure 3-2 shows the PAH profiles for the Burger and Klondike samples based on the averaged PAH concentrations in fish tissues from the two prospect areas. Sample specific PAH profiles are presented in Appendix A. All samples exhibit a predominant petrogenic PAH signature. On average, the petrogenic PAHs account for approximately 83% of the Total PAH mass detected in the samples. Pyrogenic PAH and perylene content account for the remaining Total PAH mass. The averaged PAH profiles show some differences in the fish tissue composition for these two sites – the Klondike samples show a higher relative contribution of the 3-ring PAHs and the Burger samples show a higher contribution from the 5- and 6-ring PAHs. Apparent differences in these profiles may be related to increased analytical variability at trace concentration, differences in the fish species caught and composited for each station, or the amount of sediment present in samples.

The most interesting aspect of the PAH assemblages in fish tissue samples was the absence of dibenzothiophene and alkyl-dibenzothiophenes. Dibenzothiophenes are common in petroleum (especially high-sulfur crude and refined products) and are rare in pyrogenic PAH assemblages. The absence of dibenzothiophenes in Chukchi Sea fish tissues could be due to low concentrations in local environmental sources of PAH, lack of bioaccumulation of dibenzothiophenes by fish, or efficient and rapid metabolism and elimination of dibenzothiophenes by the fish.

### **3.1.3 Comparison of PAH Results with other Studies**

The Total PAH results in the Chukchi fish tissue samples (15 to 28 ug/Kg) are consistent with the tissue concentrations found in other chemical characterization studies in the Arctic. Neff et al. (2009b) reported Total PAH concentrations, ranging from 2 to 92 ug/Kg (dry weight), in tissues of eight species fish collected as part of MMS's cANIMIDA study in the Beaufort Sea during 2004 through 2006. Several species were collected as part of both studies, i.e., Arctic cod, sculpin, and Arctic flounder. Total PAH concentrations reported in Burger and Klondike marine invertebrate samples (Neff et al., 2009a) collected from the area in 2008 ranged from 23 to 360 ug/Kg (dry weight). The PAH profiles of the Beaufort Sea fish samples and the Chukchi invertebrate samples were similar to the Chukchi fish samples with a predominance of petrogenic PAHs and lesser contributions from pyrogenic and biogenic PAH.

Figure 3-1. Total PAH Concentration in Burger and Klondike Fish Tissue Samples

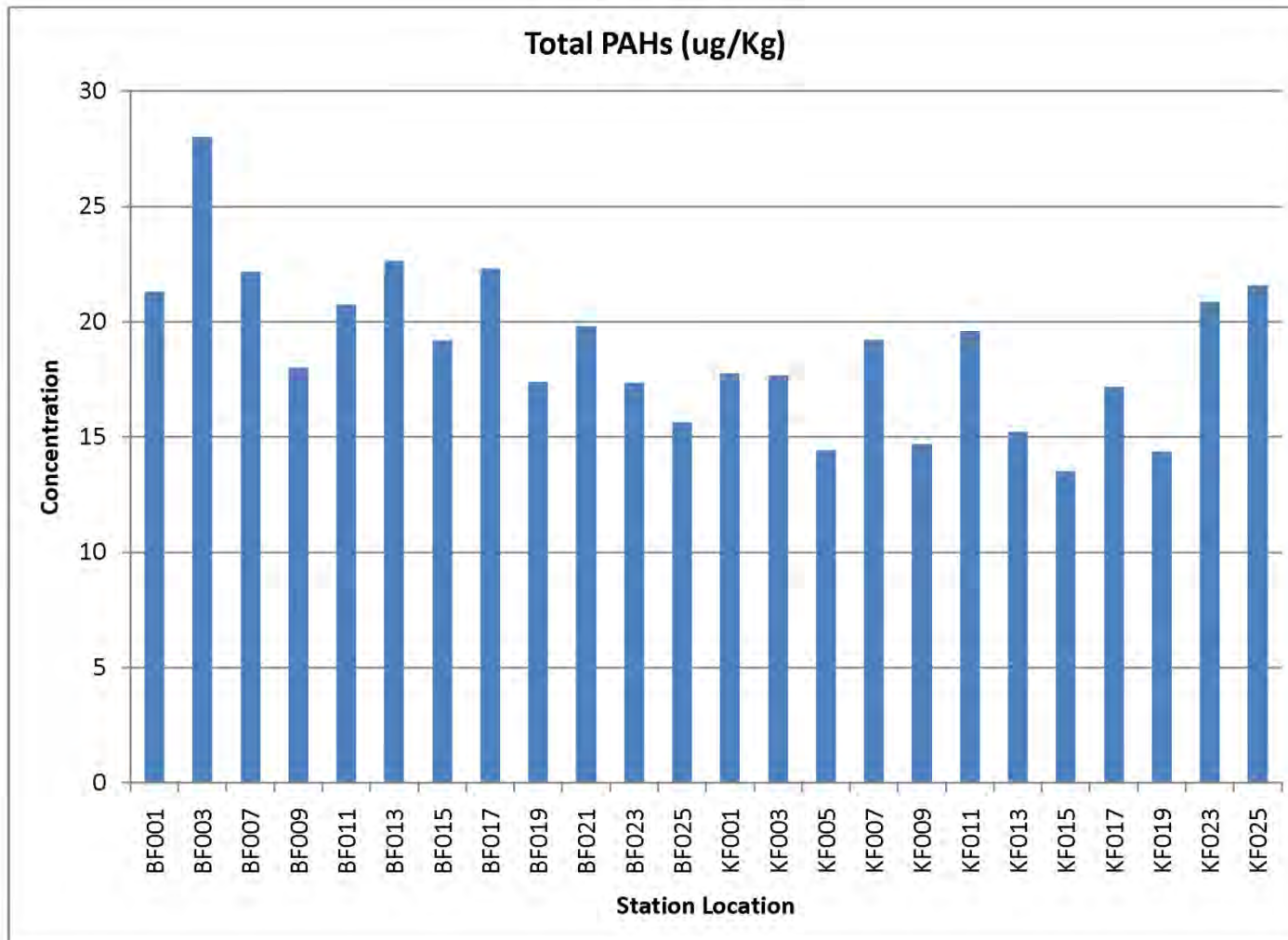
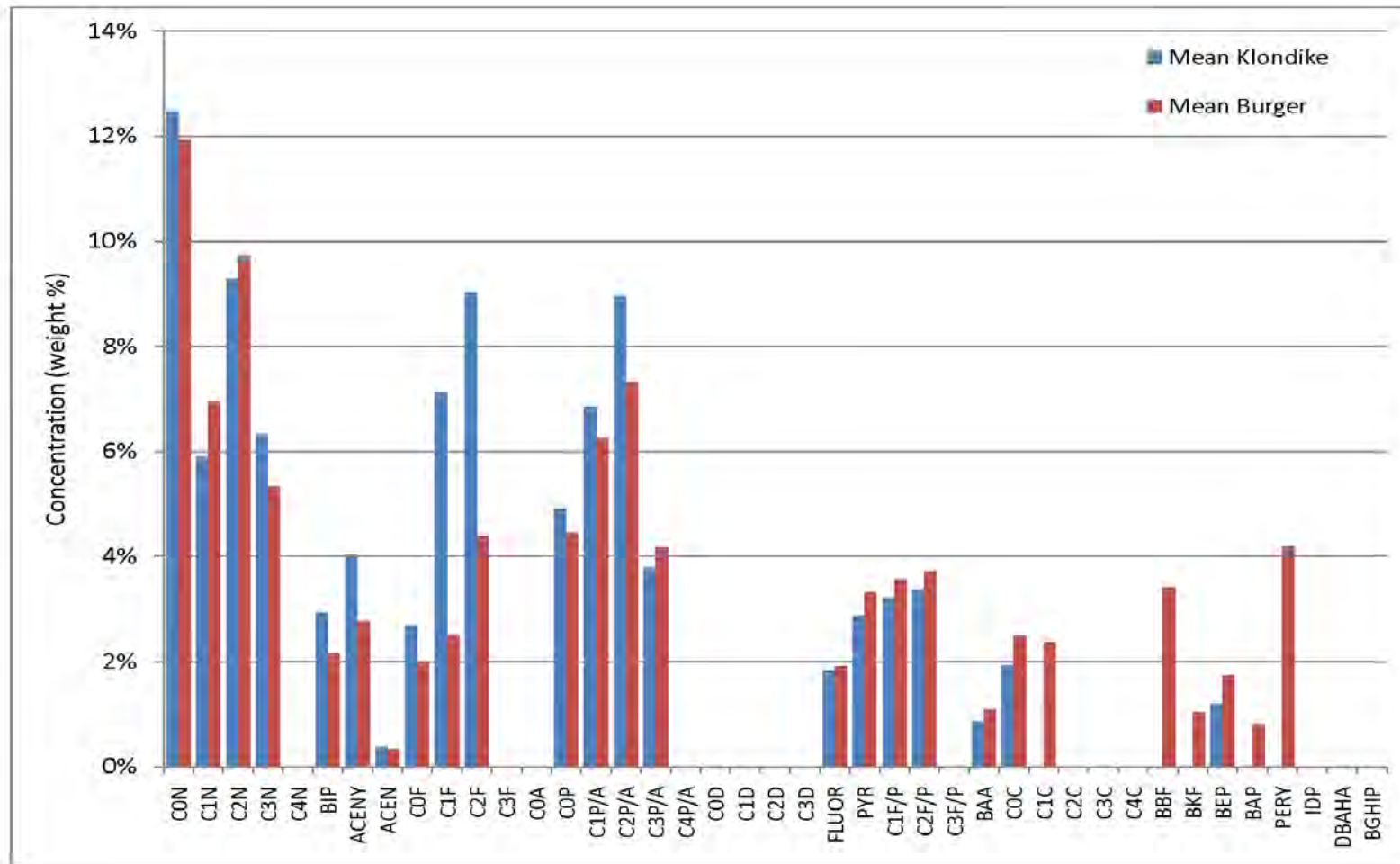




Figure 3-2. Profiles of Mean Concentrations of Individual PAH in Tissues of Fish from Burger and Klondike Collected in 2009



## 3.2 Metals

Most of the fish samples processed for metal analysis contained a composite of different fish species. Despite the mixed character of each sample, concentrations of Cu, Se and Zn in all whole fish samples from this study were rather uniform with relative standard deviations [RSD = (standard deviation/mean) x 100%] that were <15% (Tables 3-3 and 3-4, Figures 3-3f, 3-4e, 3-4f, 3-5 f, and 3-6 e, f). Although the RSDs for Cu, Se and Zn in all samples was relatively low, average values for Cu, Se and Zn in the composite samples from the Klondike area were 10 to 14% higher than found for samples from the Burger area. Due to the small standard deviations for Cu, Se and Zn in samples from each area (Table 3-4), the means from these two sets of 12 samples were significantly different (t-test, 2-tailed,  $\alpha = 0.05$ ). However, this significant difference has limited value because of the mixed fish species in each sample. Concentrations of Cu, Zn, and probably Se are homeostatically well regulated in fish by biochemical processes (e.g., Cross and Brooks, 1973; Wiener and Giesy, 1979) and the observed concentrations of Cu and Zn are believed to be at background values for the Chukchi Sea. Likewise, concentrations of Se have been shown to be regulated in fish (Beijer and Jernelöv, 1978), an observation that is consistent with the results from the present study.

Somewhat more variability was found among all samples for concentrations Ag, As, Hg and Mn with RSDs that ranged from 20 to 31% (Table 3-4 and Figures 3-3, 3-4, 3-5 and 3-6).

Concentrations of Mn in fish samples also are homeostatically controlled (Cross and Brooks, 1973) and the RSD of 26% for all Mn values is reasonably consistent with such regulation (Figures 3-4c and 3-6c). Opposite to results for Cu, Se and Zn, the average Mn value for whole fish from the Burger area was significantly greater (by 29%) than found for fish from the Klondike area (Table 3-3). Oxidic marine sediments contain high concentrations of Mn in the form of insoluble Mn oxyhydroxides. Some of this Mn is released in dissolved (bioavailable) forms if the sediments become suboxic (Trefry et al., 2003). Thus, some fish may have ingested oxidic and/or suboxic sediments.

Fish containing high Mn concentrations often contained high Fe concentrations, suggesting that these individuals had sediments in the gut. Once again, this significant difference may be due to

the different mixtures of fish in each sample. The MDLs for metals in fish tissue were typically >100 times less than the minimum concentration measured (using ICP-MS analysis, Table 3-4) and thus individual fish or tissues could be considered for future samples collection for metals.

Concentrations of total Hg in composite samples from this study were low with an average of 0.043 µg/g (dry wt.) and a range of 0.023 to 0.073 µg/g (Tables 3-3 and 3-4, Figures 3-4b and 3-6b). The RSD for the complete data set was 30% (Table 3-4) and mean values for the Burger area ( $0.045 \pm 0.013$  µg/g) were not significantly different from values obtained for the Klondike area ( $0.040 \pm 0.012$  µg/g). One sample from the Burger area (Station 15, Figure 3-4b) and one from the Klondike area (Station 25, Figure 3-6b) had total Hg concentrations that were 60% and 82% higher than the mean. The sample from Station 15 was mostly Eelpout with a few Arctic cod and the sample from Station 25 was made up predominantly of Sand lance. These differences in the species in the mixed sample are believed to be related to the differences in observed concentrations of total Hg.

**Table 3-3. Water content and trace metal concentrations (dry weight) in fish tissue samples.**

<b>Sample Identification</b>	<b>Water Content (%)</b>	<b>Ag (µg/g)</b>	<b>As (µg/g)</b>	<b>Ba (µg/g)</b>	<b>Cd (µg/g)</b>	<b>Cr (µg/g)</b>	<b>Cu (µg/g)</b>	<b>Fe (µg/g)</b>	<b>Hg (µg/g)</b>	<b>Mn (µg/g)</b>	<b>Pb (µg/g)</b>	<b>Se (µg/g)</b>	<b>Zn (µg/g)</b>
<b>Burger Stations</b>													
09-BF001-FC-01	78.4	0.14	10.8	15.9	0.38	2.08	4.1	1130	0.056	16.7	0.703	2.93	84.9
09-BF003-FC-01	78.7	0.12	11.3	7.75	0.25	1.28	3.7	525	0.037	9.6	0.402	3.07	80.2
09-BF007-FC-01	78.6	0.14	7.62	8.01	0.24	1.26	4.6	692	0.045	10.6	0.982	2.73	75.8
09-BF009-FC-01	79.3	0.084	8.65	48.4	0.22	1.08	4.2	326	0.034	10.6	0.694	2.72	81.5
09-BF011-FC-01*	79.9	0.13	9.28	9.58	0.26	1.41	4.9	1040	0.055	10.6	0.748	2.71	83.2
09-BF011-FC-01*	79.9	0.13	9.46	9.73	0.27	1.38	4.9	1030	0.057	10.5	0.732	2.76	82.4
09-BF013-FC-01	81.3	0.072	13.3	10.0	0.29	1.36	4.0	918	0.049	13.6	0.558	2.81	82.3
09-BF015-FC-01	77.0	0.060	10.5	7.99	0.17	1.43	2.9	604	0.072	9.4	0.544	3.05	83.0
09-BF017-FC-01	77.8	0.087	7.94	15.1	0.20	1.60	3.8	774	0.046	12.8	0.664	2.76	71.6
09-BF019-FC-01	77.7	0.089	8.04	7.99	0.12	1.19	3.2	675	0.051	7.4	0.610	2.83	71.2
09-BF021-FC-01	79.4	0.11	7.24	10.0	0.25	1.79	3.4	1040	0.034	13.1	0.555	3.05	77.2
09-BF023-FC-01	79.7	0.092	8.08	3.85	0.23	1.20	4.1	386	0.042	9.1	0.759	3.59	83.8
09-BF025-FC-01	81.2	0.069	7.39	2.35	0.25	0.53	4.5	124	0.023	9.3	0.520	3.39	91.3
<b>Klondike Stations</b>													
09-KF001-FC-01	80.2	0.091	10.9	1.66	0.91	0.30	4.4	153	0.041	8.2	2.29	3.42	87.5
09-KF003-FC-01	81.0	0.081	7.44	4.59	0.58	0.67	4.2	344	0.026	8.4	1.51	3.05	83.7
09-KF005-FC-01	79.0	0.10	10.0	3.22	0.46	0.39	5.1	207	0.037	7.3	1.75	3.04	86.0
09-KF007-FC-01	80.1	0.064	8.82	2.60	0.82	0.38	4.8	217	0.049	8.0	1.53	3.42	92.3
09-KF009-FC-01**	80.1	0.089	9.10	4.44	1.02	0.71	4.4	472	0.033	12.8	1.34	3.52	94.7
09-KF009-FC-02**	81.2	0.12	7.92	3.78	1.09	0.61	4.7	365	0.031	9.9	1.34	3.27	93.4
09-KF011-FC-01	80.0	0.16	11.5	1.28	0.52	0.25	4.0	131	0.036	7.5	0.557	3.60	104
09-KF013-FC-01*	81.2	0.12	10.0	2.10	0.87	0.40	4.1	216	0.036	6.6	0.740	3.51	89.9
09-KF013-FC-01*	81.1	0.12	10.0	2.33	0.87	0.40	4.2	209	0.034	6.4	0.688	3.55	89.1
09-KF015-FC-01	79.5	0.074	10.7	2.77	0.44	0.44	4.2	241	0.043	10.8	0.995	3.21	97.9
09-KF017-FC-01	79.0	0.096	14.4	1.76	0.34	0.26	4.1	144	0.040	6.0	1.20	3.44	99.8
09-KF019-FC-01	82.6	0.16	7.10	2.00	1.83	0.45	5.4	187	0.026	9.4	0.607	3.11	101
09-KF023-FC-01	79.6	0.14	8.96	4.29	0.52	0.79	4.0	218	0.041	7.7	1.13	3.20	81.4
09-KF025-FC-01	77.3	0.18	7.78	7.99	0.38	1.43	4.2	588	0.073	12.0	1.12	3.10	85.5

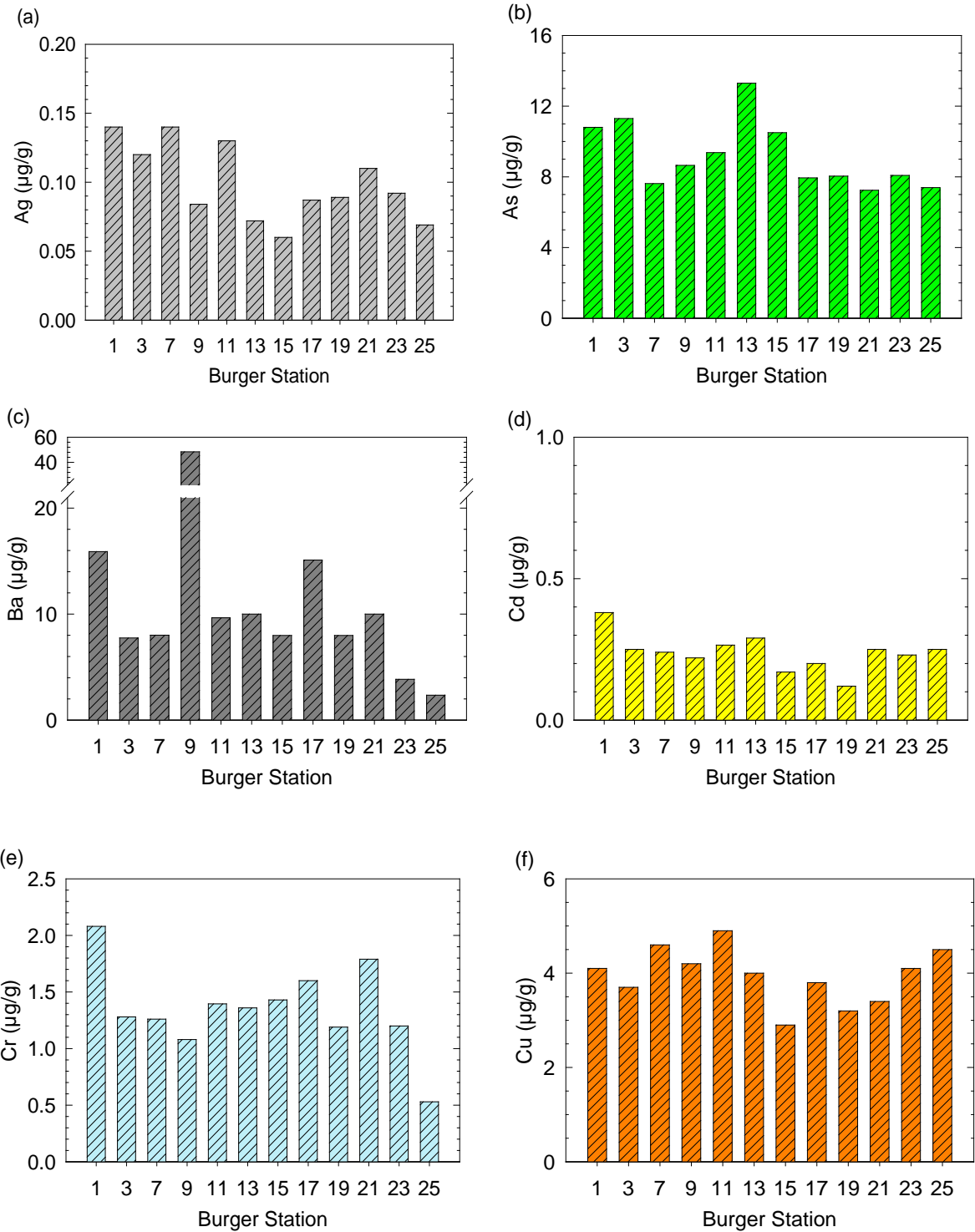
\*Laboratory duplicate.

\*\*Field duplicate.

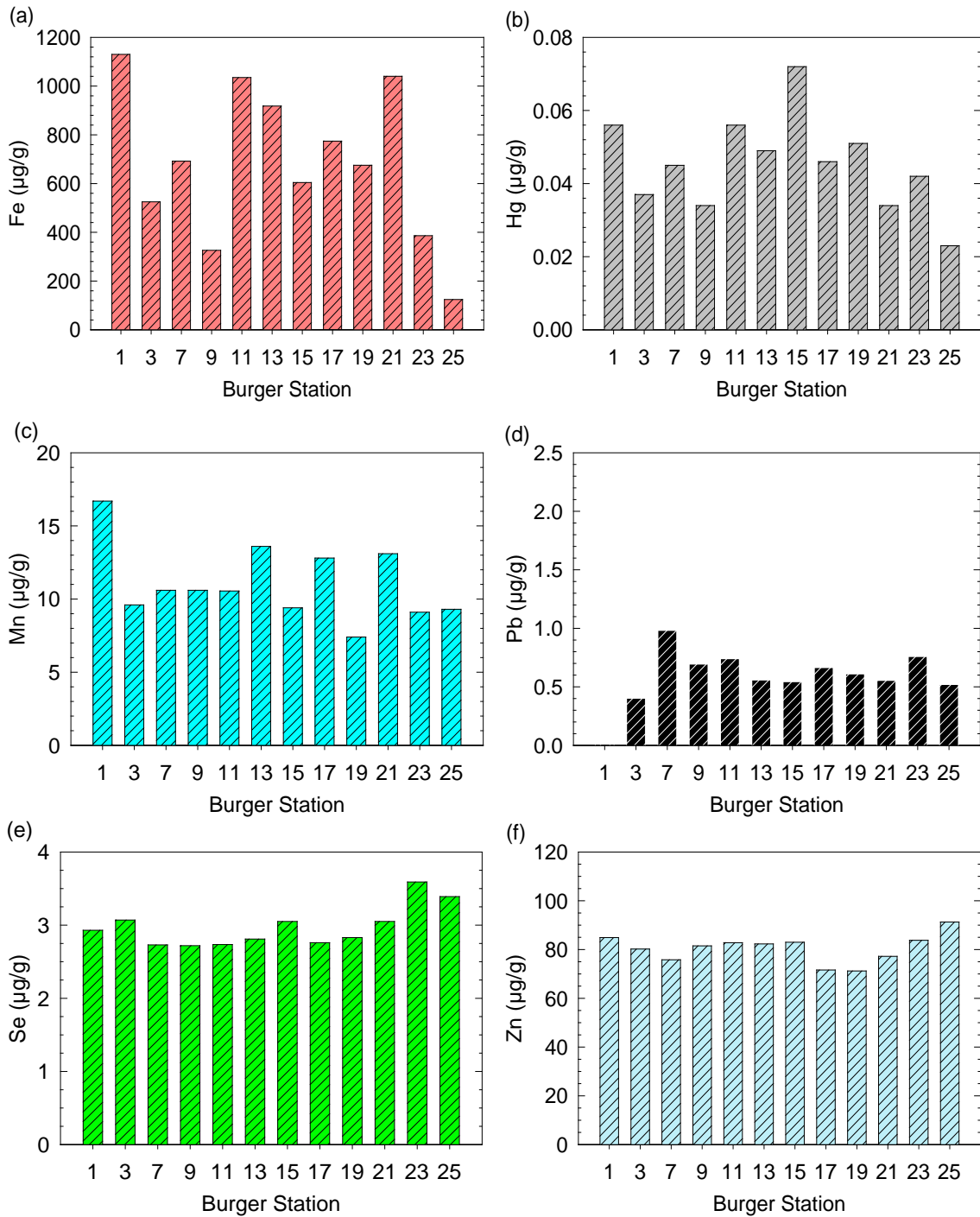
**Table 3-4. Summary statistics for trace metals (dry weight) and water content in fish tissue samples. Values for lab and field duplicates were averaged prior to statistical analysis.**

Sample Identification	Statistic	Water Content (%)	Ag (µg/g)	As (µg/g)	Ba (µg/g)	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Fe (µg/g)	Hg (µg/g)	Mn (µg/g)	Pb (µg/g)	Se (µg/g)	Zn (µg/g)	
Burger Fixed Stations	Mean	79.1	0.099	9.19	12.2	0.24	1.35	4.0	686	0.045	11.1	0.644	2.97	80.5	
	Std. Dev.	1.3	0.028	1.90	12.0	0.06	0.38	0.6	312	0.013	2.5	0.149	0.28	5.7	
	n	12	12	12	12	12	12	12	12	12	12	12	12	12	
	Maximum	81.3	0.14	13.3	48.4	0.38	2.08	4.9	1130	0.072	16.7	0.982	3.59	91.3	
	Minimum	77.0	0.060	7.24	2.35	0.12	0.53	2.9	124	0.023	7.4	0.402	2.72	71.2	
Klondike Fixed Stations	Mean	80.0	0.114	9.68	3.21	0.73	0.54	4.4	255	0.040	8.6	1.23	3.29	91.9	
	Std. Dev.	1.3	0.038	2.05	1.86	0.42	0.33	0.5	133	0.012	1.9	0.50	0.20	7.5	
	n	12	12	12	12	12	12	12	12	12	12	12	12	12	
	Maximum	82.6	0.18	14.4	7.99	1.83	1.43	5.4	588	0.073	12.0	2.29	3.60	104	
	Minimum	77.3	0.064	7.10	1.28	0.34	0.25	4.0	131	0.026	6.0	0.557	3.04	81.4	
Cumulative for Burger and Klondike stations	Mean	79.5	0.107	9.43	7.73	0.48	0.94	4.2	470	0.043	9.8	0.936	3.13	86.2	
	Std. Dev.	1.4	0.033	1.95	9.59	0.38	0.54	0.6	322	0.013	2.5	0.469	0.29	8.7	
	RSD (%)*	1.8	31	21	124	79	57	14	68	30	26	50	9	10	
	n	24	24	24	24	24	24	24	24	24	24	24	24	24	24
	Maximum	82.6	0.18	14.4	48.4	1.83	2.08	5.4	1130	0.073	16.7	2.29	3.60	104	
Minimum	77.0	0.060	7.10	1.28	0.12	0.25	2.9	124	0.023	6.0	0.402	2.72	71.2		

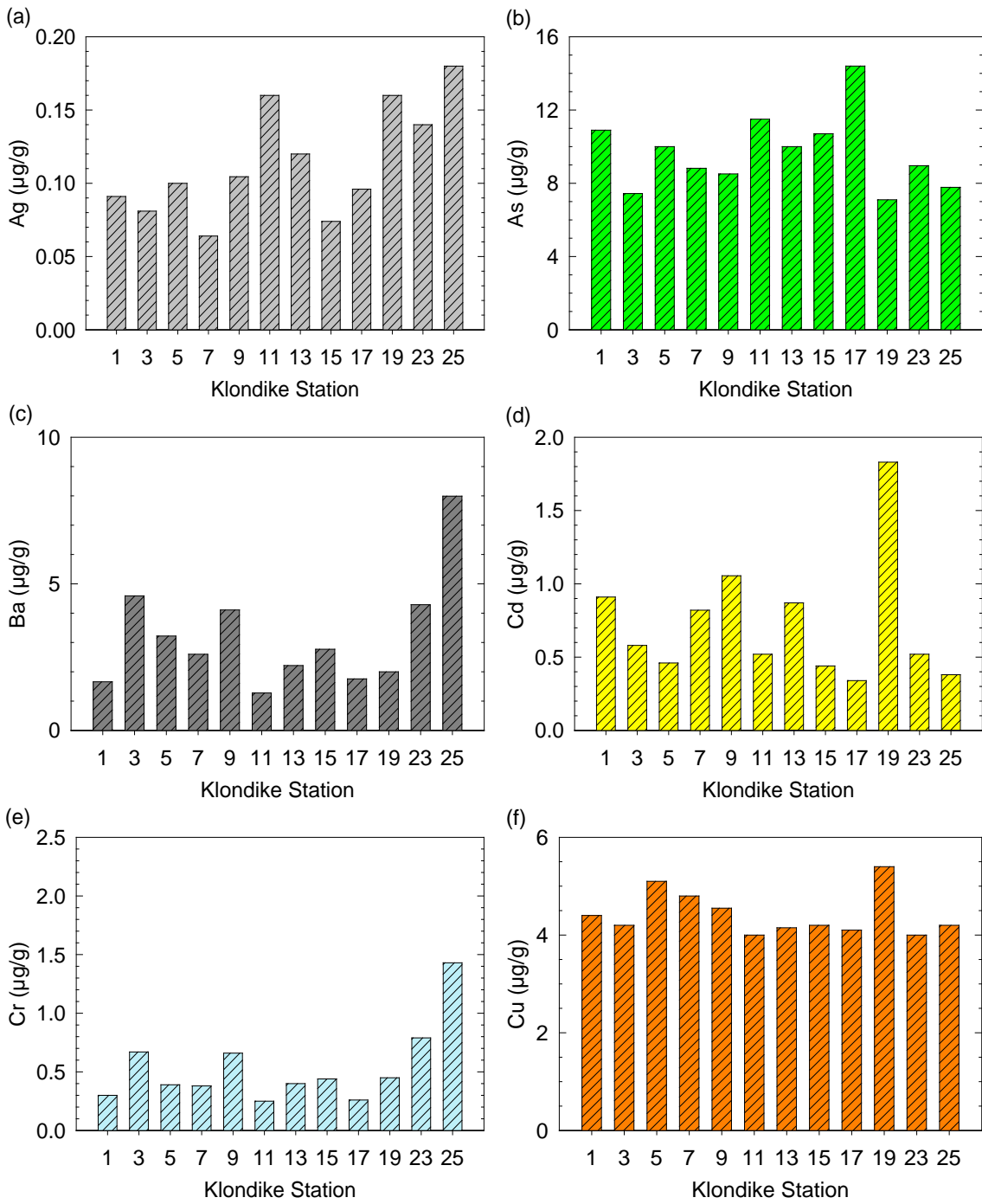
\* RSD = Relative Standard Deviation = (Standard deviation/mean) x 100%.



**Figure 3-3. Bar Graphs Showing Concentrations of (a) Ag, (b) As, (c) Ba, (d) Cd, (e) Cr and (f) Cu in Fish Samples from Stations in the Burger Area.**



**Figure 3-4. Bar Graphs Showing Concentrations of (a) Fe, (b) Hg, (c) Mn, (d) Pb, (e) Se and (f) Zn in Fish Samples from Stations in the Burger Area.**



**Figure 3-5. Bar Graphs Showing Concentrations of (a) Ag, (b) As, (c) Ba, (d) Cd, (e) Cr and (f) Cu in Fish Samples from Stations in the Klondike Area.**



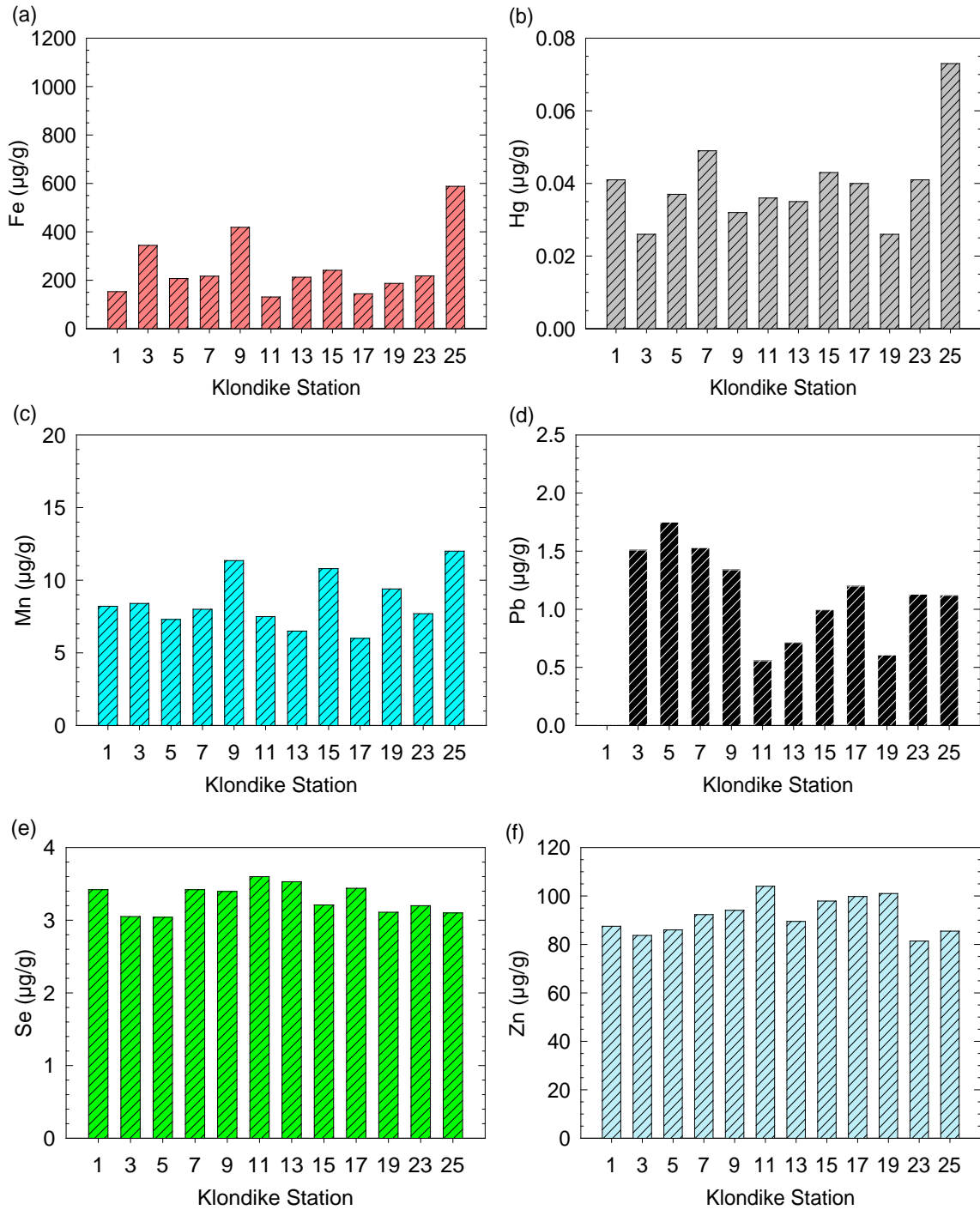


Figure 3-6. Bar Graphs Showing Concentrations of (a) Fe, (b) Hg, (c) Mn, (d) Pb, (e) Se and (f) Zn in Fish Samples from Stations in the Burger Area.

### **3.3 Quality Control**

This section provides an evaluation of the quality and usability of the environmental data based on the results for the field and laboratory QC samples collected and analyzed as described in Section 2.3.3 and 2.3.4. The results for the hydrocarbon and metals QC samples and measures are presented in Appendix C. Detailed QC narratives describing QC matters have been delivered with the analytical data.

In general, no serious data quality issues were noted that would adversely affect the quality or use of the PAH or metals data.

#### **3.3.1 Field Quality Control**

##### **3.3.1.1 Field Blanks**

Field blanks were collected during the survey, however they were not analyzed to assess potential sample contamination introduced from surficial sediment sample collection and handling procedures.

##### **3.3.1.2 Replicate Samples**

One field duplicate sample was collected at Station KF009 to assess overall precision and representativeness of the sampling and analytical efforts. For the duplicate samples collected at sampling Station KF009, the precision criterion of less than 50 percent RSD for tissues was met for all metals and PAH results detected at concentrations greater than the reporting limit.

Overall, the field replicate precision was acceptable.

#### **3.3.2 Hydrocarbon Quality Control Results**

The hydrocarbon laboratory quality control measures included recovery of surrogate compounds, evaluation of procedural blanks, laboratory control sample recoveries (matrix spikes), laboratory duplicates and standard reference material analyses (sediment certified for organic target analytes). The majority of the quality control results associated with the PAH samples met the

DQOs and acceptance limits. Minor quality control exceedances included trace level blank contamination, matrix spike recovery exceedances, and SRM recovery exceedances. Table 3-5 summarizes the PAH laboratory QC results for fish tissue samples. The surrogate recovery results, a useful measure of overall method performance, are acceptable in all samples. No serious data quality issues were observed that would adversely affect the quality or use of the PAH data. The analytical data that were generated are of high quality, and can be used with confidence. Discussion and interpretation of the results are provided in the following sections.

### **3.3.2.1 Surrogate Results**

Surrogate compounds were added to all environmental and QC samples prior to sample preparation to monitor the efficiency of the sample extraction and analysis procedures. Surrogate recoveries were evaluated to assess analytical method accuracy relative to sample matrix and laboratory performance. All surrogate recovery results were within the acceptance limits for the field samples and quality control samples.

### **3.3.2.2 Procedural Blanks**

One laboratory PB was prepared with the fish tissue sample preparation batch by extracting a blank sample matrix (sodium sulfate) as if it were one of the environmental samples. Procedural blanks are used to assess the potential of contamination introduced during sample preparation and analysis. Trace concentrations of seven PAHs (naphthalene, C1-naphthalene, biphenyl, phenanthrene, fluoranthene, pyrene, and chrysene) were detected in the tissue PBs at concentrations less than the MDL. The majority of the associated tissue sample results were within 5 times the associated PB concentration and were qualified with a “B” to indicate that the compound was also present in the blank. Results qualified with a “B” may be biased high or may be false positives.

### **3.3.2.3 Laboratory Control Sample Recoveries**

A LCS was prepared with each sample preparation batch by spiking a blank sample matrix (*Tilapia* for tissues) with known concentrations of a subset of the target compounds. Laboratory control samples are used to assess the accuracy of the sample preparation and analysis

procedures independent of sample matrix effects. The LCS was spiked with PAH matrix spike compounds. The LCS recoveries were within the acceptance limits.

#### **3.3.2.4 Matrix Spike Sample Recoveries**

A MS was prepared with the fish tissue sample batch by spiking a separate sample with known concentrations of a subset of the target compounds. MS samples are used to assess the accuracy and precision of the sample preparation and analysis procedures. The MS was spiked with PAH matrix spike compounds. For the PAH analyses, the MS sample had three recoveries outside of the acceptance limits. Benzo(b)fluoranthene recovered above the acceptance limits and indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene recovered below the acceptance limits. The MS recovery exceedances do not seriously affect the quality or usability of the associated sample data. The sample results associated with these exceedances may be biased high or low as indicated by the matrix spike recoveries.

#### **3.3.2.5 Laboratory Duplicates**

One laboratory duplicate set was prepared with each batch by extracting a second aliquot of an of the fish tissue sample. Laboratory duplicate results were evaluated to assess analytical precision related to laboratory performance. For the PAH analysis, the precision of the duplicate samples could not be accurately assessed since all reported compounds were detected at concentrations less than the reporting limit. The laboratory duplicate precision criterion does not apply to compounds detected below the reporting limit (or less than 10 times the MDL) due to increased variability at low concentrations. Nonetheless, the duplicate results were reviewed and found to show good agreement.

#### **3.3.2.6 Standard Reference Materials**

A SRM was prepared and analyzed with the fish tissue sample preparation batch to assess the accuracy of the analytical method based on measured concentrations compared to certified concentrations. All recoveries were acceptable with the exception of the recoveries of benzo(a)pyrene, perylene, and benzo(g,h,i)perylene were recovered below the certified values.

The sample results associated with these exceedances may be biased low as indicated by the SRM recoveries.

**Table 3-5. PAH QC results summary.**

<b>QC Sample or Measurement Type</b>	<b>Data Quality Objective</b>	<b>Quality Control Results Summary</b>	<b>Impact to Data Quality and Usability</b>
Initial Calibration	%RSD <25% for all compounds (up to 10% of compounds can be >25%, but <35%)	All objectives were met.	None.
Continuing Calibration	%D <25% for all compounds (up to 10% of compounds can be >25%, but <35%)	All objectives were met.	None.
Surrogate Recoveries	40 to 120% recovery	All objectives were met.	None.
Procedural Blank	No compound to exceed 5 times the MDL unless sample amount is >10 times blank amount	Seven PAH compounds were detected at concentration less than the reporting limit.	Associated sample results within 5 times the blank levels were qualified with a "B" and may be biased high or false positives.
Laboratory Control Sample Recoveries	70 to 130% recovery for spiked compounds	All objectives were met.	None.
Laboratory Duplicate	RPD <30% for all compounds >10 times the MDL	All objectives were met.	None. No target compounds were detected at concentrations great enough to be used for data quality assessment.
Matrix Spike Recoveries	70 to 130% recovery for spiked compounds	Recoveries met the MQO with a few exceptions: benzo(b)fluoranthrene, indeno(1,2,3-c,d) pyrene, and benzo (g,h,i)perylene (161%, 65%, and 50%, respectively).	Minor. The results for these compounds in the associated samples may be biased low or high.
Tissue SRM (2977)	Measured values must be within 30% of the true value on average for all compounds, not to exceed 35% of true value for more than 30% of the compounds	Recoveries met the MQO with a few exceptions: benzo(a)pyrene, perylene, and benzo (g,h,i)perylene (37%, 47%, and 48%, respectively).	Minor. The results for these compounds in the associated samples may be biased low.
Oil Reference Standard (North Slope Crude)	RPD< 30% from control values for 90% of the analytes.	All objectives were met.	None.

### **3.3.3 Metals Quality Control Results**

All Data Quality Objectives for metals in fish tissues (as listed in Table 2-5) were met for results from this study. Quality assurance results for certified reference standards, percent spike recovery and precision are listed in Table 3-6. Overall, the checks on data quality support a high quality data set for metals in these fish samples from the 2009 fish collection survey of the Chukchi Sea.

#### **3.3.3.1 Sample Tracking Procedure**

Upon receipt, the fish tissue samples received by the Marine & Environmental Chemistry Laboratories at Florida Institute of Technology were carefully inspected to insure they were intact and that the identification on each sample container matched that found on the custody sheet. All samples were received in good condition and kept frozen at -20°C until processed for analysis.

#### **3.3.3.2 Analytical QA/QC Measurements**

To insure data quality, QA/QC requirements included the following: calibration of balances, pipettes, FAAS, ICP-MS and CVAAS instruments, standard checks, matrix spike analyses for each metal, duplicate sample analyses, Certified Reference Material (CRM) and Standard Reference Material (SRM) analyses, and procedural blank analyses.

#### **3.3.3.3 Instrument Calibrations**

Electronic balances used for weighing samples and reagents were calibrated prior to each use with their internal electronic calibration and then verified with certified standard weights (NIST-traceable). All pipettes (electronic or manual) were calibrated prior to use. Each of the spectrometers used for metal analysis was initially standardized with a three- to seven-point calibration and a linear correlation coefficient ( $r \geq 0.999$ ) was required before experimental samples were analyzed. Three- to five-point calibrations and/or single standard checks were carried out after every eight samples until all analyses were complete. The RSD between complete calibration and standard check was <10% or recalibration and reanalysis of the affected samples was performed.

#### **3.3.3.4 Certified and Standard Reference Material Analyses**

The tissue trace metal and Hg digestions included one sample of the NRC fish tissue CRM DORM-2 (Dogfish muscle) and the NIST oyster tissue SRM #1566b. The NIST trace elements in natural water SRM #1640 was included for Ba, which is not certified in DORM-2, and Cr, which is not certified SRM #1566b. All metal concentrations determined for the CRM and SRMs during this study were within the limits of the certified and reference values (Table 3-6).

#### **3.3.3.5 Matrix Spike Analyses**

Matrix spikes were prepared for two of the fish tissue subsamples analyzed by FAAS and ICP-MS, and included each of the metals to be determined. Six matrix spikes were prepared for the fish tissue subsamples analyzed for Hg by CVAAS. The spike recoveries were all within acceptable limits for the analytical procedures used.

#### **3.3.3.6 Laboratory Duplicate Sample Analyses**

Two laboratory duplicates were prepared and analyzed. Analytical precision was acceptable based on percent RSD ranging from 0 to 7.3% (Table 3-6).

#### **3.3.3.7 Field Replicate Sample Analyses**

A field replicate fish tissue sample was provided with the experimental samples. The percent RSD in metal concentrations from this replicate ranged from 0 to 21.0% (Table 3-6) and was considered to be acceptable.

#### **3.3.3.8 Procedural Blank Analyses**

Two PBs were prepared with the set of fish tissue samples to monitor the potential for metal contamination. These blanks contained the same reagents and were prepared using the same handling techniques and analytical scheme as the experimental samples. Metal concentrations due to impurities in reagents were within acceptable limits, generally below the MDL.



**Table 3-6. Metals QC results summary.**

Reference Material	Ag (µg/g)	As (µg/g)	Ba (µg/g)	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Fe (µg/g)	Hg (µg/g)	Mn (µg/g)	Pb (µg/g)	Se (µg/g)	Zn (µg/g)
CRM DORM-2 (This Study)	0.035	18.4	2.64	0.037	31.4	2.2	141	4.64	3.6	0.065	1.34	26.5
CRM DORM-2 (NRC Certified Values)	0.041 ±0.013	18.0 ± 1.1	- -	0.043 ±0.008	34.7 ± 5.5	2.34 ± 0.16	142 ± 10	4.64 ± 0.26	3.66 ± 0.34	0.065 ± 0.007	1.40 ± 0.09	25.6 ± 2.3
SRM #1566b (This Study)	0.658	7.65	8.51	2.42	0.33	70.4	200	0.037	18.3	0.301	1.93	1435
SRM #1566b (NIST Certified Values)	0.666 ±0.009	7.65 ± 0.65	8.6* ± 0.3	2.48 ± 0.08	- -	71.6 ± 1.6	205.8 ± 6.8	0.0371 ± 0.0013	18.5 ± 0.2	0.308 ± 0.009	2.06 ± 0.15	1424 ± 46
SRM #1640 (This Study)	-	-	(µg/L) 146.5	-	(µg/L) 38.1	-	-	-	-	-	-	-
SRM #1640 (NIST Certified Values)	-	-	148.0 ± 2.2	-	38.6 ± 1.6	-	-	-	-	-	-	-
Analytical Precision from two sets of lab replicates**	0.0 0.0	1.4 0.0	1.1 7.3	2.7 0.0	1.5 0.0	0.0 1.7	0.7 2.3	2.5 4.0	0.7 2.2	1.5 5.1	1.3 0.8	0.7 0.6

\* Reference Value, not a Certified Value.

\*Precision presented as % relative standard deviation = (SD/mean) x 100%.

## 4 Discussion

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### 4.1 Polycyclic Aromatic Hydrocarbons

Overall, the level of Total PAHs detected in the fish tissue samples are within the range of values reported from previous studies of other Alaskan coastal continental shelf areas and are representative of background tissue concentrations. TPAH in the fish tissues of the Burger and Klondike areas of the Chukchi Sea can be attributed to the wide variety of potential sources contributing to the background hydrocarbons concentrations such as shoreline/coastal erosion, terrestrial plant material, marine plant material, natural hydrocarbon sources (seeps, kerogen containing source rock, and possibly peat and coal), and long-range atmospheric transport and deposition (Valette-Silver et al 1999). Since PAH, and other hydrocarbons, do not biomagnify in the marine food chains (Neff, 2002), trophic transfer is inefficient and hydrocarbon concentrations are unlikely to reach high concentrations in upper trophic level animals, such as birds, whales, seals, and polar bears.

The Total PAH results detected in the fish tissue samples were compared to USEPA's Fish Advisory Limits (USEPA, 2000) to assess potential human health risk associated with fish consumption, even though, the fish collected in this study are unlikely to be used for human consumption. In order to compare the reported tissue results to the USEPA Guidance levels, the results were first adjusted from a dry weight to a wet weight basis and only the parent PAHs were included in the sum of Total PAH. The current cancer slope factor (CSF) for PAH is based on benzo[a]pyrene. The concentrations for the other PAHs were adjusted to benzo(a)pyrene equivalents based on the order-of-magnitude relative potencies reported for these PAHs (Nisbet and LaGoy, 1992; USEPA, 1993). The adjusted Total PAH tissue concentrations (determined as benzo[a]pyrene equivalents) ranged from 0.0016 to 0.2001 ppb (or 0.0000016 to 0.0002 ppm). Based on this adjusted Total PAH fish tissue concentration, the risk based consumption limit is considered to be unrestricted as shown in Table 4-1.

**Table 4-1. Monthly fish consumption limits for carcinogenic health endpoint – PAHs.**

<b>Risk Based Consumption Limit<sub>a</sub></b>	<b>Cancer Health Endpoints</b>
<b>Fish Meals/Month</b>	<b>Fish Tissue Concentrations (ppm, wet weight)</b>
Unrestricted (>16)	0 - 0.0004
16	>0.0004 - 0.0008
12	>0.0008 - 0.0011
8	>0.0011 - 0.0016
4	>0.0016 - 0.0032
3	>0.0032 - 0.0043
2	>0.0043 - 0.0064
1	>0.0064 - 0.013
0.5	>0.013 - 0.026
None (<0.5)	>0.026

Source: USEPA 2000

## 4.2 Metals

Although small fish such as those collected for this study are not likely to be consumed by humans, the total Hg values obtained during this study can be placed in context with Hg advisories on fish consumption. Based on the USEPA (2001) fish advisories, monthly fish consumption limits for Hg were set at  $1 \times 10^{-4}$  mg/kg-day. This means that an adult that weighs 70 kg (154 lbs) could consume 7 µg of Hg per day [ $(1 \times 10^{-4}$  mg/kg-day) x 70 kg = 0.007 mg/day or 7 µg/day]. The USEPA (2001) set an average fish meal at 8 oz (227 g), 1 month was set equal to 30.44 days, and a monthly intake of Hg for a 70-kg human could be 213 µg [(7 µg Hg/day) x 30.44 days/mo) = 213 µg Hg/mo). Hypothetically, if fish for consumption contained Hg at 0.06 µg/g (wet wt), then the individual could eat ~16 fish meals per month and be near compliance with the Hg advisory as shown in the calculation below and in Table 5.

$$(16 \text{ meals/month}) \times (227 \text{ g fish/meal}) \times (0.06 \text{ } \mu\text{g Hg/g fish}) = 218 \text{ } \mu\text{g Hg/month}$$

If the same calculations are used with an average Hg value of 0.0088 µg/g (wet wt.) for fish from the present study of the Chukchi Sea, the number of fish meals per month would be 106.

$$(213 \mu\text{g Hg/mo}) / (0.0088 \mu\text{g Hg/g fish}) \times (227 \text{ g fish/meal}) = \sim 106 \text{ meals/month}$$

The wet weight values for fish from the present study were calculated using the Hg concentration as dry weight along with the mean water content [(0.043 µg Hg/g dry wt.) x (1.00 g wet wt – 0.795 g water) = 0.0088 µg/g wet wt.]. The point of this exercise is that the total Hg concentrations in fish from this study were very low and do not pose a human health risk.

**Table 4-2. Monthly fish consumption as meals per month based on the criteria set by USEPA (2001) and listed above in the text with supporting calculations plus the number of fish meals that could be consumed for fish collected for this study.**

Recommended Fish meals/month	Fish Hg content (µg/g, wet wt.)
16	>0.03 to 0.06
12	>0.06 to 0.08
8	>0.08 to 0.12
4	>0.12 to 0.24
3	>0.24 to 0.32
2	>0.32 to 0.48
1	>0.48 to 0.97
0.5	>0.97 to 1.9
None	>1.9
Projections based on Hg content of fish from this study in the Chukchi Sea	
106	0.0088 ± 0.0031

Concentrations of Ag in fish from this study also were low and relatively uniform (RSD for all samples was 31%, Tables 3-3 and 3-4, Figures 3-1a and 3-3a). No significant difference was found for mean Ag values for fish from the Burger area versus the Klondike area. For As, concentrations were relatively uniform (RSD = 21% for all samples) and no significant

difference in mean values was found between the two sites (Tables 3 and 4, Figures 1b and 3b). Data for Ag, As and other metals in fish from this study will be compared below with data for Arctic cod from the Beaufort Sea and other organisms (amphipods, clams and crabs) collected from the Chukchi Sea during 2008.

The greatest variability in metal concentrations (RSDs  $\geq 50\%$ ) was observed for Ba, Cd, Cr, Fe, and Pb. Some of the variability observed for concentrations of Fe and Mn in fish was due to visible sediment in some samples. The average concentration of Fe in sediments from the Chukchi Sea is about 60 times greater than found in the fish samples (Table 6) and thus a small amount of sediment can enhance the Fe value for fish tissue. For discussion purposes, the lowest Fe in fish value of 124  $\mu\text{g/g}$  will be assumed to be the tissue Fe value. The highest Fe value for the mixed fish samples was 1130  $\mu\text{g/g}$ . If we consider this highest Fe value for fish to result from mixing fish tissue Fe (124  $\mu\text{g/g}$ ) and sediment Fe (28,600  $\mu\text{g/g}$ ), then a measured Fe value of 1130  $\mu\text{g/g}$  could result from mixing 956 mg of tissue with 44 mg of sediment (i.e., the sample contains about 4.4% sediment by mass, dry weight) as shown in the example calculation below.

$$[(28,600 \mu\text{g/g}) (x)] + [(124 \mu\text{g/g})(1-x)] = 1130 \mu\text{g/g}$$

Where,  $x$  = the fraction of sediment in the composite fish sample

$(1-x)$  = the fraction of fish tissue in the composite fish sample

Rearranging,  $28,600x - 124x = 1130 + 124$

$$28,476 x = 1254$$

$$x = 0.044 \text{ or } 4.4\%$$

If this approach, based on Fe, is applied to all fish tissue samples from this study, the average sediment portion of the composite sample is  $2.1 \pm 1.1\%$  (range 0.44 to 4.4%).

Some trace metals, such as Ba and Cr, are present in low concentrations in fish tissue relative to sediment (Table 6). Therefore, when small amounts of sediment are incorporated into a tissue

sample, the resulting concentration of Ba or Cr may be increased by the Ba and Cr in the sediment. In this study, strong correlations for Ba versus Fe and Cr versus Fe resulted from sediment in the composite fish sample (Figure 5). The quantitative impact of sediment on the Ba and Cr values for the fish tissue in this sample is difficult to assess. However, previous data for Arctic cod from the Beaufort Sea show that the Ba and Cr values obtained for fish tissue in this study were about 2 times greater than concentrations obtained for sediment-free samples from the Beaufort Sea (Table 7). Some care needs to be exercised in using the Ba, Cr and Fe values from his study because these metals will only be slightly released from sediment during passage of the mixed fish sample through the gut of an organism (Trefry and Metz, 1984). More importantly, background concentrations of Ba and Cr in the fish tissue are very low.

**Table 4-3. Average concentrations of metals in fish tissue from this study, sediments from the Chukchi Sea and the ratio for metals in sediments/fish.**

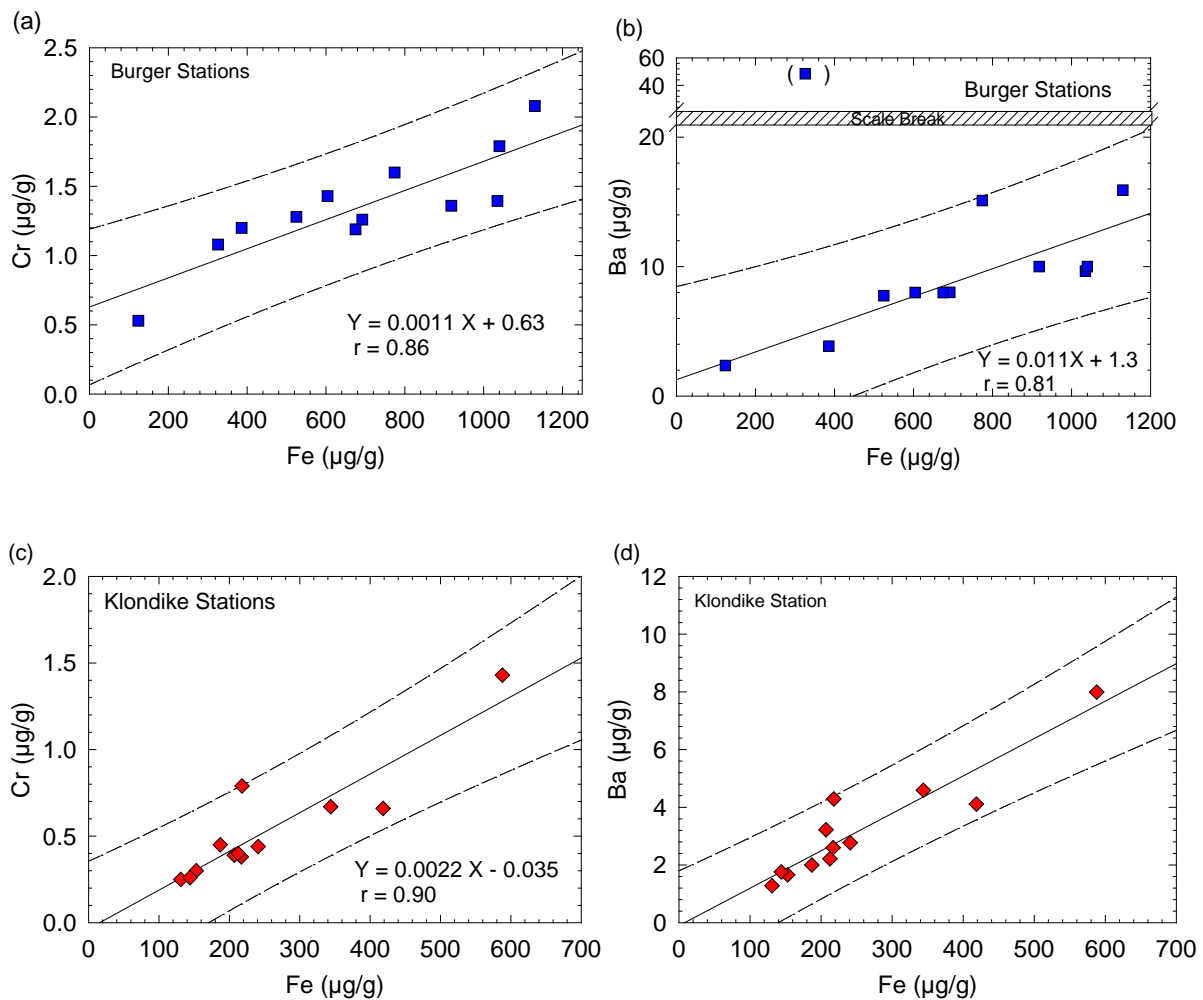
<b>Metal</b>	<b>Average concentration in fish from Chukchi Sea (this study) (µg/g, dry wt.)</b>	<b>Average concentration in sediment from Chukchi Sea* (µg/g, dry wt.)</b>	<b>(Sediment/Fish)</b>
Ag	0.11	0.12	1.1
As	9.4	13.5	1.4
Ba	7.7	853	<b>111</b>
Cd	0.48	0.18	0.4
Cr	0.94	75.0	<b>80</b>
Cu	4.2	13.6	3.2
Fe	470	28,600	<b>61</b>
Hg	0.043	0.034	0.8
Mn	9.8	298	<b>30</b>
Pb	0.94	12.3	13
Se	3.1	0.55	0.2
Zn	86	71.8	0.8

For Ba, a very high and unexplained maximum value of 48.4 µg/g was obtained for the sample from Burger Station 9, relative to the second highest value of 15.9 µg/g for Burger Station 1. Elevated Ba values were previously found in sediments from an old drill site in the Burger area (Neff et al., 2009b) and the slight enhancement of Ba could be from a minute amount of drilling mud barite in the sediment portion of the composite fish sample. Most of the barium in clean marine sediments worldwide is in the form of minute particles of nearly insoluble barite in the fine-grained fraction of the sediments (Neff, 2002).

Lead concentrations in the composite fish sample were all <1 µg/g (Tables 4 and 5 and Figures 2d and 4d). As much as 50% of this Pb could be linked to sediment incorporation in the sample based on the sediment/tissue ratio of 13 (Table 6) and the concentration of Pb in Arctic cod from the Beaufort Sea (Table 7). Average Pb values for the Klondike stations were significantly higher than found for the Burger stations. However, the Pb values for the fish samples from this study were very low.

Concentrations of Cd in the composite fish tissue samples from the Burger area were all  $<0.5$   $\mu\text{g/g}$  with an average of  $0.24 \pm 0.06$   $\mu\text{g/g}$  (RSD = 25%, Tables 3 and 4 and Figure 1d). In contrast Cd values for fish samples from the Klondike area were significantly higher at  $0.73 \pm 0.42$   $\mu\text{g/g}$  (Tables 3 and 4 and Figure 3d). The highest Cd value was found for Klondike Station 19 where Snailfish, a demersal species, were an important fraction of the total samples relative to other stations. Sediment Cd values were low and a few percent sediment incorporation would not have an impact on Cd values for the fish tissue. As a point of clarification, concentrations of metals such as Ag, As, Cd, Cu, Hg, Se and Zn in tissues are similar to or greater than values for sediments and thus a few percent sediment in the composite sample does not distort the metal value for the tissue sample (Table 6).





**Figure 4-1. Concentrations of Fe versus (a) Cr and (b) Ba for fish tissue from the Burger area and (c) Cr and (d) Ba for fish tissue from the Klondike area. Equations and lines are from linear regression analysis, r is the correlation coefficient and the dashed lines show the 95% prediction interval. Data point in parentheses on (b) was not included in the linear regression calculation.**

Concentrations of metals in fish tissue from this study were in reasonably close agreement with data for Arctic cod from the Beaufort Sea (Table 4-4). In particular, average concentrations of Ag, As, Cu, Se and Zn in Arctic cod from the Beaufort Sea were within 10% of the average value for the composite fish tissue from this study in the Chukchi Sea (Table 4-4). Arctic cod

were an important component of the Chukchi Sea composite samples from this study and this similarity in metal values reinforces that observation.

Concentrations of Ag, Ba, Cd, Cu and Mn in the composite fish sample from this study were lower than previously reported for amphipods, clams and crabs from a 2008 survey in the Chukchi Sea (Neff et al., 2009b). The remaining metals were present in the fish samples from this study at concentrations that were within 2% to a factor of two of concentrations for one or more of the biota samples from the Chukchi Sea.

**Table 4-4. Summary statistics for water content and trace metals (dry weight) in fish tissue samples from this study, Arctic cod from the Beaufort Sea (Neff et al., 2009a), and amphipods, clams and crabs from the Chukchi Sea (Neff et al., 2009b).**

Sample Identification	Statistic	Water Content (%)	Ag (µg/g)	As (µg/g)	Ba (µg/g)	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Fe (µg/g)	Hg (µg/g)	Mn (µg/g)	Pb (µg/g)	Se (µg/g)	Zn (µg/g)
Fish Tissue	Mean	79.5	0.11	9.4	7.7	0.5	0.94	4.2	470	0.045	10	0.9	3.1	86
	Std. Dev.	1.4	0.03	2.0	9.6	0.4	0.54	0.6	322	0.013	2	0.5	0.3	9
Arctic cod** Beaufort Sea	Mean	74.6	0.10	9.4	4.0	0.17	0.5	4.5	172	0.031	-	0.30	3.3	83
	Std. Dev.	6.7	0.03	5.3	2.3	0.11	0.4	2.2	165	0.018	-	0.19	1.0	22
Amphipods* (n = 8)	Mean	75.3	0.82	12.4	8.4	3.8	0.6	39	234	0.096	11	0.14	3.6	165
	Std. Dev.	1.4	0.20	2.8	1.86	0.7	0.3	7	45	0.018	2	0.03	0.9	27
Clams* (n = 20)	Mean	81.4	0.26	11.7	15	30	1.4	12	1470	0.056	23	0.8	7.8	83
	Std. Dev.	2.1	0.16	1.5	8	17	0.3	5	720	0.018	11	0.2	2.5	8
Crabs*	Mean	71.2	0.73	9.2	12	1.1	0.6	43	350	0.026	24	0.16	2.9	63
	Std. Dev.	1.5	0.33	1.3	1	0.3	0.1	16	99	0.006	8	0.04	0.6	4

\*from Neff et al. (2009b).

\*\* from Neff et al. (2009a).

## 5 Conclusions

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The fish tissue samples collected have very low concentrations of Total PAHs that are within the range of values reported from previous studies of other Alaskan coastal and continental shelf areas and are representative of background tissue concentrations. The Total PAH values are also low as compared to the USEPA (2000) fish advisory consumption limits for PAH and pose no human health risk if consumed. Since PAH, and other hydrocarbons, do not biomagnify in the marine food chains (Neff, 2002), trophic transfer is inefficient and hydrocarbon concentrations are unlikely to reach high concentrations in upper trophic level animals, such as birds, whales, seals, and polar bears.

Fish tissues samples collected from the Chukchi Sea for this study have very low concentrations of Ag, Cd, Hg and Pb. Copper Se and Zn are regulated by fish and the values for fish from this study are within 10% of concentrations reported for Arctic cod from the Beaufort Sea and are most likely represent background conditions. Concentrations of Ba and Cr were low, but enhanced by the presence of sediment in the composite fish samples. Even though the samples for this study were made up of mixed fish species, the data show that the fish tissue is essentially free of metal contamination and comparable with clean fish samples from the Beaufort Sea.

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## **Appendix A**

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### **Organics Data, Quality Control Results, and Histograms**



**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-KF001-FC-01	09-04-KF003-FC-01	09-04-KF005-FC-01	09-04-KF007-FC-01
Battelle ID	Q8723-P	Q8724-P	Q8725-P	Q8726-P
Sample Type	SA	SA	SA	SA
Collection Date	09/26/09	09/27/09	09/29/09	09/27/09
Extraction Date	03/03/10	03/03/10	03/03/10	03/03/10
Analysis Date	03/11/10	03/11/10	03/12/10	03/12/10
Analytical Instrument	MS	MS	MS	MS
% Moisture	82.18	82.35	81.14	82.87
% Lipid	3.29	3.37	3.39	3.03
Matrix	TISSUE	TISSUE	TISSUE	TISSUE
Sample Size	3.57	3.54	3.79	3.43
Size Unit-Basis	G_DRY	G_DRY	G_DRY	G_DRY
Units	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY
Naphthalene	2.17 BJ	2.32 BJ	2.29 BJ	2.2 BJ
C1-Naphthalenes	0.86 BJ	0.99 BJ	1.02 BJ	0.98 BJ
C2-Naphthalenes	1.67 J	1.53 J	1.39 J	1.49 J
C3-Naphthalenes	1.01 J	1.06 J	0.8 J	1.23 J
C4-Naphthalenes	U	U	U	U
Biphenyl	0.54 BJ	0.57 BJ	0.58 BJ	0.53 BJ
Acenaphthylene	0.31 J	0.34 J	0.37 J	0.2 J
Acenaphthene	U	U	U	0.06 J
Fluorene	0.57 J	0.52 J	0.48 J	0.45 J
C1-Fluorenes	1.79 J	1.06 J	0.71 J	3.85 J
C2-Fluorenes	3.32 J	3.38 J	1.44 J	1.43 J
C3-Fluorenes	U	U	U	U
Anthracene	U	U	U	U
Phenanthrene	0.76 BJ	1.12 BJ	0.68 BJ	0.78 BJ
C1-Phenanthrenes/Anthracenes	0.93 J	1.12 J	1.24 J	1.51 J
C2-Phenanthrenes/Anthracenes	1.64 J	1.69 J	1.53 J	1.7 J
C3-Phenanthrenes/Anthracenes	0.75 J	0.59 J	0.55 J	0.92 J
C4-Phenanthrenes/Anthracenes	U	U	U	U
Dibenzothiophene	U	U	U	U
C1-Dibenzothiophenes	U	U	U	U
C2-Dibenzothiophenes	U	U	U	U
C3-Dibenzothiophenes	U	U	U	U
Fluoranthene	0.35 BJ	0.42 BJ	0.31 BJ	0.41 BJ
Pyrene	0.61 BJ	0.55 BJ	0.53 BJ	0.7 BJ
C1-Fluoranthenes/Pyrenes	U	U	U	U
C2-Fluoranthenes/Pyrenes	U	U	U	U
C3-Fluoranthenes/Pyrenes	U	U	U	U
Benzo(a)anthracene	0.14 J	U	0.16 J	0.18 J
Chrysene	0.35 BJ	0.4 BJ	0.34 BJ	0.41 BJ
C1-Chrysenes	U	U	U	U
C2-Chrysenes	U	U	U	U
C3-Chrysenes	U	U	U	U
C4-Chrysenes	U	U	U	U
Benzo(b)fluoranthene	U	U	U	U
Benzo(k)fluoranthene	U	U	U	U
Benzo(e)pyrene	U	U	U	0.16 J
Benzo(a)pyrene	U	U	U	U
Perylene	U	U	U	U
Indeno(1,2,3-cd)pyrene	U	U	U	U
Dibenz(a,h)anthracene	U	U	U	U
Benzo(g,h,i)perylene	U	U	U	U
Total PAH	17.77 J	17.66 J	14.42 J	19.19 J

**Surrogate Recoveries (%)**

Naphthalene-d8	74	74	78	73
Acenaphthene-d10	80	84	84	80
Phenanthrene-d10	75	78	77	70
Benzo(a)pyrene-d12	48	40	56	72





**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-KF009-FC-01	09-04-KF011-FC-01	09-04-KF013-FC-01	09-04-KF015-FC-01
Battelle ID	Q8727-P	Q8728-P	Q8729-P	Q8730-P
Sample Type	SA	SA	SA	SA
Collection Date	09/28/09	09/26/09	09/28/09	09/29/09
Extraction Date	03/03/10	03/03/10	03/03/10	03/03/10
Analysis Date	03/12/10	03/12/10	03/12/10	03/12/10
Analytical Instrument	MS	MS	MS	MS
% Moisture	82.39	82.87	83.27	81.84
% Lipid	2.97	3.32	2.85	2.45
Matrix	TISSUE	TISSUE	TISSUE	TISSUE
Sample Size	3.56	3.44	3.37	3.65
Size Unit-Basis	G_DRY	G_DRY	G_DRY	G_DRY
Units	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY
Naphthalene	1.72 BJ	2.87 BJ	2.78 BJ	1.99 BJ
C1-Naphthalenes	0.9 BJ	1 BJ	1.06 BJ	1.05 BJ
C2-Naphthalenes	1.74 J	1.33 J	1.74 J	1.56 J
C3-Naphthalenes	1.23 J	1.22 J	1.14 J	1.18 J
C4-Naphthalenes	U	U	U	U
Biphenyl	0.47 BJ	0.67 BJ	0.5 BJ	0.45 BJ
Acenaphthylene	0.34 J	0.44 J	0.54 J	0.1 J
Acenaphthene	U	U	U	U
Fluorene	0.4 J	0.69 J	0.54 J	0.43 J
C1-Fluorenes	0.52 J	1.66 J	0.49 J	0.42 J
C2-Fluorenes	1.57 J	1.61 J	1.2 J	1.38 J
C3-Fluorenes	U	U	U	U
Anthracene	U	U	U	U
Phenanthrene	0.75 BJ	1.71 BJ	0.75 BJ	0.75 BJ
C1-Phenanthrenes/Anthracenes	1.17 J	1.64 J	1.12 J	1.34 J
C2-Phenanthrenes/Anthracenes	2.06 J	1.81 J	1.56 J	1.33 J
C3-Phenanthrenes/Anthracenes	0.81 J	0.88 J	0.59 J	0.29 J
C4-Phenanthrenes/Anthracenes	U	U	U	U
Dibenzothiophene	U	U	U	U
C1-Dibenzothiophenes	U	U	U	U
C2-Dibenzothiophenes	U	U	U	U
C3-Dibenzothiophenes	U	U	U	U
Fluoranthene	0.35 BJ	0.65 BJ	0.22 BJ	0.21 BJ
Pyrene	0.54 BJ	0.76 BJ	0.35 BJ	0.32 BJ
C1-Fluoranthenes/Pyrenes	U	U	U	U
C2-Fluoranthenes/Pyrenes	U	U	U	U
C3-Fluoranthenes/Pyrenes	U	U	U	U
Benzo(a)anthracene	0.14 J	0.22 J	0.17 J	0.12 J
Chrysene	0.36 BJ	0.42 BJ	0.3 BJ	0.28 BJ
C1-Chrysenes	U	U	U	U
C2-Chrysenes	U	U	U	U
C3-Chrysenes	U	U	U	U
C4-Chrysenes	U	U	U	U
Benzo(b)fluoranthene	U	U	U	U
Benzo(k)fluoranthene	U	U	U	U
Benzo(e)pyrene	U	U	0.17 J	0.3 J
Benzo(a)pyrene	U	U	U	U
Perylene	U	U	U	U
Indeno(1,2,3-cd)pyrene	U	U	U	U
Dibenz(a,h)anthracene	U	U	U	U
Benzo(g,h,i)perylene	U	U	U	U
Total PAH	15.07 J	19.58 J	15.22 J	13.5 J

**Surrogate Recoveries (%)**

Naphthalene-d8	76	72	74	71
Acenaphthene-d10	81	75	78	80
Phenanthrene-d10	74	68	75	76
Benzo(a)pyrene-d12	68	63	74	69



**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-KF017-FC-01	09-04-KF019-FC-01	09-04-KF023-FC-01	09-04-KF025-FC-01
Battelle ID	Q8731-P	Q8732-P	Q8733-P	Q8734-P
Sample Type	SA	SA	SA	SA
Collection Date	09/26/09	09/28/09	09/27/09	09/29/09
Extraction Date	03/03/10	03/03/10	03/03/10	03/03/10
Analysis Date	03/12/10	03/12/10	03/12/10	03/12/10
Analytical Instrument	MS	MS	MS	MS
% Moisture	81.22	84.89	82.39	80.94
% Lipid	3.83	2.15	3.39	3.13
Matrix	TISSUE	TISSUE	TISSUE	TISSUE
Sample Size	3.79	3.03	3.57	3.86
Size Unit-Basis	G_DRY	G_DRY	G_DRY	G_DRY
Units	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY
Naphthalene	2.61 BJ	2.43 BJ	3 BJ	1.93 BJ
C1-Naphthalenes	1.11 BJ	1.2 BJ	1.65 BJ	1.33 BJ
C2-Naphthalenes	1.65 J	1.99 J	2.22 J	2.08 J
C3-Naphthalenes	1.3 J	1.07 J	1.49 J	1.37 J
C4-Naphthalenes	U	U	U	U
Biphenyl	0.6 BJ	0.54 BJ	0.61 BJ	0.56 BJ
Acenaphthylene	0.52 J	0.26 J	1.32 J	4.6
Acenaphthene	U	U	0.08 J	U
Fluorene	0.67 J	0.36 J	0.55 J	0.43 J
C1-Fluorenes	3.58 J	0.55 J	0.82 J	0.47 J
C2-Fluorenes	U	1.06 J	1.07 J	1.05 J
C3-Fluorenes	U	U	U	U
Anthracene	U	U	U	U
Phenanthrene	0.87 BJ	0.74 BJ	1.16 BJ	0.97 BJ
C1-Phenanthrenes/Anthracenes	1.11 J	1.04 J	1.64 J	1.56 J
C2-Phenanthrenes/Anthracenes	1.41 J	1.4 J	1.95 J	1.59 J
C3-Phenanthrenes/Anthracenes	0.58 J	0.52 J	1.08 J	0.7 J
C4-Phenanthrenes/Anthracenes	U	U	U	U
Dibenzothiophene	U	U	U	U
C1-Dibenzothiophenes	U	U	U	U
C2-Dibenzothiophenes	U	U	U	U
C3-Dibenzothiophenes	U	U	U	U
Fluoranthene	0.25 BJ	0.21 BJ	0.45 BJ	0.37 BJ
Pyrene	0.37 BJ	0.35 BJ	0.86 BJ	0.56 BJ
C1-Fluoranthenes/Pyrenes	U	U	U	0.6 J
C2-Fluoranthenes/Pyrenes	U	U	U	0.63 J
C3-Fluoranthenes/Pyrenes	U	U	U	U
Benzo(a)anthracene	0.12 J	0.17 J	0.2 J	0.13 J
Chrysene	0.25 BJ	0.29 BJ	0.42 BJ	0.38 BJ
C1-Chrysenes	U	U	U	U
C2-Chrysenes	U	U	U	U
C3-Chrysenes	U	U	U	U
C4-Chrysenes	U	U	U	U
Benzo(b)fluoranthene	U	U	U	U
Benzo(k)fluoranthene	U	U	U	U
Benzo(e)pyrene	0.16 J	0.19 J	0.28 J	0.25 J
Benzo(a)pyrene	U	U	U	U
Perylene	U	U	U	U
Indeno(1,2,3-cd)pyrene	U	U	U	U
Dibenz(a,h)anthracene	U	U	U	U
Benzo(g,h,i)perylene	U	U	U	U
Total PAH	17.16 J	14.37 J	20.85 J	21.56 J

**Surrogate Recoveries (%)**

Naphthalene-d8	79	74	73	81
Acenaphthene-d10	81	81	78	82
Phenanthrene-d10	76	77	78	82
Benzo(a)pyrene-d12	67	78	65	78



**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-KF009-FC-02	09-04-BF001-FC-01	09-04-BF003-FC-01	09-04-BF007-FC-01
Battelle ID	Q8741-P	Q8744-P	Q8745-P	Q8746-P
Sample Type	SA	SA	SA	SA
Collection Date	09/28/09	10/01/09	10/10/09	10/06/09
Extraction Date	03/03/10	03/03/10	03/03/10	03/03/10
Analysis Date	03/13/10	03/13/10	03/13/10	03/13/10
Analytical Instrument	MS	MS	MS	MS
% Moisture	83.46	80.79	81.94	81.97
% Lipid	2.34	2.52	2.94	2.98
Matrix	TISSUE	TISSUE	TISSUE	TISSUE
Sample Size	3.34	3.86	3.64	3.60
Size Unit-Basis	G_DRY	G_DRY	G_DRY	G_DRY
Units	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY
Naphthalene	1.92 BJ	2.78 BJ	3.01 BJ	2.3 BJ
C1-Naphthalenes	1.15 BJ	1.77 BJ	1.79 BJ	1.6 BJ
C2-Naphthalenes	2.1 J	2.45 J	2.25 J	2.68 J
C3-Naphthalenes	1.26 J	1.24 J	1.51 J	1.53 J
C4-Naphthalenes	U	U	U	U
Biphenyl	0.49 BJ	0.51 BJ	0.48 BJ	0.47 BJ
Acenaphthylene	0.43 J	0.52 J	0.68 J	0.66 J
Acenaphthene	U	U	U	U
Fluorene	0.44 J	0.55 J	0.46 J	0.41 J
C1-Fluorenes	U	0.58 J	0.61 J	0.51 J
C2-Fluorenes	U	0.99 J	1.3 J	1.09 J
C3-Fluorenes	U	U	U	U
Anthracene	U	U	U	U
Phenanthrene	0.87 BJ	1.26 BJ	1.11 BJ	1.12 BJ
C1-Phenanthrenes/Anthracenes	1.19 J	1.75 J	1.63 J	1.6 J
C2-Phenanthrenes/Anthracenes	2.04 J	1.89 J	2.18 J	2.08 J
C3-Phenanthrenes/Anthracenes	0.93 J	0.85 J	1.44 J	0.97 J
C4-Phenanthrenes/Anthracenes	U	U	U	U
Dibenzothiophene	U	U	U	U
C1-Dibenzothiophenes	U	U	U	U
C2-Dibenzothiophenes	U	U	U	U
C3-Dibenzothiophenes	U	U	U	U
Fluoranthene	0.25 BJ	0.45 BJ	0.47 BJ	0.49 BJ
Pyrene	0.5 BJ	0.85 BJ	0.84 BJ	0.74 BJ
C1-Fluoranthenes/Pyrenes	U	0.71 J	1.35 J	0.87 J
C2-Fluoranthenes/Pyrenes	U	U	1.55 J	0.68 J
C3-Fluoranthenes/Pyrenes	U	U	U	U
Benzo(a)anthracene	U	0.19 J	0.3 J	0.29 J
Chrysene	0.47 BJ	0.6 BJ	0.83 J	0.61 J
C1-Chrysenes	U	U	0.86 J	U
C2-Chrysenes	U	U	U	U
C3-Chrysenes	U	U	U	U
C4-Chrysenes	U	U	U	U
Benzo(b)fluoranthene	U	U	0.79 J	U
Benzo(k)fluoranthene	U	U	0.24 J	U
Benzo(e)pyrene	0.28 J	0.41 J	0.63 J	0.4 J
Benzo(a)pyrene	U	0.13 J	0.33 J	U
Perylene	U	0.83 J	1.36 J	1.06 J
Indeno(1,2,3-cd)pyrene	U	U	U	U
Dibenz(a,h)anthracene	U	U	U	U
Benzo(g,h,i)perylene	U	U	U	U
Total PAH	14.32 J	21.31 J	28 J	22.16 J

**Surrogate Recoveries (%)**

Naphthalene-d8	71	74	75	73
Acenaphthene-d10	76	78	81	80
Phenanthrene-d10	71	75	80	80
Benzo(a)pyrene-d12	73	75	68	74



**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-BF009-FC-01	09-04-BF011-FC-01	09-04-BF013-FC-01	09-04-BF015-FC-01
Battelle ID	Q8747-P	Q8748-P	Q8749-P	Q8750-P
Sample Type	SA	SA	SA	SA
Collection Date	10/09/09	10/01/09	10/06/09	10/07/09
Extraction Date	03/03/10	03/03/10	03/03/10	03/03/10
Analysis Date	03/13/10	03/13/10	03/13/10	03/13/10
Analytical Instrument	MS	MS	MS	MS
% Moisture	82.62	82.4	83.5	79.34
% Lipid	2.43	2.38	2.53	4.17
Matrix	TISSUE	TISSUE	TISSUE	TISSUE
Sample Size	3.48	3.53	3.32	4.17
Size Unit-Basis	G_DRY	G_DRY	G_DRY	G_DRY
Units	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY
Naphthalene	2.97 BJ	2.63 BJ	2.73 BJ	2.56 BJ
C1-Naphthalenes	1.6 BJ	1.56 BJ	1.89 BJ	1.54 BJ
C2-Naphthalenes	1.71 J	2.27 J	2.87 J	2.09 J
C3-Naphthalenes	1.16 J	1.41 J	1.36 J	1.17 J
C4-Naphthalenes	U	U	U	U
Biphenyl	0.4 BJ	0.42 BJ	0.58 BJ	0.55 BJ
Acenaphthylene	0.32 J	0.35 J	U	0.32 J
Acenaphthene	U	0.06 J	U	U
Fluorene	0.52 J	0.34 J	0.42 J	0.39 J
C1-Fluorenes	0.47 J	0.55 J	0.81 J	0.59 J
C2-Fluorenes	1.08 J	1.07 J	U	0.81 J
C3-Fluorenes	U	U	U	U
Anthracene	U	U	U	U
Phenanthrene	1.02 BJ	1.01 BJ	1.19 BJ	0.99 BJ
C1-Phenanthrenes/Anthracenes	1.47 J	1.44 J	1.86 J	1.6 J
C2-Phenanthrenes/Anthracenes	1.39 J	1.79 J	2.15 J	1.39 J
C3-Phenanthrenes/Anthracenes	0.95 J	0.98 J	1.18 J	0.84 J
C4-Phenanthrenes/Anthracenes	U	U	U	U
Dibenzothiophene	U	U	U	U
C1-Dibenzothiophenes	U	U	U	U
C2-Dibenzothiophenes	U	U	U	U
C3-Dibenzothiophenes	U	U	U	U
Fluoranthene	0.51 BJ	0.52 BJ	0.58 BJ	0.44 BJ
Pyrene	0.95 BJ	0.87 BJ	0.96 BJ	0.76 BJ
C1-Fluoranthenes/Pyrenes	0.62 J	0.77 J	0.74 J	0.69 J
C2-Fluoranthenes/Pyrenes	U	0.74 J	0.77 J	0.61 J
C3-Fluoranthenes/Pyrenes	U	U	U	U
Benzo(a)anthracene	0.2 J	0.28 J	0.24 J	0.37 J
Chrysene	0.41 BJ	0.63 J	0.58 BJ	0.67 J
C1-Chrysenes	U	U	U	0.36 J
C2-Chrysenes	U	U	U	U
C3-Chrysenes	U	U	U	U
C4-Chrysenes	U	U	U	U
Benzo(b)fluoranthene	U	U	U	U
Benzo(k)fluoranthene	U	U	U	U
Benzo(e)pyrene	0.25 J	0.36 J	0.51 J	0.44 J
Benzo(a)pyrene	U	0.16 J	0.14 J	U
Perylene	U	0.53 J	1.08 J	U
Indeno(1,2,3-cd)pyrene	U	U	U	U
Dibenz(a,h)anthracene	U	U	U	U
Benzo(g,h,i)perylene	U	U	U	U
Total PAH	18 J	20.74 J	22.64 J	19.18 J

**Surrogate Recoveries (%)**

Naphthalene-d8	72	77	85	77
Acenaphthene-d10	80	82	93	84
Phenanthrene-d10	81	77	92	83
Benzo(a)pyrene-d12	63	81	70	63



**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-BF017-FC-01	09-04-BF019-FC-01	09-04-BF021-FC-01	09-04-BF023-FC-01
Battelle ID	Q8751-P	Q8752-P	Q8753-P	Q8754-P
Sample Type	SA	SA	SA	SA
Collection Date	10/06/09	10/07/09	10/01/09	10/06/09
Extraction Date	03/03/10	03/03/10	03/03/10	03/03/10
Analysis Date	03/13/10	03/13/10	03/13/10	03/13/10
Analytical Instrument	MS	MS	MS	MS
% Moisture	81.69	80.77	81.7	82.76
% Lipid	3.59	3.52	3.48	2.84
Matrix	TISSUE	TISSUE	TISSUE	TISSUE
Sample Size	3.69	3.86	3.66	3.48
Size Unit-Basis	G_DRY	G_DRY	G_DRY	G_DRY
Units	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY	UG/KG_DRY
Naphthalene	2.83 BJ	2.39 BJ	2.77 BJ	2.6 BJ
C1-Naphthalenes	1.79 BJ	1.66 BJ	1.59 BJ	1.32 BJ
C2-Naphthalenes	2.48 J	2.56 J	2.12 J	2.17 J
C3-Naphthalenes	1.46 J	1.14 J	1.28 J	0.89 J
C4-Naphthalenes	U	U	U	U
Biphenyl	0.57 BJ	0.56 BJ	0.5 BJ	0.52 BJ
Acenaphthylene	0.81 J	0.54 J	0.54 J	1.58 J
Acenaphthene	U	0.08 J	U	U
Fluorene	0.49 J	0.45 J	0.52 J	0.35 J
C1-Fluorenes	0.76 J	0.56 J	0.54 J	0.46 J
C2-Fluorenes	1.06 J	0.68 J	0.82 J	1.2 J
C3-Fluorenes	U	U	U	U
Anthracene	U	U	U	U
Phenanthrene	1.14 BJ	0.94 BJ	1.03 BJ	0.89 BJ
C1-Phenanthrenes/Anthracenes	1.46 J	1.3 J	1.23 J	1.19 J
C2-Phenanthrenes/Anthracenes	1.81 J	1.46 J	1.55 J	1.37 J
C3-Phenanthrenes/Anthracenes	1.43 J	0.64 J	0.98 J	0.7 J
C4-Phenanthrenes/Anthracenes	U	U	U	U
Dibenzothiophene	U	U	U	U
C1-Dibenzothiophenes	U	U	U	U
C2-Dibenzothiophenes	U	U	U	U
C3-Dibenzothiophenes	U	U	U	U
Fluoranthene	0.5 BJ	0.32 BJ	0.38 BJ	0.32 BJ
Pyrene	0.87 BJ	0.5 BJ	0.58 BJ	0.61 BJ
C1-Fluoranthenes/Pyrenes	0.96 J	U	0.74 J	U
C2-Fluoranthenes/Pyrenes	U	U	0.83 J	U
C3-Fluoranthenes/Pyrenes	U	U	U	U
Benzo(a)anthracene	0.28 J	0.17 J	0.24 J	0.24 J
Chrysene	0.62 J	0.49 BJ	0.61 J	0.48 BJ
C1-Chrysenes	0.51 J	0.5 J	0.52 J	U
C2-Chrysenes	U	U	U	U
C3-Chrysenes	U	U	U	U
C4-Chrysenes	U	U	U	U
Benzo(b)fluoranthene	U	U	U	U
Benzo(k)fluoranthene	U	U	U	U
Benzo(e)pyrene	0.48 J	0.42 J	0.42 J	0.28 J
Benzo(a)pyrene	U	U	U	0.17 J
Perylene	U	U	U	U
Indeno(1,2,3-cd)pyrene	U	U	U	U
Dibenz(a,h)anthracene	U	U	U	U
Benzo(g,h,i)perylene	U	U	U	U
Total PAH	22.31 J	17.36 J	19.79 J	17.34 J

**Surrogate Recoveries (%)**

Naphthalene-d8	76	82	77	82
Acenaphthene-d10	79	87	82	84
Phenanthrene-d10	80	87	83	85
Benzo(a)pyrene-d12	76	79	72	78



**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-BF025-FC-01
Battelle ID	Q8755-P
Sample Type	SA
Collection Date	10/07/09
Extraction Date	03/03/10
Analysis Date	03/14/10
Analytical Instrument	MS
% Moisture	83.99
% Lipid	3.03
Matrix	TISSUE
Sample Size	3.20
Size Unit-Basis	G_DRY
Units	UG/KG_DRY

Naphthalene	3.55 BJ
C1-Naphthalenes	1.19 BJ
C2-Naphthalenes	1.42 J
C3-Naphthalenes	0.66 J
C4-Naphthalenes	U
Biphenyl	0.42 BJ
Acenaphthylene	0.75 J
Acenaphthene	0.09 J
Fluorene	0.64 J
C1-Fluorenes	0.52 J
C2-Fluorenes	1.08 J
C3-Fluorenes	U
Anthracene	U
Phenanthrene	0.67 BJ
C1-Phenanthrenes/Anthracenes	0.83 J
C2-Phenanthrenes/Anthracenes	1.29 J
C3-Phenanthrenes/Anthracenes	0.62 J
C4-Phenanthrenes/Anthracenes	U
Dibenzothiophene	U
C1-Dibenzothiophenes	U
C2-Dibenzothiophenes	U
C3-Dibenzothiophenes	U
Fluoranthene	0.35 BJ
Pyrene	0.68 BJ
C1-Fluoranthenes/Pyrenes	U
C2-Fluoranthenes/Pyrenes	U
C3-Fluoranthenes/Pyrenes	U
Benzo(a)anthracene	0.24 J
Chrysene	0.42 BJ
C1-Chrysenes	U
C2-Chrysenes	U
C3-Chrysenes	U
C4-Chrysenes	U
Benzo(b)fluoranthene	U
Benzo(k)fluoranthene	U
Benzo(e)pyrene	0.22 J
Benzo(a)pyrene	U
Perylene	U
Indeno(1,2,3-cd)pyrene	U
Dibenz(a,h)anthracene	U
Benzo(g,h,i)perylene	U
Total PAH	15.64 J

**Surrogate Recoveries (%)**

Naphthalene-d8	77
Acenaphthene-d10	82
Phenanthrene-d10	83
Benzo(a)pyrene-d12	81

# Battelle

The Business of Innovation

**Project Client:** Exponent, Inc.

**Project Name:** Chukchi Sea Contaminants Program

**Project Number:** N106753-0001

Client ID	Procedural Blank
Battelle ID	BN729PB-P
Sample Type	PB
Collection Date	03/03/10
Extraction Date	03/03/10
Analysis Date	03/11/10
Analytical Instrument	MS
% Moisture	82.21
% Lipid	NA
Matrix	TISSUE
Sample Size	3.64
Size Unit-Basis	G_DRY
Units	UG/KG_DRY

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Naphthalene	2.25 J
C1-Naphthalenes	1.07 J
C2-Naphthalenes	U
C3-Naphthalenes	U
C4-Naphthalenes	U
Biphenyl	0.17 J
Acenaphthylene	U
Acenaphthene	U
Fluorene	U
C1-Fluorenes	U
C2-Fluorenes	U
C3-Fluorenes	U
Anthracene	U
Phenanthrene	0.43 J
C1-Phenanthrenes/Anthracenes	U
C2-Phenanthrenes/Anthracenes	U
C3-Phenanthrenes/Anthracenes	U
C4-Phenanthrenes/Anthracenes	U
Dibenzothiophene	U
C1-Dibenzothiophenes	U
C2-Dibenzothiophenes	U
C3-Dibenzothiophenes	U
Fluoranthene	0.17 J
Pyrene	0.41 J
C1-Fluoranthenes/Pyrenes	U
C2-Fluoranthenes/Pyrenes	U
C3-Fluoranthenes/Pyrenes	U
Benzo(a)anthracene	U
Chrysene	0.12 J
C1-Chrysenes	U
C2-Chrysenes	U
C3-Chrysenes	U
C4-Chrysenes	U
Benzo(b)fluoranthene	U
Benzo(k)fluoranthene	U
Benzo(e)pyrene	U
Benzo(a)pyrene	U
Perylene	U
Indeno(1,2,3-cd)pyrene	U
Dibenz(a,h)anthracene	U
Benzo(g,h,i)perylene	U
Total PAH	4.62 J

## Surrogate Recoveries (%)

Naphthalene-d8	53
Acenaphthene-d10	75
Phenanthrene-d10	81
Benzo(a)pyrene-d12	94

# Battelle

The Business of Innovation

**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	100107-01: Tilapia			
Battelle ID	BN730LCS-P			
Sample Type	LCS			
Collection Date	03/03/10			
Extraction Date	03/03/10			
Analysis Date	03/11/10			
Analytical Instrument	MS			
% Moisture	78.5			
% Lipid	2.96			
Matrix	TISSUE			
Sample Size	4.32			
Size Unit-Basis	G_DRY			
Units	UG/KG_DRY	Target	% Recovery	Qualifier
Naphthalene	126.45	115.94	109	
C1-Naphthalenes	U			
C2-Naphthalenes	U			
C3-Naphthalenes	U			
C4-Naphthalenes	U			
Biphenyl	128.85	115.86	111	
Acenaphthylene	133.2	116.01	115	
Acenaphthene	123.19	116.04	106	
Fluorene	124.9	115.94	108	
C1-Fluorenes	U			
C2-Fluorenes	U			
C3-Fluorenes	U			
Anthracene	131.68	116.02	113	
Phenanthrene	124.65	115.98	107	
C1-Phenanthrenes/Anthracenes	U			
C2-Phenanthrenes/Anthracenes	U			
C3-Phenanthrenes/Anthracenes	U			
C4-Phenanthrenes/Anthracenes	U			
Dibenzothiophene	130.55	116.04	113	
C1-Dibenzothiophenes	U			
C2-Dibenzothiophenes	U			
C3-Dibenzothiophenes	U			
Fluoranthene	122.2	115.97	105	
Pyrene	126.34	115.90	109	
C1-Fluoranthenes/Pyrenes	U			
C2-Fluoranthenes/Pyrenes	U			
C3-Fluoranthenes/Pyrenes	U			
Benzo(a)anthracene	135.21	116.02	117	
Chrysene	129.17	116.09	111	
C1-Chrysenes	U			
C2-Chrysenes	U			
C3-Chrysenes	U			
C4-Chrysenes	U			
Benzo(b)fluoranthene	105.93	115.90	91	
Benzo(k)fluoranthene	106.28	115.93	92	
Benzo(e)pyrene	116.27	116.04	100	
Benzo(a)pyrene	119.35	116.08	103	
Perylene	108.87	116.02	94	
Indeno(1,2,3-cd)pyrene	138.43	116.05	119	
Dibenz(a,h)anthracene	139.24	116.02	120	
Benzo(g,h,i)perylene	124.16	115.90	107	
Total PAH	2494.92			

### Surrogate Recoveries (%)

Naphthalene-d8	77
Acenaphthene-d10	78
Phenanthrene-d10	80
Benzo(a)pyrene-d12	84

Surrogate Corrected

Analyzed by Lizotte Jr, Robert  
5/11/2010

LCS: T10-0026MS-Master\_157-Final



# Battelle

The Business of Innovation

Project Client: Exponent, Inc.

Project Name: Chukchi Sea Contaminants Program

Project Number: N106753-0001

Client ID	090714-01: Nist 2977				
Battelle ID	BN731SRM-P				
Sample Type	SRM				
Collection Date	03/03/10				
Extraction Date	03/03/10				
Analysis Date	03/11/10				
Analytical Instrument	MS				
% Moisture	NA				
% Lipid	8.66				
Matrix	TISSUE				
Sample Size	1.40				
Size Unit-Basis	G_DRY	Certified	Actual		
Units	UG/KG_DRY	Value	%Difference	Qualifier	
Naphthalene	8.77 J				
C1-Naphthalenes	9.53 J				
C2-Naphthalenes	51.27				
C3-Naphthalenes	155.72				
C4-Naphthalenes	225.13				
Biphenyl	2.21 J				
Acenaphthylene	2.78 J				
Acenaphthene	2.52 J				
Fluorene	7.87 J	10.3	23.6		
C1-Fluorenes	35.45				
C2-Fluorenes	139.44				
C3-Fluorenes	221.82				
Anthracene	4.88 J				
Phenanthrene	38.54	36.2	6.5		
C1-Phenanthrenes/Anthracenes	139.75				
C2-Phenanthrenes/Anthracenes	394.32				
C3-Phenanthrenes/Anthracenes	419.43				
C4-Phenanthrenes/Anthracenes	183.52				
Dibenzothiophene	26.21				
C1-Dibenzothiophenes	211.16				
C2-Dibenzothiophenes	594.29				
C3-Dibenzothiophenes	688.54				
Fluoranthene	36.56	38.9	6		
Pyrene	71.93	77.4	7.1		
C1-Fluoranthenes/Pyrenes	69.67				
C2-Fluoranthenes/Pyrenes	92.53				
C3-Fluoranthenes/Pyrenes	75				
Benzo(a)anthracene	22.29	20.19	10.4		
Chrysene	95.94				
C1-Chrysenes	85.14				
C2-Chrysenes	67				
C3-Chrysenes	35.68				
C4-Chrysenes	15.99				
Benzo(b)fluoranthene	14.8	11.1	33.3		
Benzo(k)fluoranthene	9.88 J				
Benzo(e)pyrene	15.82	13.29	19		
Benzo(a)pyrene	3.33 J	5.3	37.2	N	
Perylene	1.97 J	3.69	46.6	N	
Indeno(1,2,3-cd)pyrene	3.66 J	4.76	23.1		
Dibenz(a,h)anthracene	1.29 J				
Benzo(g,h,i)perylene	4.92 J	9.45	47.9	N	
Total PAH	4286.55				

## Surrogate Recoveries (%)

Naphthalene-d8	78
Acenaphthene-d10	81
Phenanthrene-d10	81
Benzo(a)pyrene-d12	76

Analyzed by Lizotte Jr, Robert  
5/11/2010

Surrogate Corrected

SRM: T10-0026MS-Master\_157-Final



The Business of Innovation

Project Client: Exponent, Inc.  
Project Name: Chukchi Sea Contaminants Program  
Project Number: N106753-0001

GV73: North Slope  
Crude

Client ID				
Battelle ID	BN732NSC-P			
Sample Type	NSC			
Collection Date	03/09/10			
Extraction Date	03/09/10			
Analysis Date	03/11/10			
Analytical Instrument	MS			
% Moisture	NA			
% Lipid	NA			
Matrix	OIL			
Sample Size	5.05			
Size Unit-Basis	MG_OIL			
Units	MG/KG_OIL	Target	% Difference	Qualifier

Naphthalene	705.09	740.29	4.8	
C1-Naphthalenes	1413.85	1516.04	6.7	
C2-Naphthalenes	1898.1	2000.10	5.1	
C3-Naphthalenes	1430.13	1526.96	6.3	
C4-Naphthalenes	865	898.03	3.7	
Biphenyl	216.81	220.82	1.8	
Acenaphthylene	7.28			
Acenaphthene	15.91	14.50	9.7	
Fluorene	99.93	92.51	8.0	
C1-Fluorenes	262.01	227.01	15.4	
C2-Fluorenes	383.74	367.09	4.5	
C3-Fluorenes	346.62	326.32	6.2	
Anthracene	U			
Phenanthrene	291.15	249.49	16.7	
C1-Phenanthrenes/Anthracenes	570.7	549.17	3.9	
C2-Phenanthrenes/Anthracenes	688.95	642.72	7.2	
C3-Phenanthrenes/Anthracenes	506.16	446.11	13.5	
C4-Phenanthrenes/Anthracenes	174.47	180.02	3.1	
Dibenzothiophene	245.22	210.35	16.6	
C1-Dibenzothiophenes	504.01	409.03	23.2	
C2-Dibenzothiophenes	646.23	551.46	17.2	
C3-Dibenzothiophenes	560.98	471.36	19.0	
Fluoranthene	4.06 J			
Pyrene	16.76	12.99	29.0	
C1-Fluoranthenes/Pyrenes	74.84	70.92	5.5	
C2-Fluoranthenes/Pyrenes	138.2	117.89	17.2	
C3-Fluoranthenes/Pyrenes	141.95	137.25	3.4	
Benzo(a)anthracene	6.28 J			
Chrysene	58.53	47.18	24.1	
C1-Chrysenes	100.56	78.82	27.6	
C2-Chrysenes	117.27	102.67	14.2	
C3-Chrysenes	86.08	85.36	0.8	
C4-Chrysenes	67.24	61.99	8.5	
Benzo(b)fluoranthene	6.82 J	6.08	12.2	
Benzo(k)fluoranthene	U			
Benzo(e)pyrene	10.76	12.88	16.5	
Benzo(a)pyrene	2.14 J			
Perylene	U			
Indeno(1,2,3-cd)pyrene	U			
Dibenz(a,h)anthracene	1.01 J			
Benzo(g,h,i)perylene	2.73 J	3.44	20.6	
Total PAH	12667.57			

Surrogate Recoveries (%)

Naphthalene-d8	96
Acenaphthene-d10	98
Phenanthrene-d10	94

Analyzed by Lizotte Jr, Robert  
5/11/2010

Surrogate Corrected

NSC: T10-0026MS-Master\_157-Final

# Battelle

The Business of Innovation

**Project Client:** Exponent, Inc.

**Project Name:** Chukchi Sea Contaminants Program

**Project Number:** N106753-0001

Client ID	GV73: North Slope			
	Crude			
Battelle ID	BN732NSC-P			
Sample Type	NSC			
Collection Date	03/09/10			
Extraction Date	03/09/10			
Analysis Date	03/11/10			
Analytical Instrument	MS			
% Moisture	NA			
% Lipid	NA			
Matrix	OIL			
Sample Size	5.05			
Size Unit-Basis	MG_OIL			
Units	MG/KG_OIL	Target	% Difference	Qualifier
<hr/>				
Benzo(a)pyrene-d12	112			

# Battelle

The Business of Innovation

**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-KF001-FC-01	09-04-KF001-FC-01		
Battelle ID	Q8723-P	Q8723DUP-P		
Sample Type	SA	QADU		
Collection Date	09/26/09	09/26/09		
Extraction Date	03/03/10	03/03/10		
Analysis Date	03/11/10	03/11/10		
Analytical Instrument	MS	MS		
% Moisture	82.18	82.13		
% Lipid	3.29	3.48		
Matrix	TISSUE	TISSUE		
Sample Size	3.57	3.60		
Size Unit-Basis	G_DRY	G_DRY		
Units	UG/KG_DRY	UG/KG_DRY	RPD	Qualifier
Naphthalene	2.17 J	2.76 J	NA	
C1-Naphthalenes	0.86 J	1.06 J	NA	
C2-Naphthalenes	1.67 J	1.77 J	NA	
C3-Naphthalenes	1.01 J	1.08 J	NA	
C4-Naphthalenes	U	U	NA	
Biphenyl	0.54 J	0.63 J	NA	
Acenaphthylene	0.31 J	0.31 J	NA	
Acenaphthene	U	U	NA	
Fluorene	0.57 J	0.81 J	NA	
C1-Fluorenes	1.79 J	1.89 J	NA	
C2-Fluorenes	3.32 J	3.46 J	NA	
C3-Fluorenes	U	U	NA	
Anthracene	U	U	NA	
Phenanthrene	0.76 J	0.73 J	NA	
C1-Phenanthrenes/Anthracenes	0.93 J	1.08 J	NA	
C2-Phenanthrenes/Anthracenes	1.64 J	1.62 J	NA	
C3-Phenanthrenes/Anthracenes	0.75 J	1.18 J	NA	
C4-Phenanthrenes/Anthracenes	U	U	NA	
Dibenzothiophene	U	U	NA	
C1-Dibenzothiophenes	U	U	NA	
C2-Dibenzothiophenes	U	U	NA	
C3-Dibenzothiophenes	U	U	NA	
Fluoranthene	0.35 J	0.45 J	NA	
Pyrene	0.61 J	0.77 J	NA	
C1-Fluoranthenes/Pyrenes	U	U	NA	
C2-Fluoranthenes/Pyrenes	U	U	NA	
C3-Fluoranthenes/Pyrenes	U	U	NA	
Benzo(a)anthracene	0.14 J	0.14 J	NA	
Chrysene	0.35 J	0.37 J	NA	
C1-Chrysenes	U	U	NA	
C2-Chrysenes	U	U	NA	
C3-Chrysenes	U	U	NA	
C4-Chrysenes	U	U	NA	
Benzo(b)fluoranthene	U	U	NA	
Benzo(k)fluoranthene	U	U	NA	
Benzo(e)pyrene	U	U	NA	
Benzo(a)pyrene	U	U	NA	
Perylene	U	U	NA	
Indeno(1,2,3-cd)pyrene	U	U	NA	
Dibenz(a,h)anthracene	U	U	NA	
Benzo(g,h,i)perylene	U	U	NA	
Total PAH	17.77 J	20.11 J	NA	

### Surrogate Recoveries (%)

Naphthalene-d8	74	85
Acenaphthene-d10	80	78
Phenanthrene-d10	75	72
Benzo(a)pyrene-d12	48	49

# Battelle

The Business of Innovation

**Project Client:** Exponent, Inc.  
**Project Name:** Chukchi Sea Contaminants Program  
**Project Number:** N106753-0001

Client ID	09-04-KF003-FC-01	09-04-KF003-FC-01			
Battelle ID	Q8724-P	Q8724MS-P			
Sample Type	SA	MS			
Collection Date	09/27/09	09/27/09			
Extraction Date	03/03/10	03/03/10			
Analysis Date	03/11/10	03/12/10			
Analytical Instrument	MS	MS			
% Moisture	82.35	82.67			
% Lipid	3.37	3.43			
Matrix	TISSUE	TISSUE			
Sample Size	3.54	3.48			
Size Unit-Basis	G_DRY	G_DRY			
Units	UG/KG_DRY	UG/KG_DRY	Target	% Recovery	Qualifier
Naphthalene	2.32 J	148.44	143.92	102	
C1-Naphthalenes	0.99 J	192.22			
C2-Naphthalenes	1.53 J	99.73			
C3-Naphthalenes	1.06 J	83.26			
C4-Naphthalenes	U	U			
Biphenyl	0.57 J	169.41	143.82	117	
Acenaphthylene	0.34 J	170.89	144.01	118	
Acenaphthene	U	156.39	144.05	109	
Fluorene	0.52 J	157.67	143.92	109	
C1-Fluorenes	1.06 J	2.49 J			
C2-Fluorenes	3.38 J	U			
C3-Fluorenes	U	U			
Anthracene	U	170.18	144.02	118	
Phenanthrene	1.12 J	153.88	143.98	106	
C1-Phenanthrenes/Anthracenes	1.12 J	119.97			
C2-Phenanthrenes/Anthracenes	1.69 J	120.53			
C3-Phenanthrenes/Anthracenes	0.59 J	1.53 J			
C4-Phenanthrenes/Anthracenes	U	U			
Dibenzothiophene	U	157.73	144.05	109	
C1-Dibenzothiophenes	U	U			
C2-Dibenzothiophenes	U	U			
C3-Dibenzothiophenes	U	U			
Fluoranthene	0.42 J	164.16	143.97	114	
Pyrene	0.55 J	172.37	143.88	119	
C1-Fluoranthenes/Pyrenes	U	U			
C2-Fluoranthenes/Pyrenes	U	U			
C3-Fluoranthenes/Pyrenes	U	U			
Benzo(a)anthracene	U	179.42	144.02	125	
Chrysene	0.4 J	174.1	144.11	121	
C1-Chrysenes	U	U			
C2-Chrysenes	U	U			
C3-Chrysenes	U	U			
C4-Chrysenes	U	U			
Benzo(b)fluoranthene	U	231.54	143.88	161	N
Benzo(k)fluoranthene	U	160.85	143.91	112	
Benzo(e)pyrene	U	143.01	144.05	99	
Benzo(a)pyrene	U	150.08	144.09	104	
Perylene	U	134.69	144.02	94	
Indeno(1,2,3-cd)pyrene	U	94	144.07	65	N
Dibenz(a,h)anthracene	U	101.98	144.02	71	
Benzo(g,h,i)perylene	U	71.35	143.88	50	N
Total PAH	17.66 J	3681.87			

### Surrogate Recoveries (%)

Naphthalene-d8	74	94
Acenaphthene-d10	84	84
Phenanthrene-d10	78	78
Benzo(a)pyrene-d12	40	51

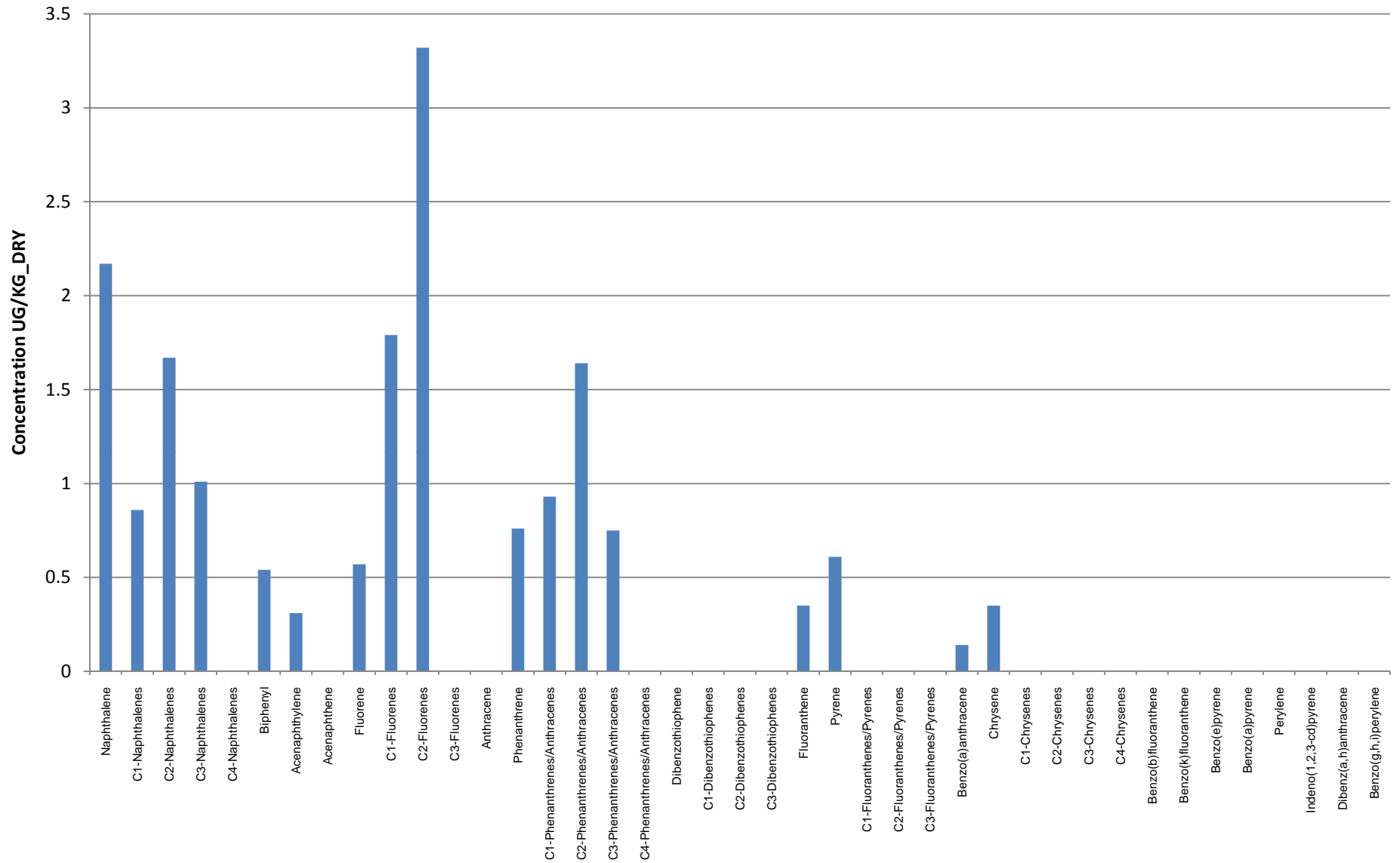
Surrogate Corrected

Analyzed by Lizotte Jr, Robert  
5/11/2010

MS: T10-0026MS-Master\_157-Final

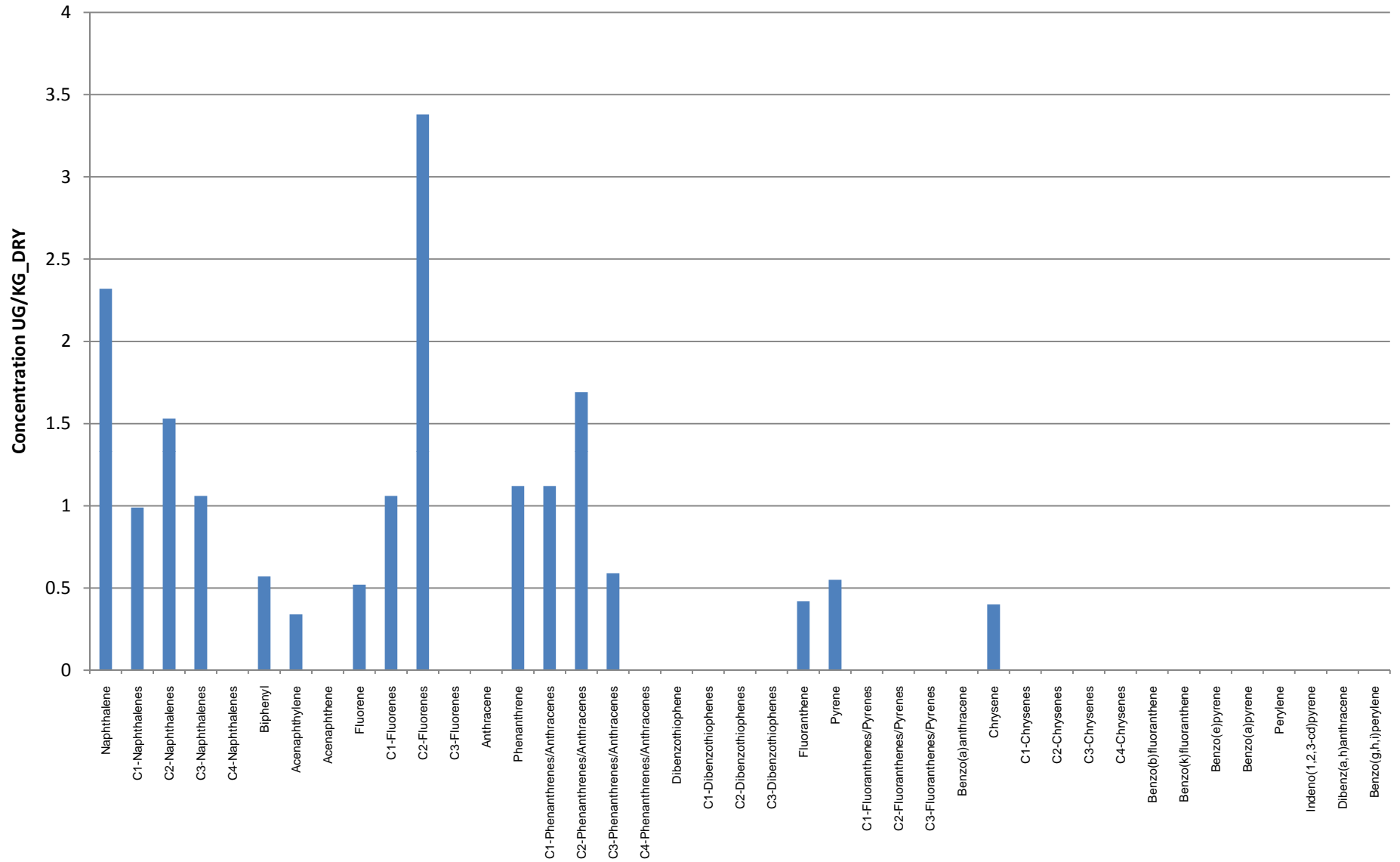
# Analyte Profile Histogram

## 09-04-KF001-FC-01 - Q8723-P

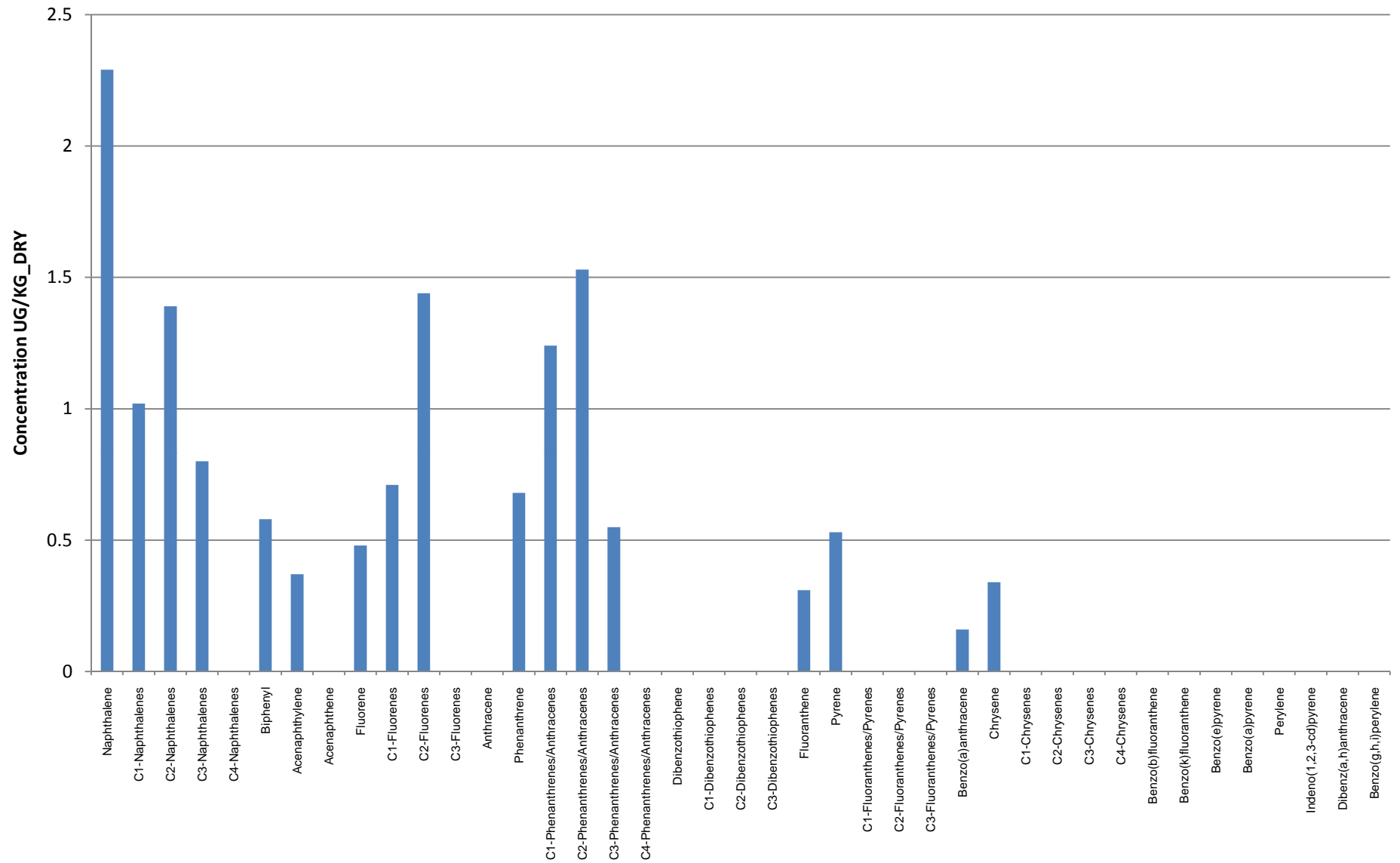


# Analyte Profile Histogram

## 09-04-KF003-FC-01 - Q8724-P



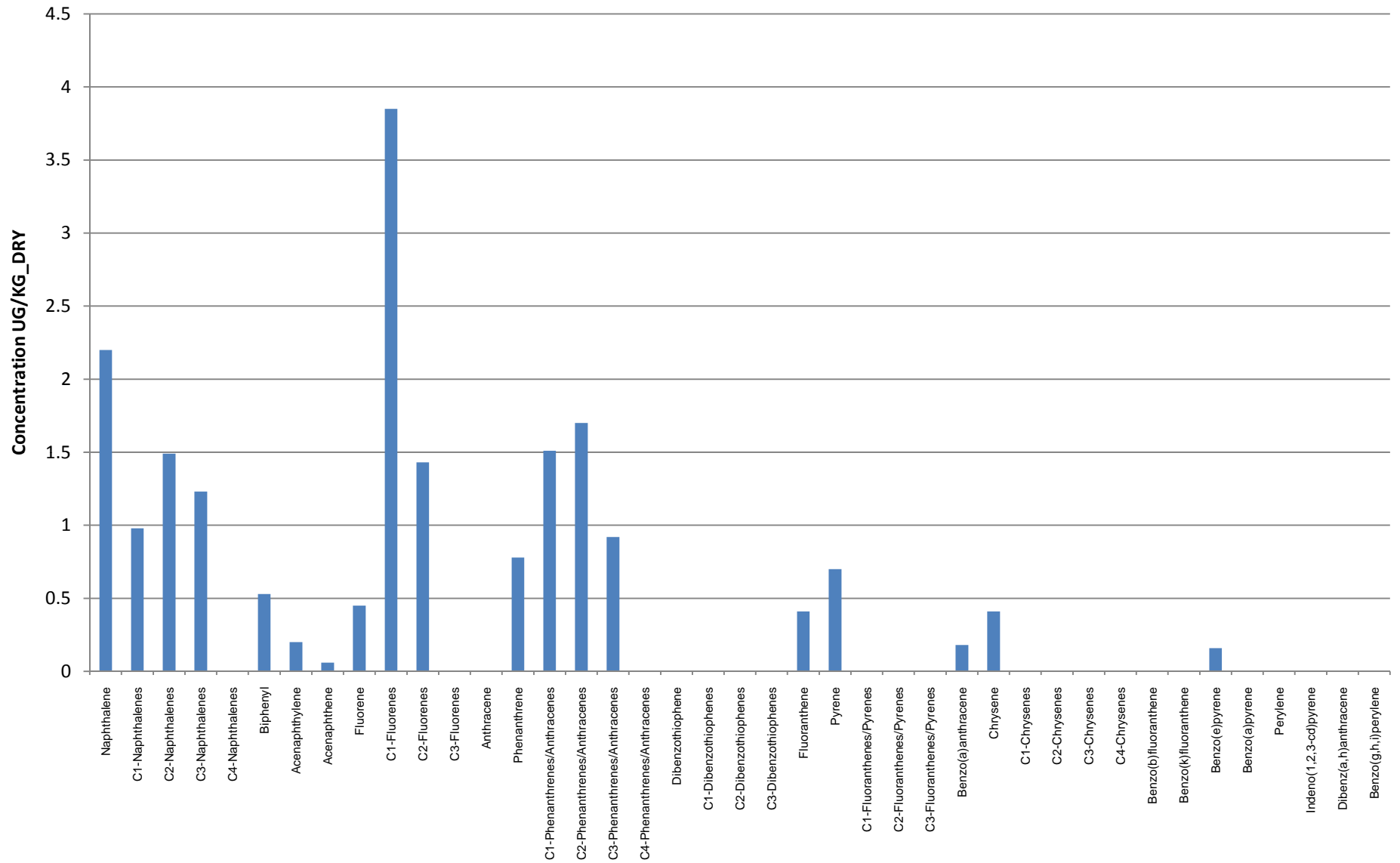
## Analyte Profile Histogram 09-04-KF005-FC-01 - Q8725-P





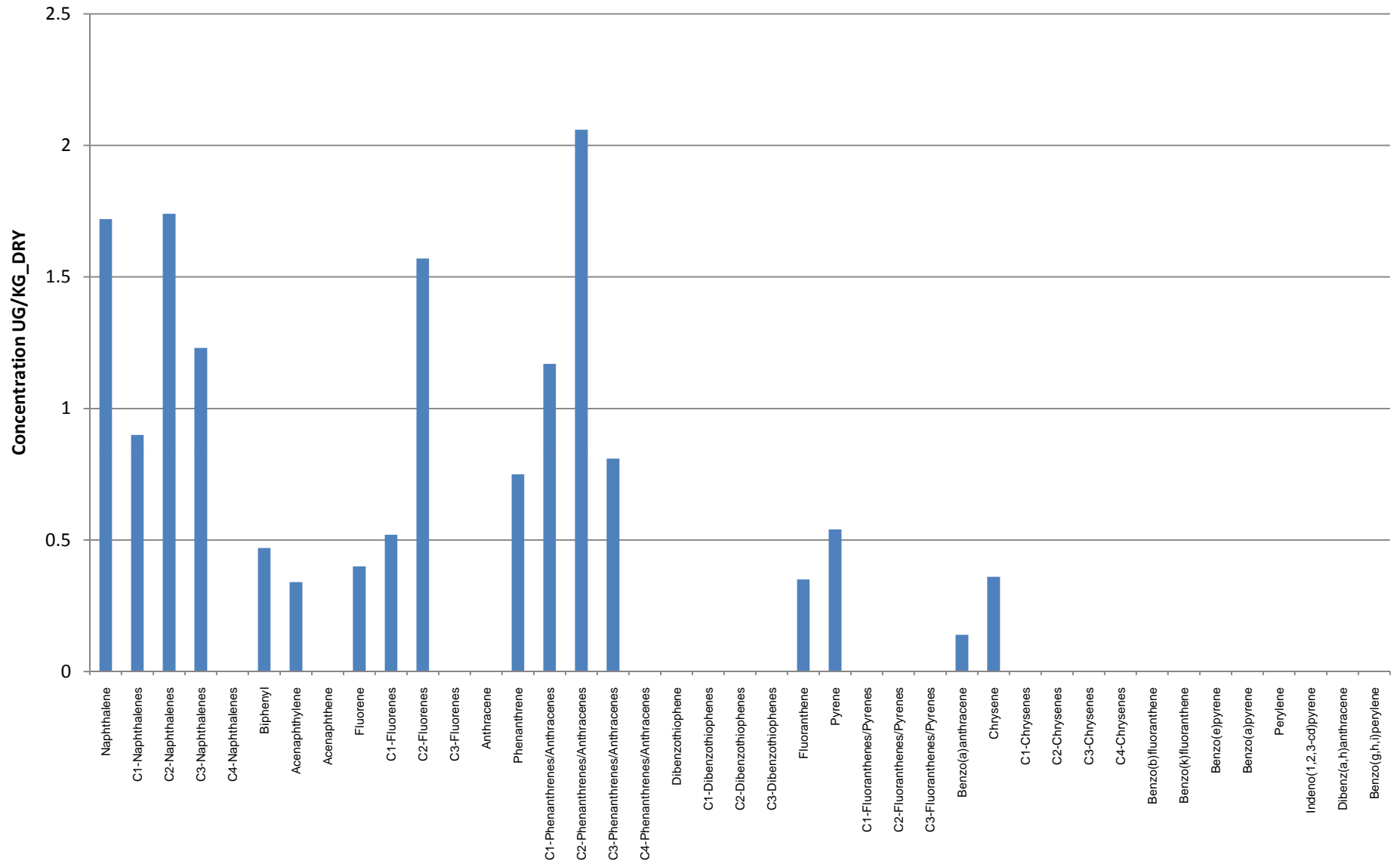
## Analyte Profile Histogram

### 09-04-KF007-FC-01 - Q8726-P



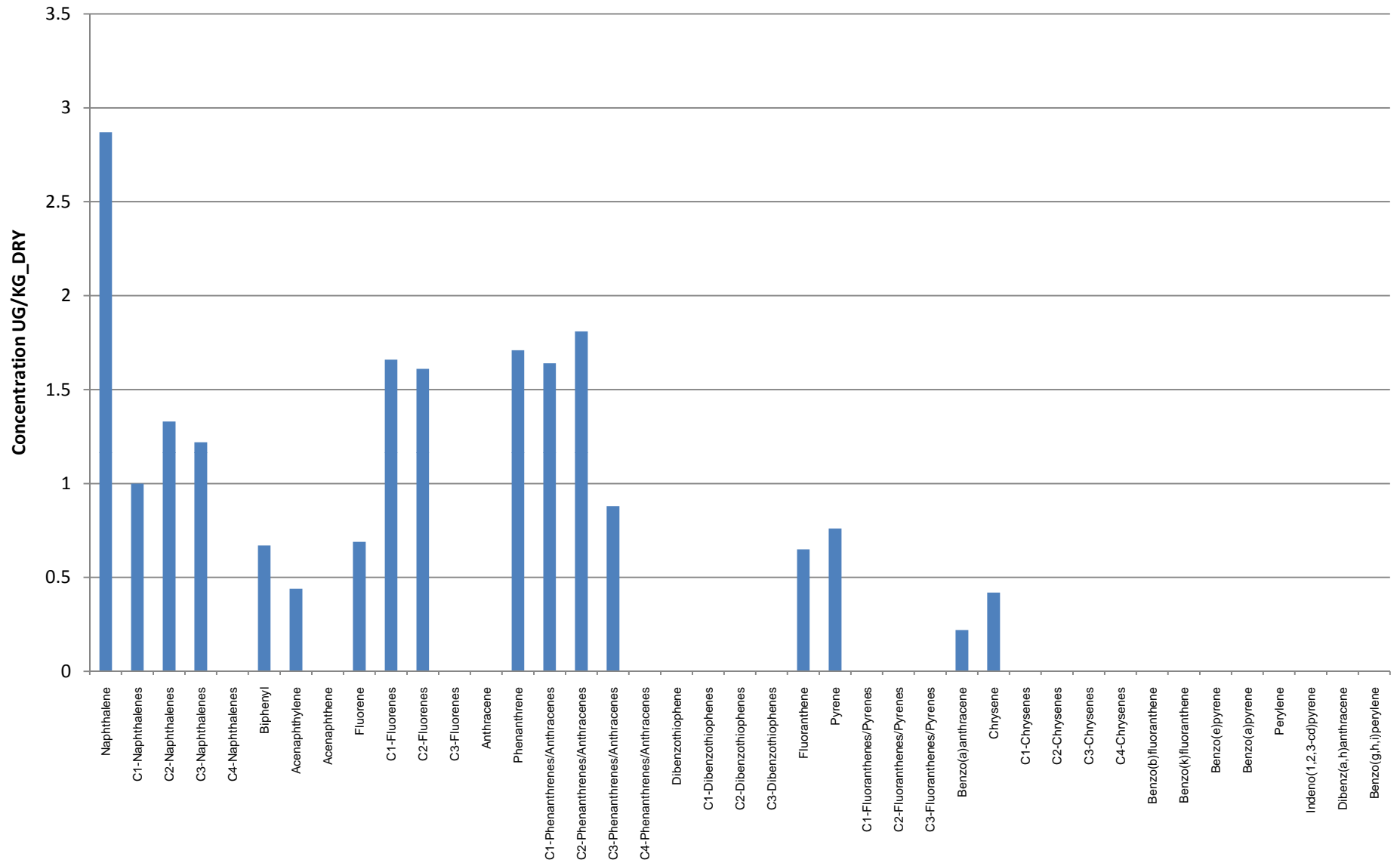
## Analyte Profile Histogram

### 09-04-KF009-FC-01 - Q8727-P



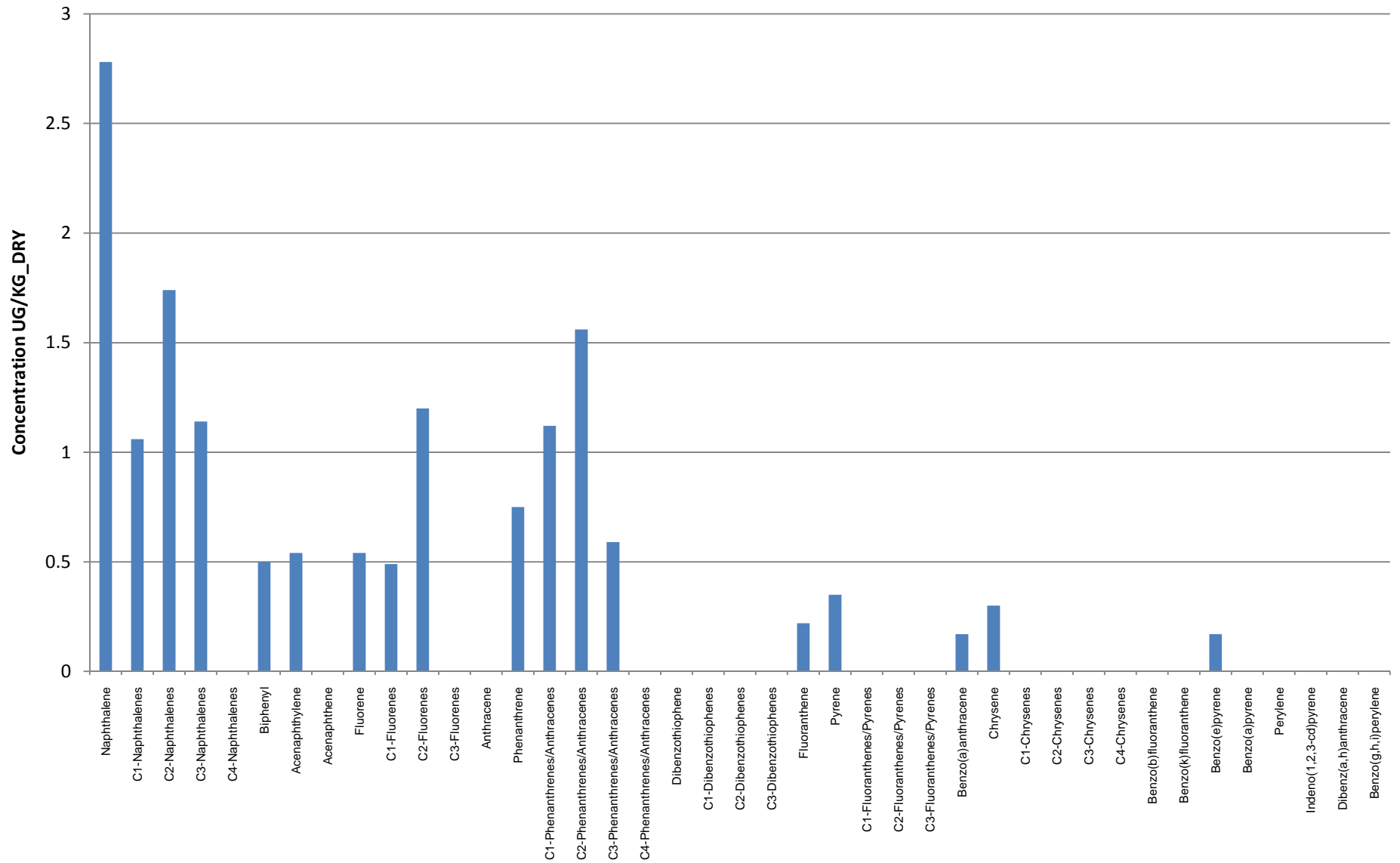
## Analyte Profile Histogram

### 09-04-KF011-FC-01 - Q8728-P



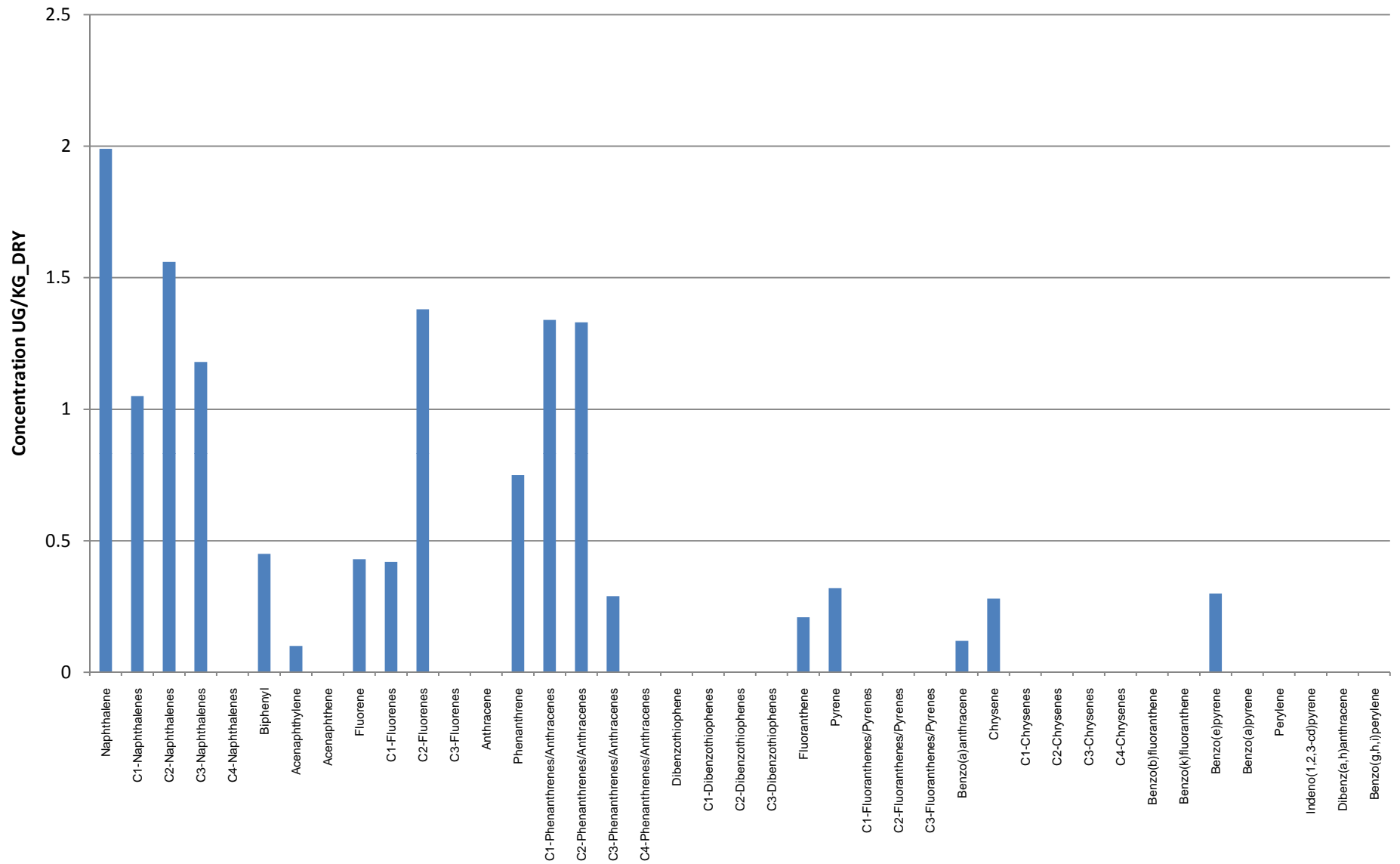
## Analyte Profile Histogram

### 09-04-KF013-FC-01 - Q8729-P



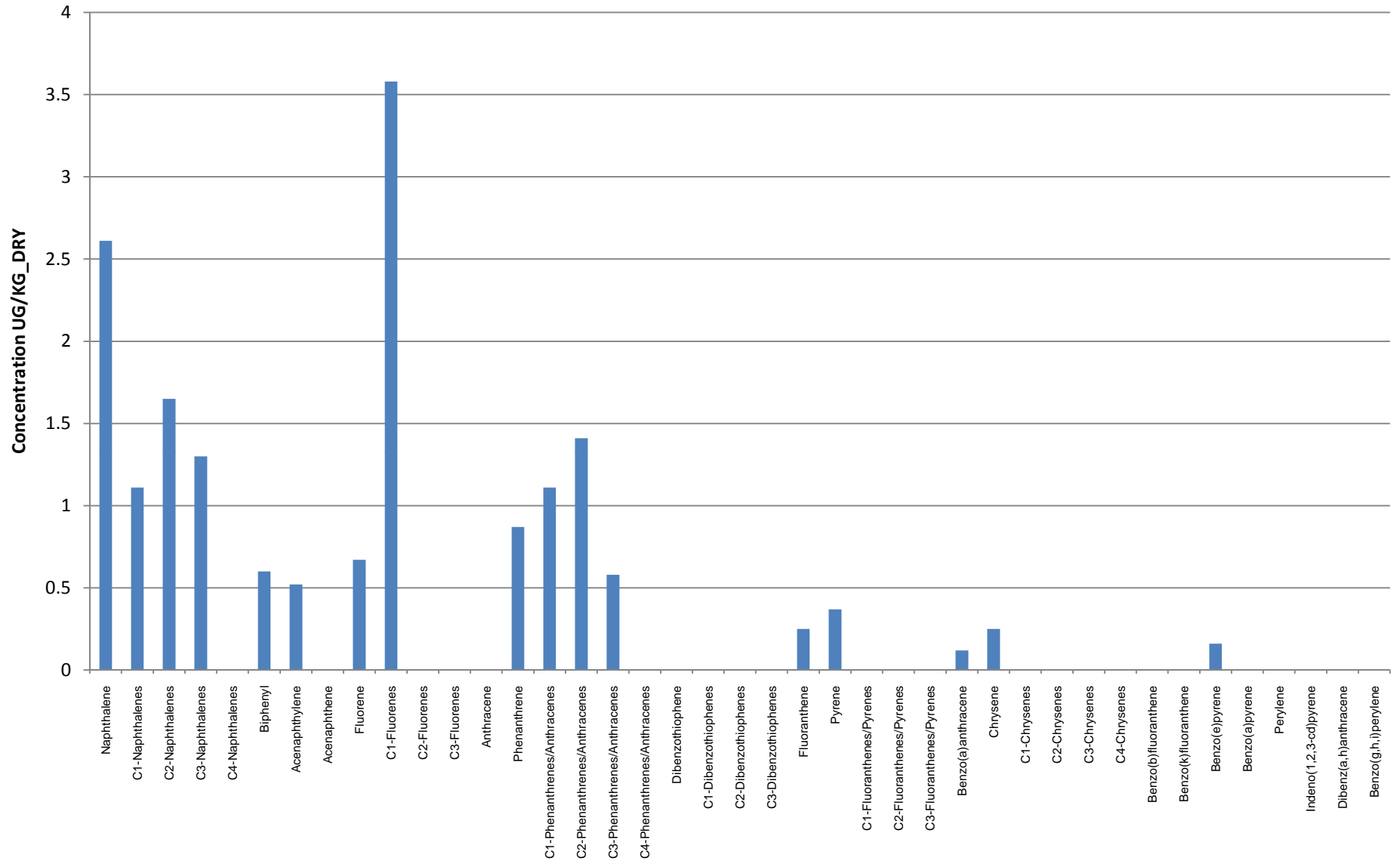
## Analyte Profile Histogram

### 09-04-KF015-FC-01 - Q8730-P



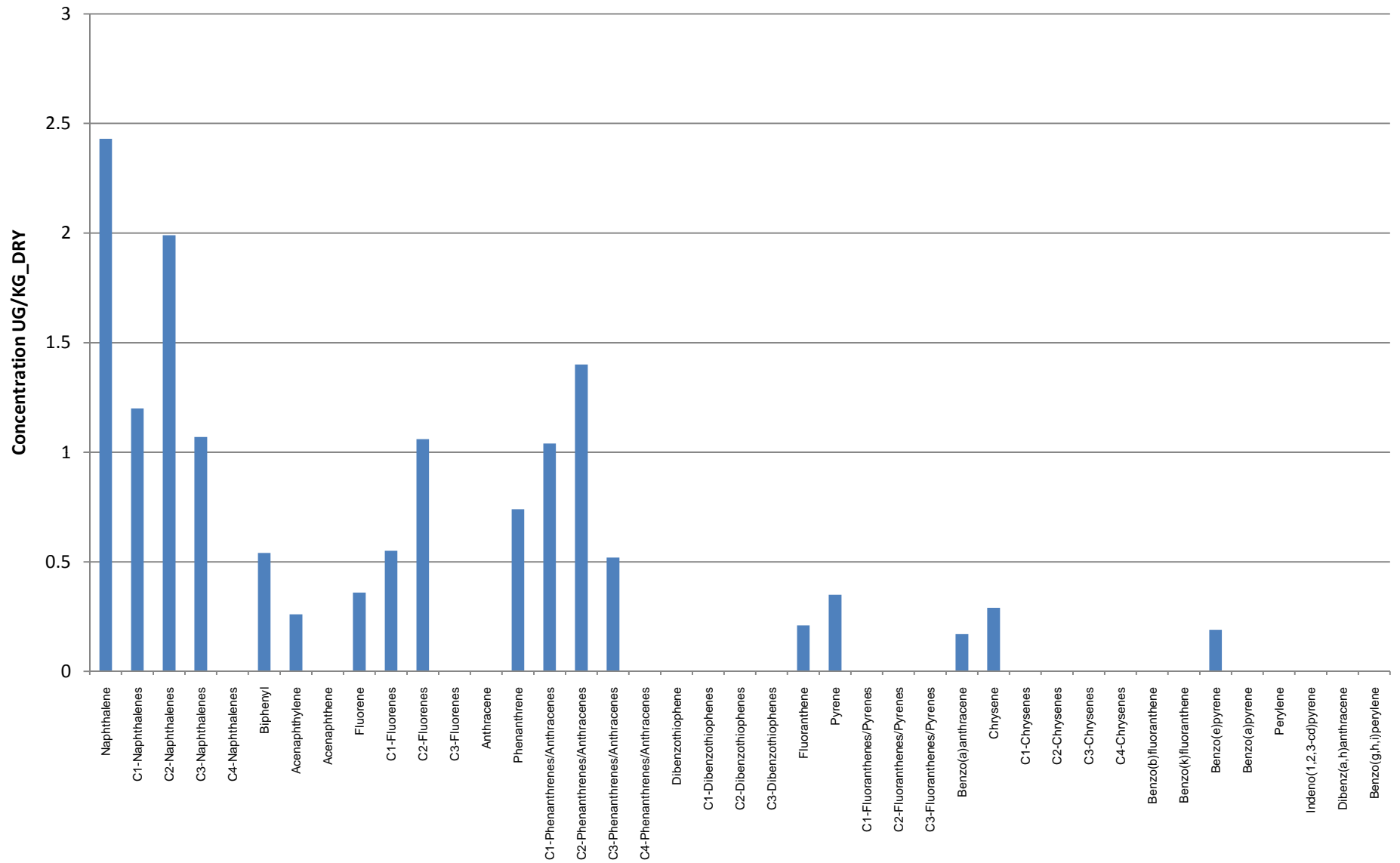
## Analyte Profile Histogram

### 09-04-KF017-FC-01 - Q8731-P



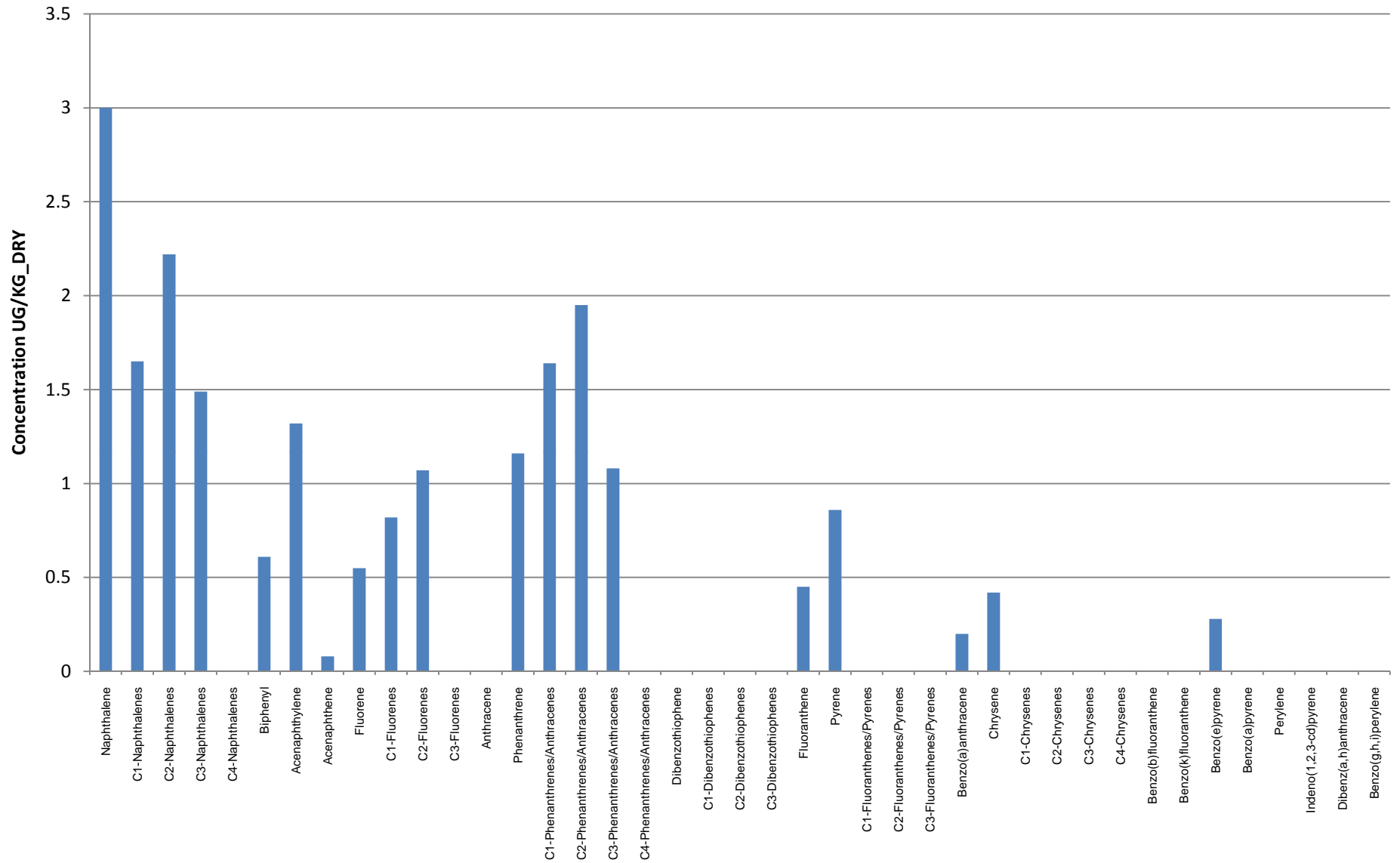
## Analyte Profile Histogram

### 09-04-KF019-FC-01 - Q8732-P



# Analyte Profile Histogram

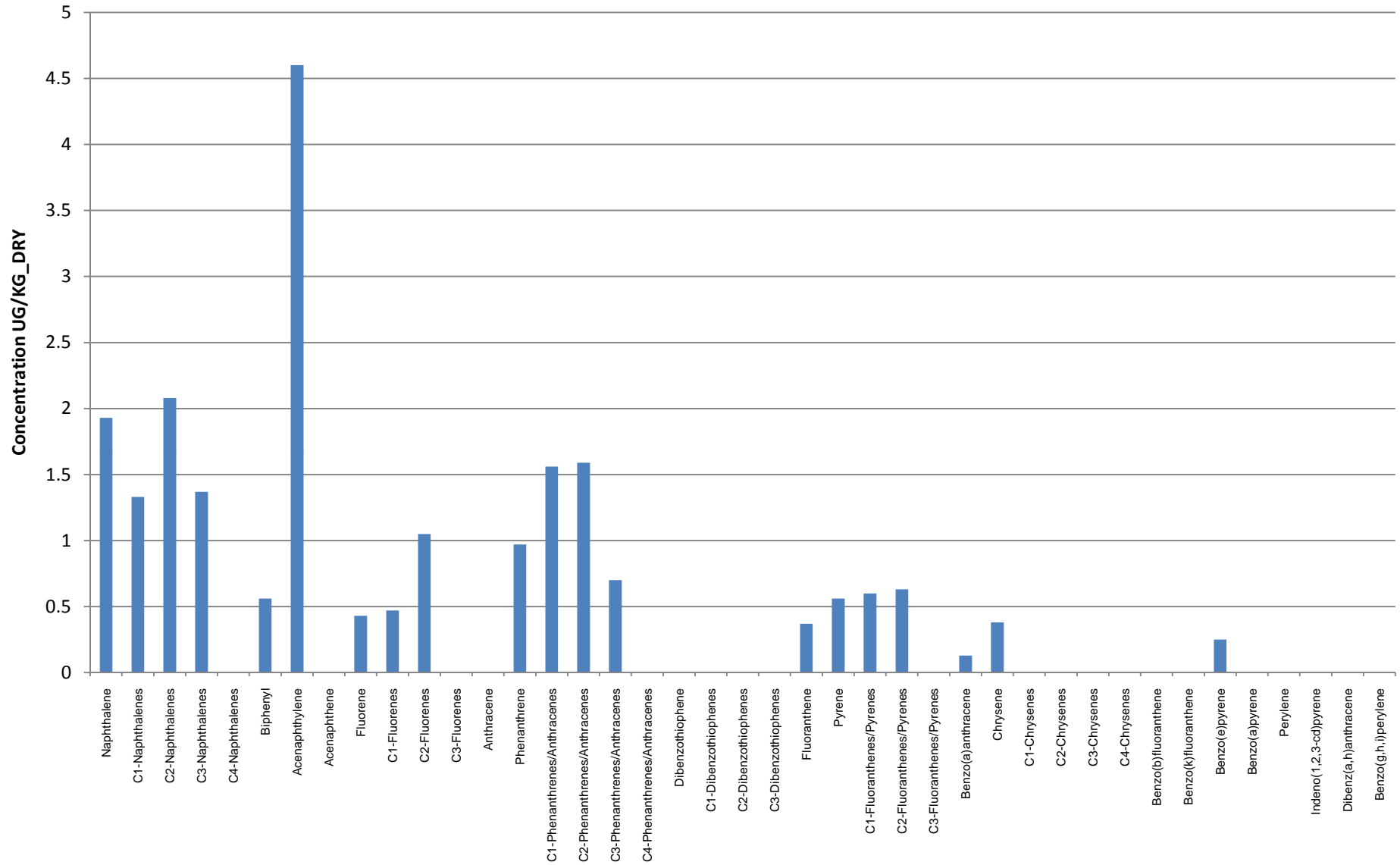
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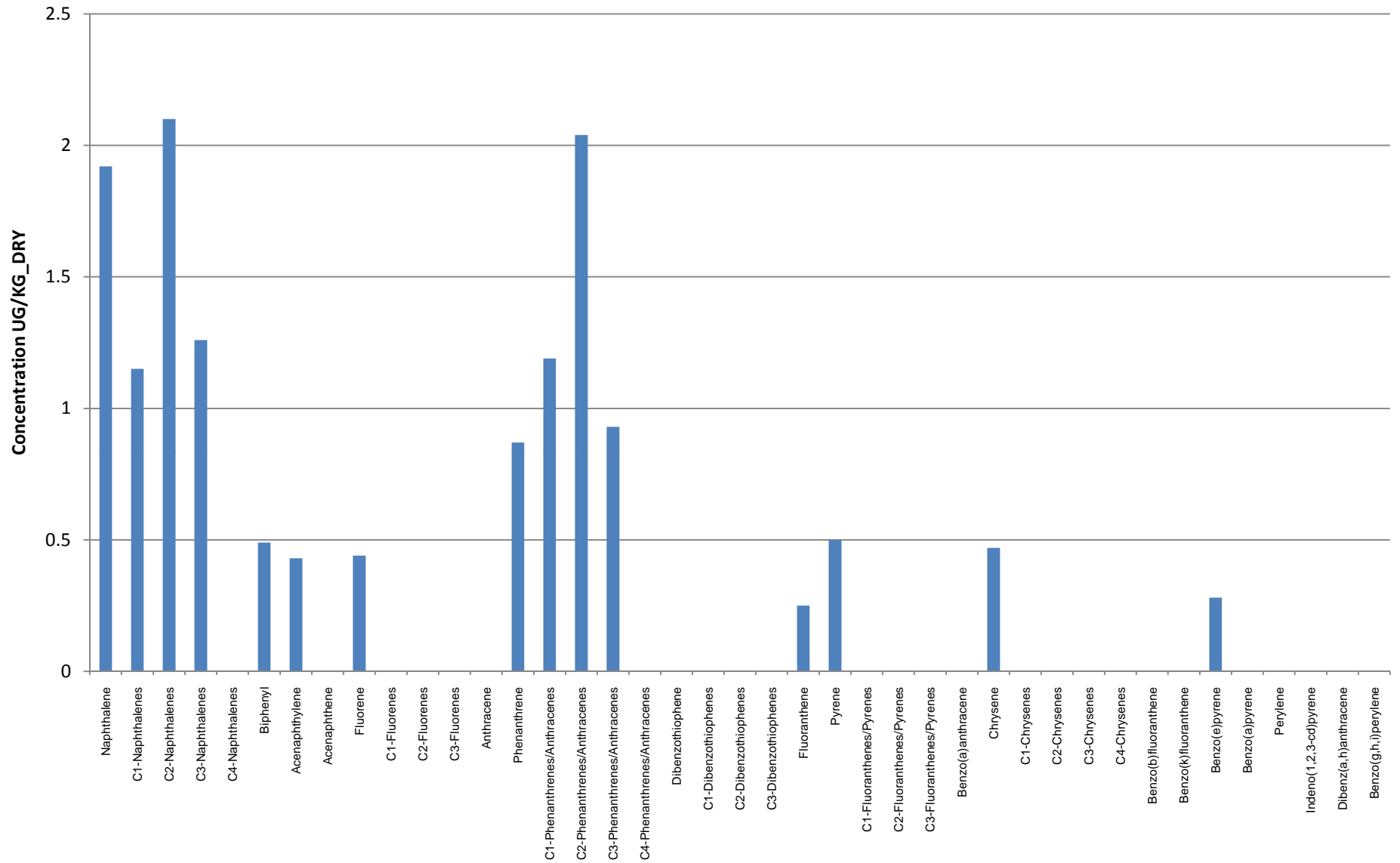
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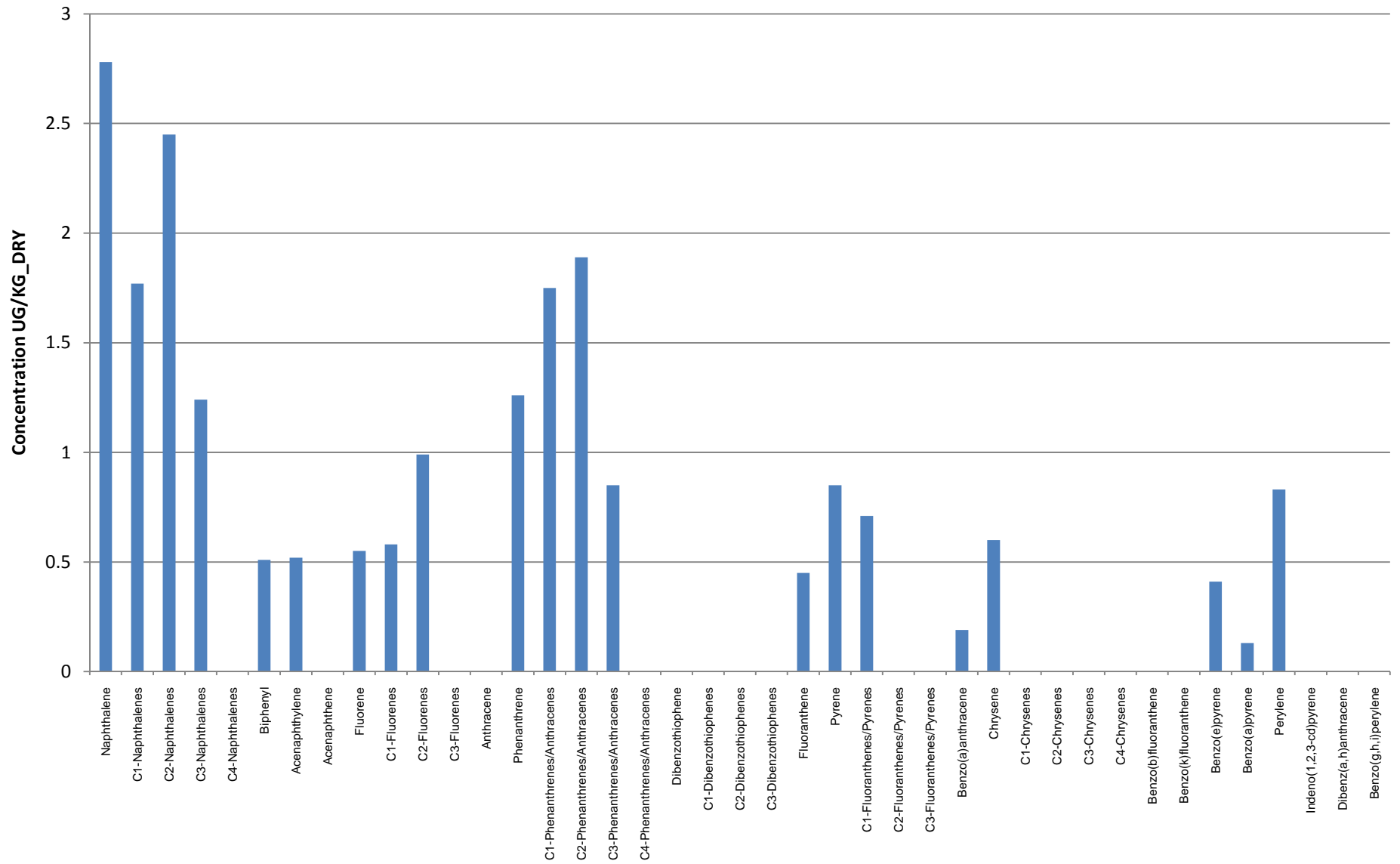
# Analyte Profile Histogram

## 09-04-KF009-FC-02 - Q8741-P



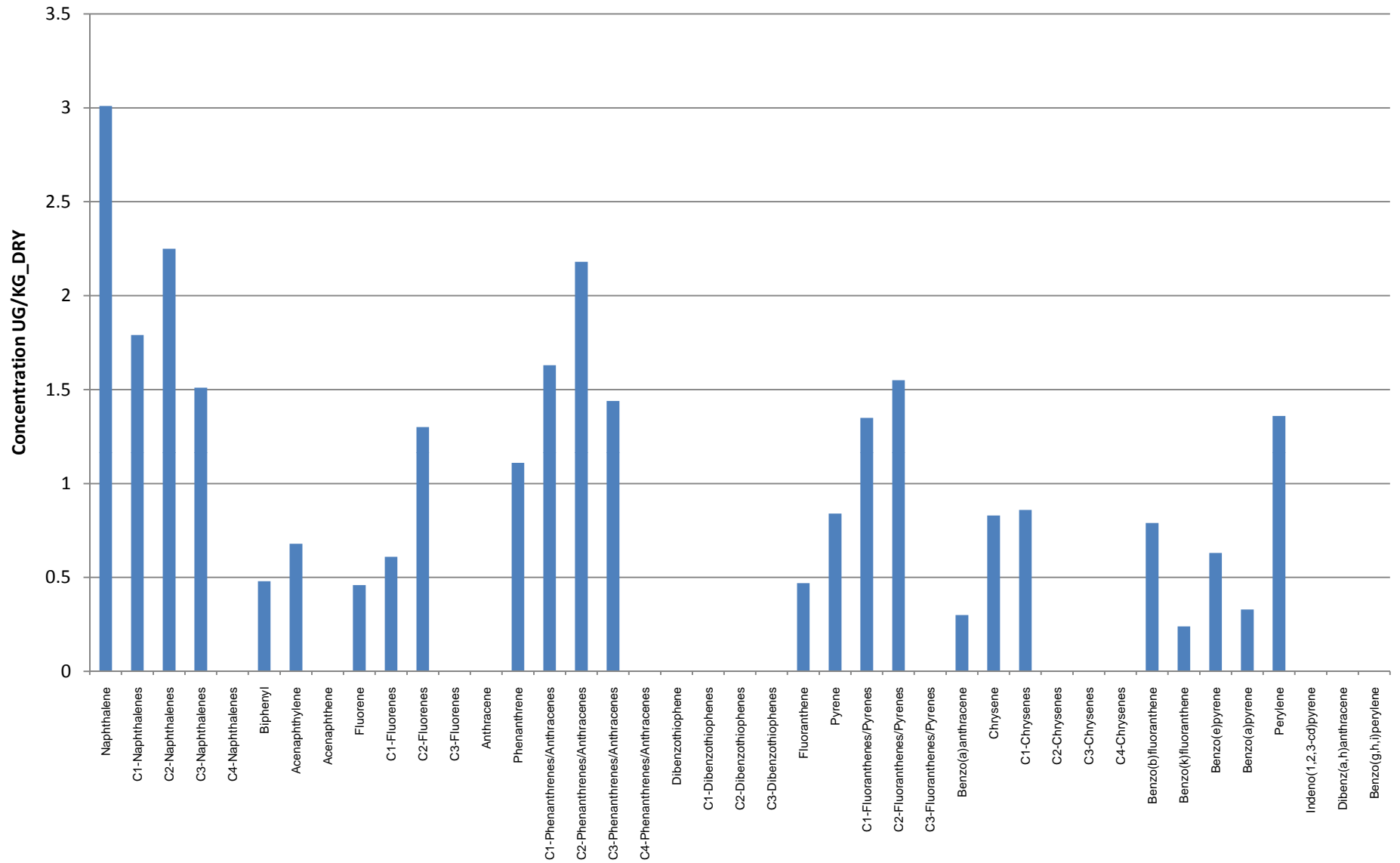
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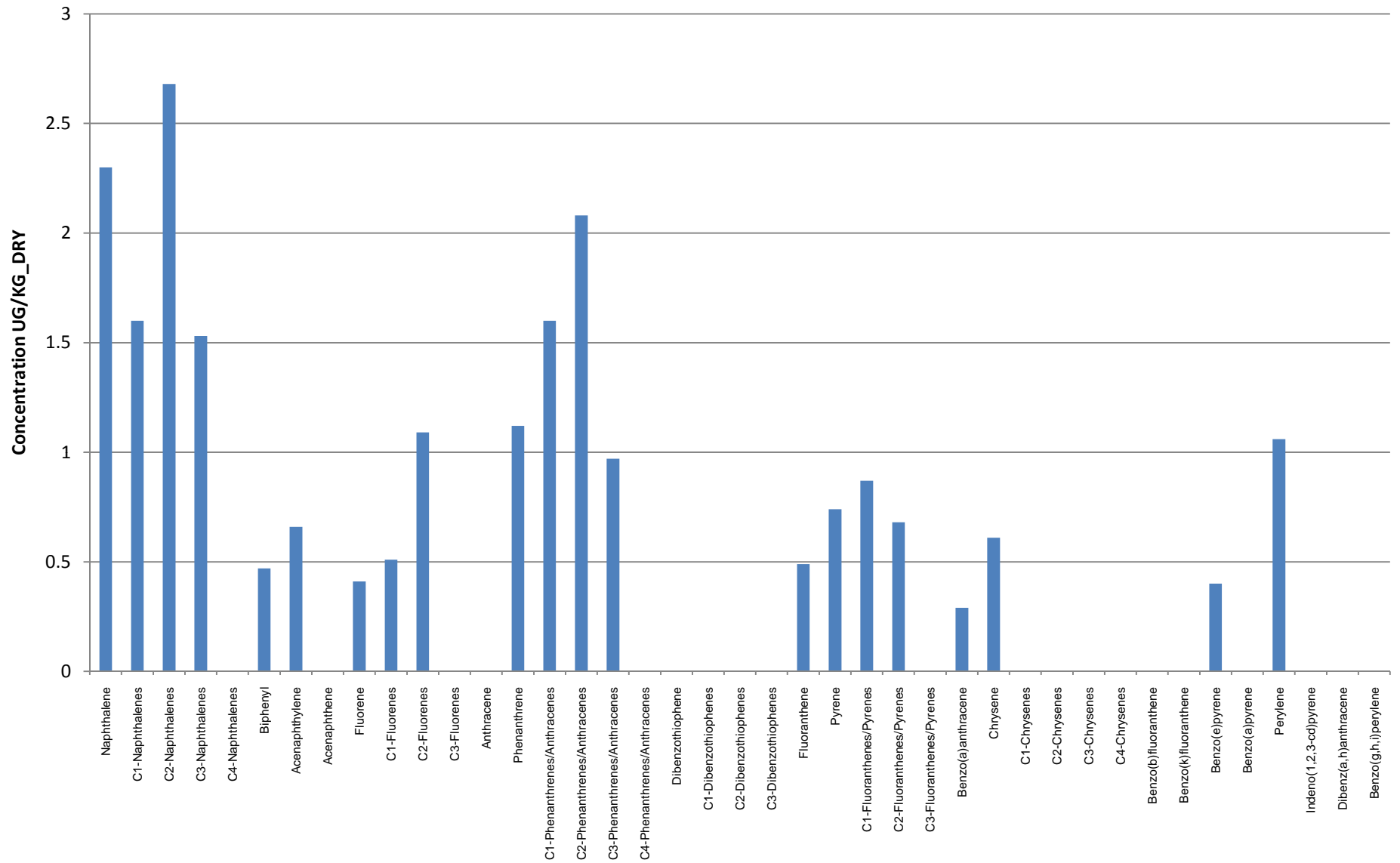
## Analyte Profile Histogram

### 09-04-BF003-FC-01 - Q8745-P



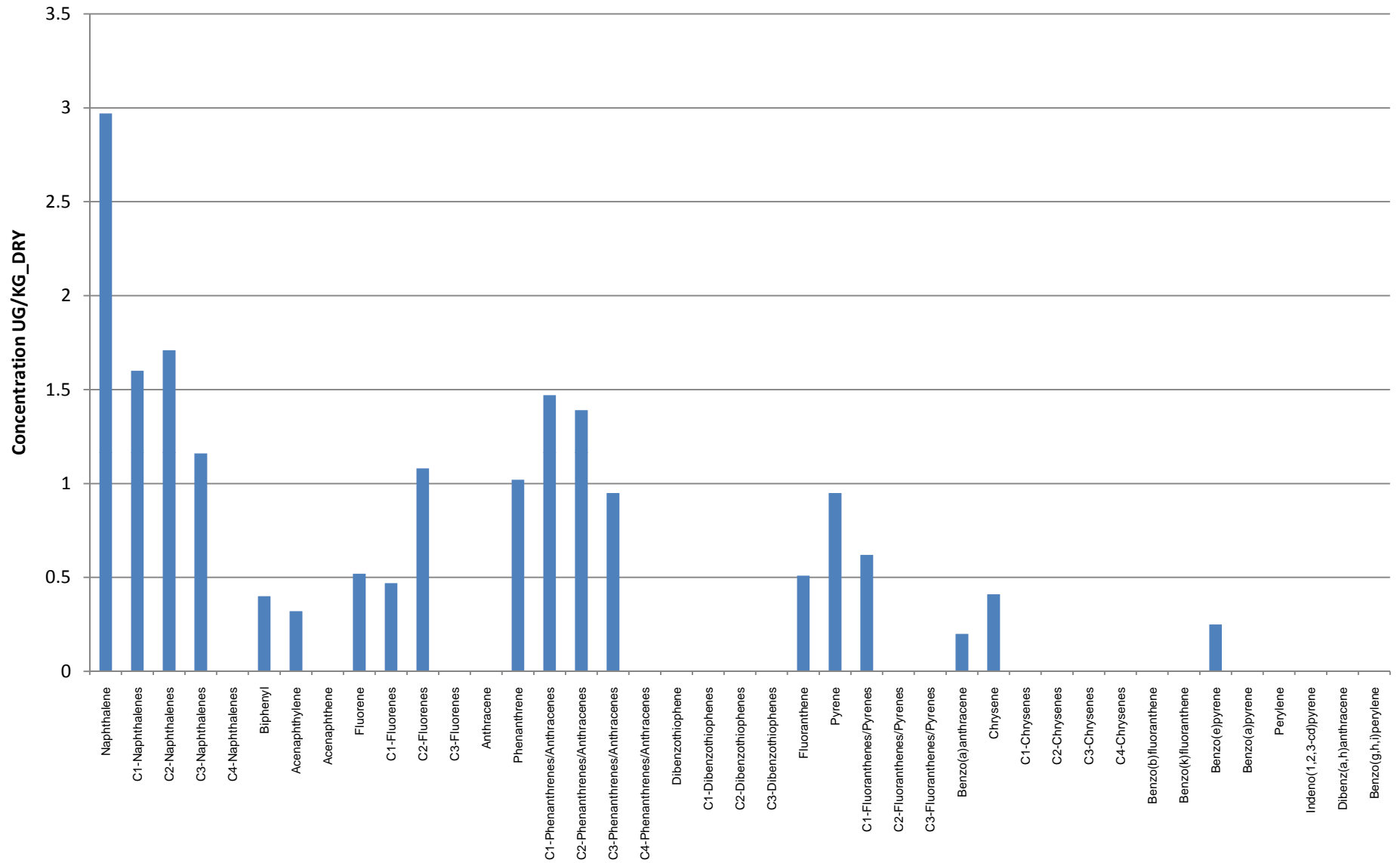
## Analyte Profile Histogram

### 09-04-BF007-FC-01 - Q8746-P



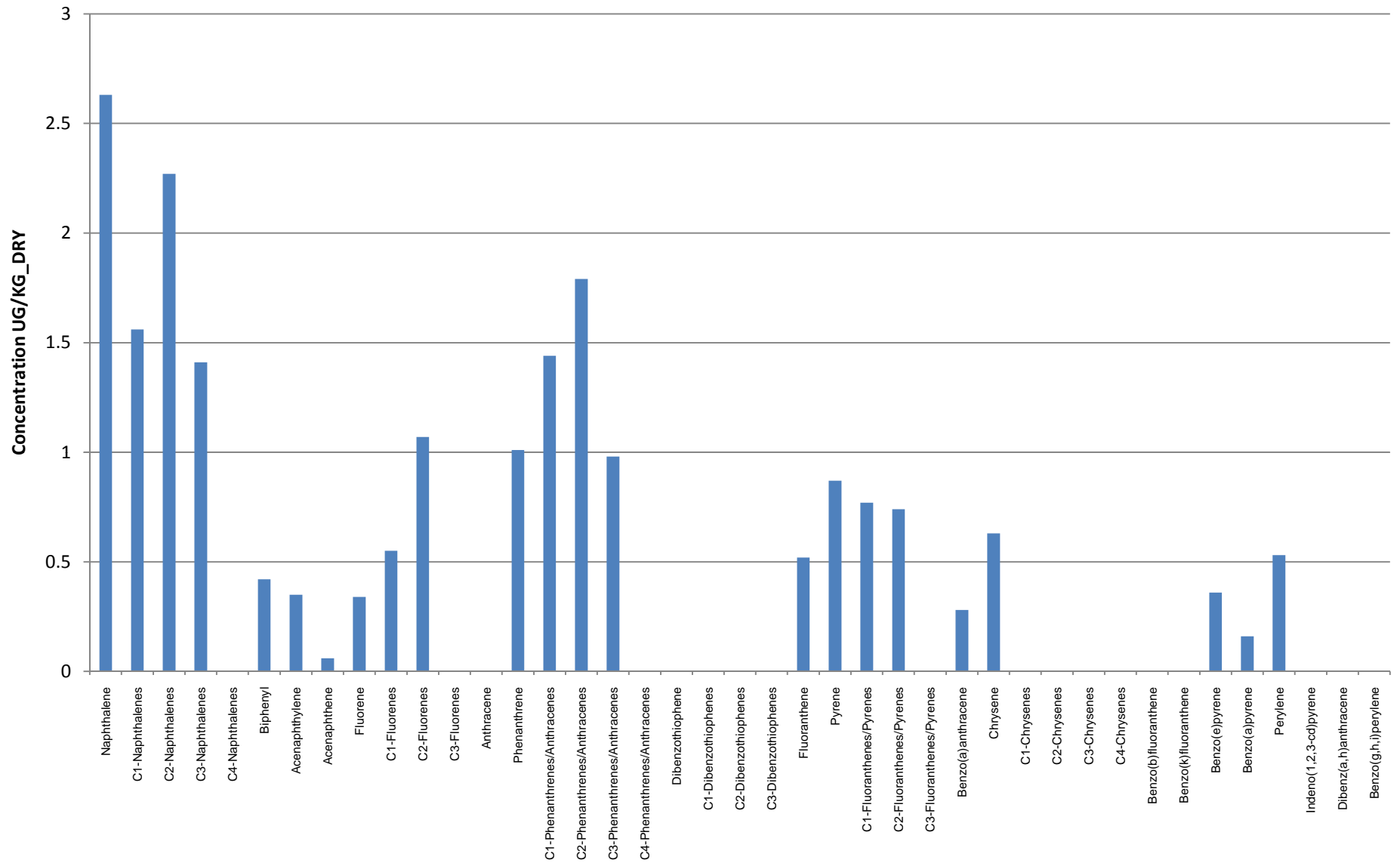
## Analyte Profile Histogram

### 09-04-BF009-FC-01 - Q8747-P



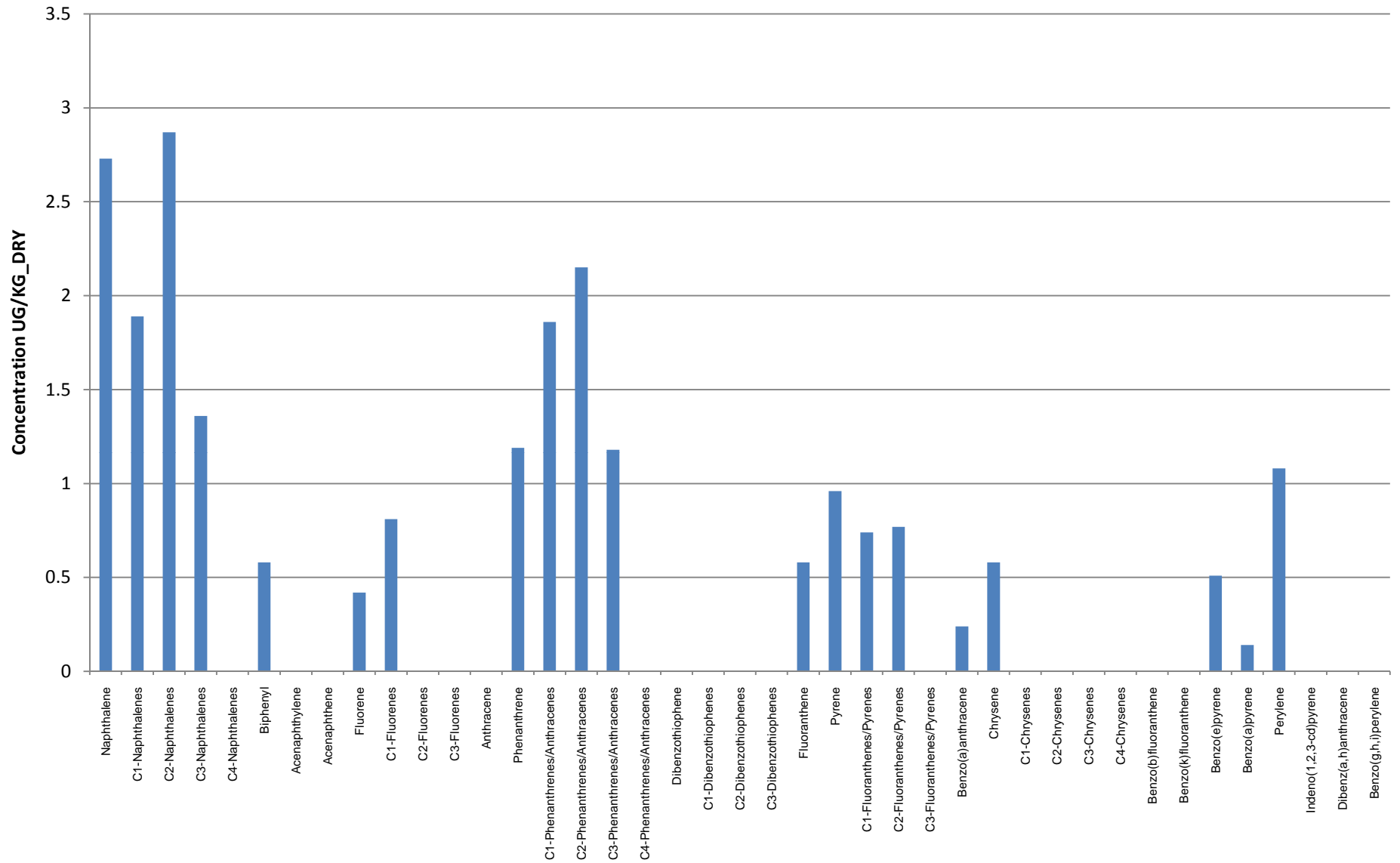
## Analyte Profile Histogram

### 09-04-BF011-FC-01 - Q8748-P



## Analyte Profile Histogram

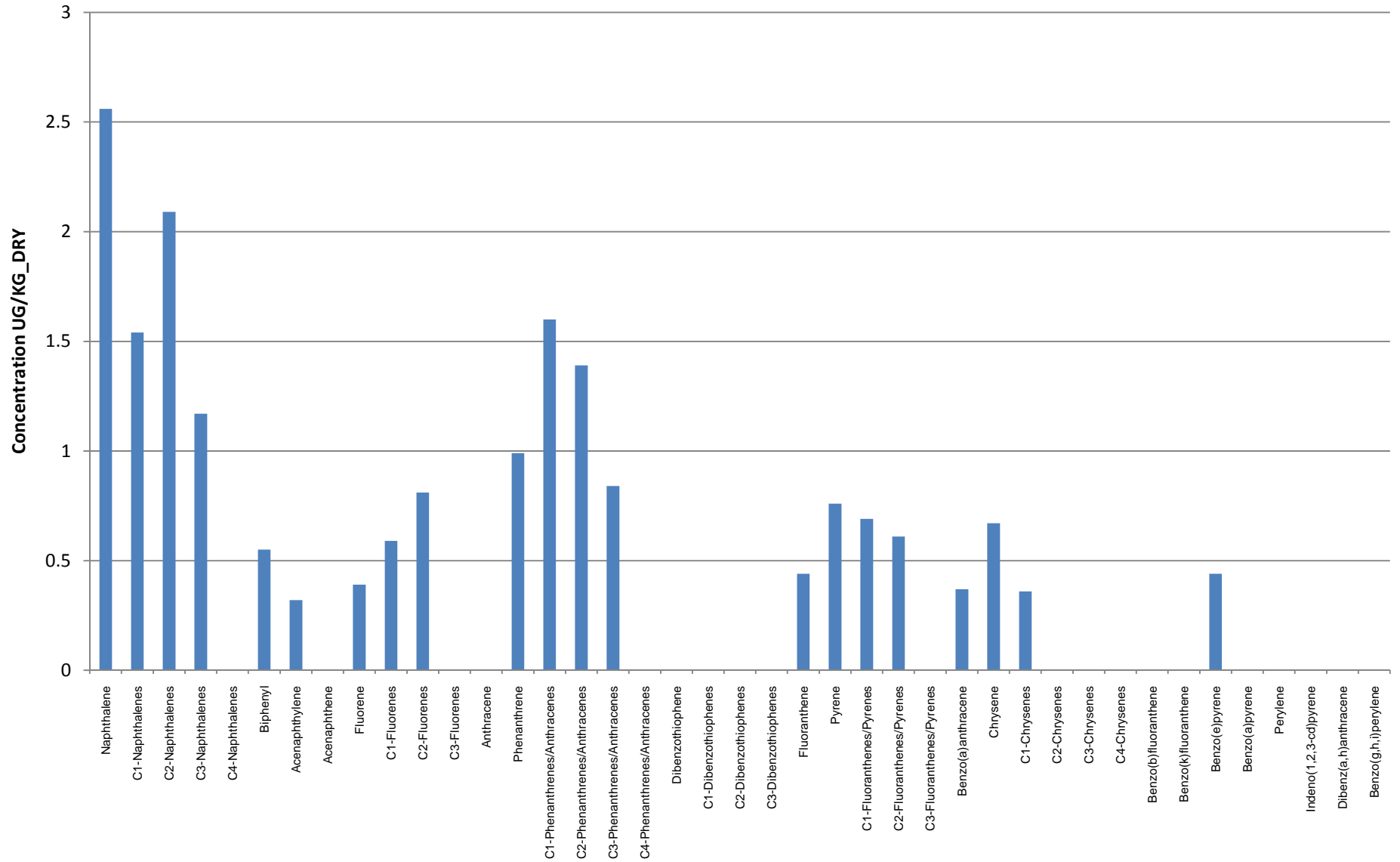
### 09-04-BF013-FC-01 - Q8749-P





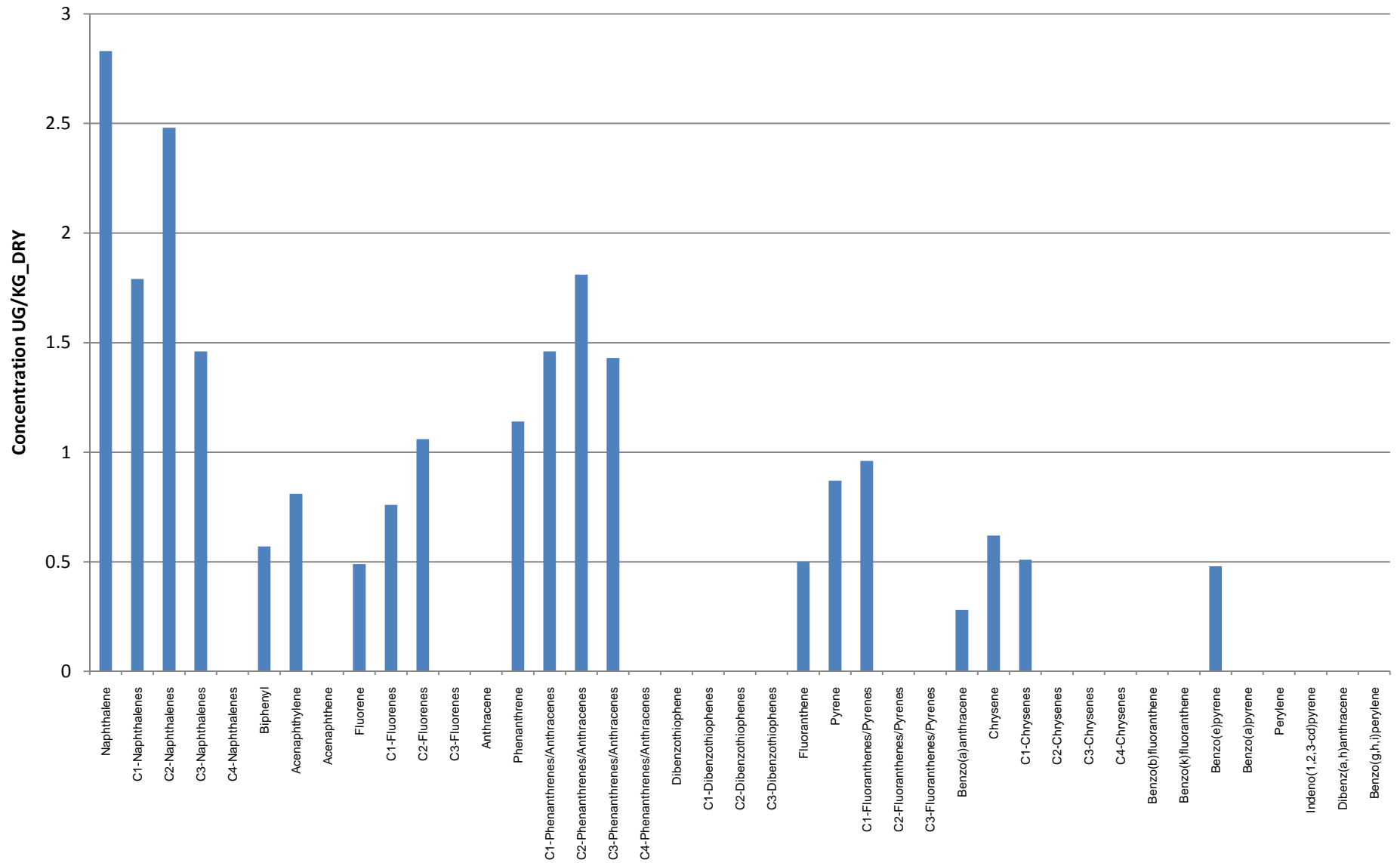
## Analyte Profile Histogram

### 09-04-BF015-FC-01 - Q8750-P



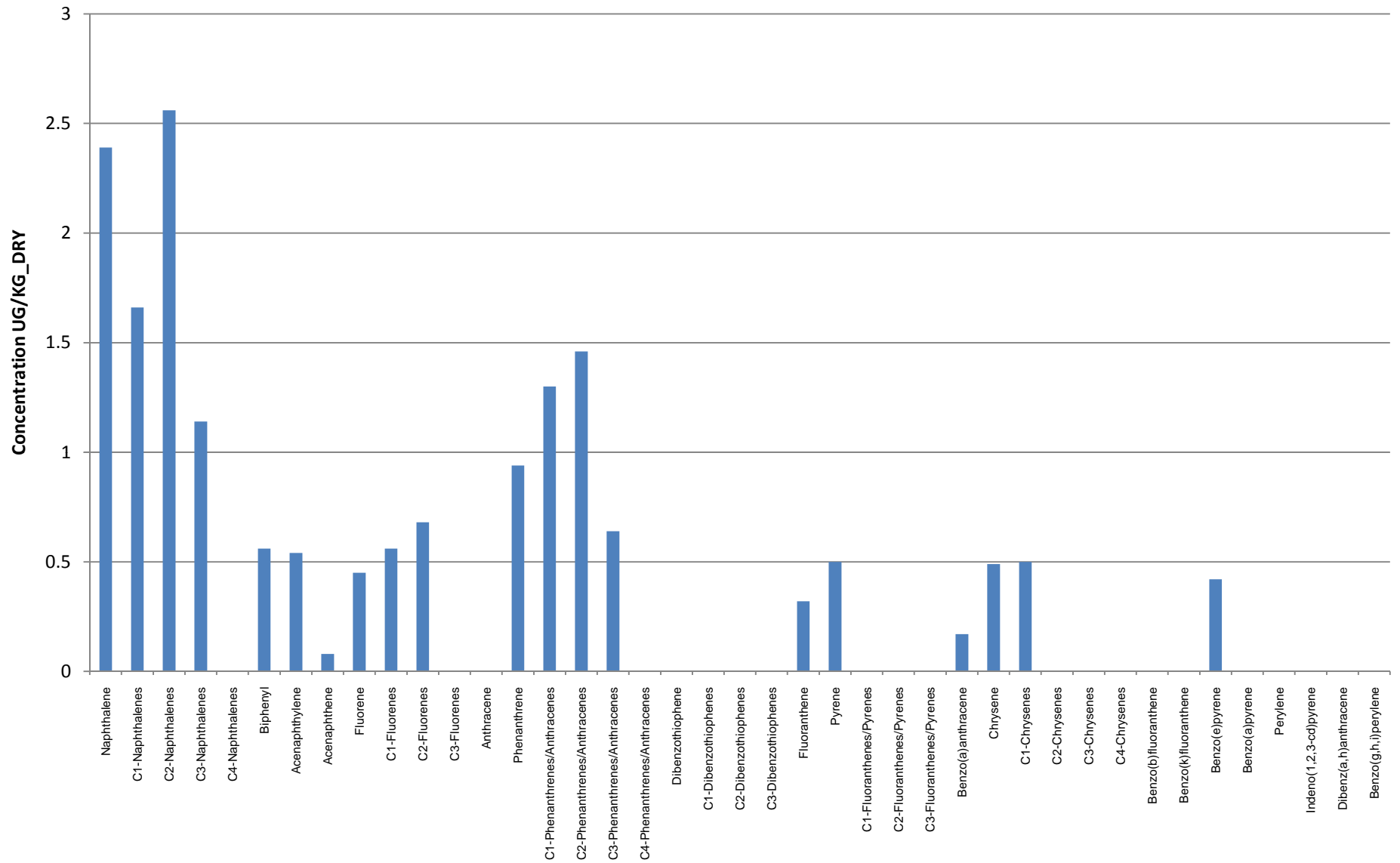
## Analyte Profile Histogram

### 09-04-BF017-FC-01 - Q8751-P



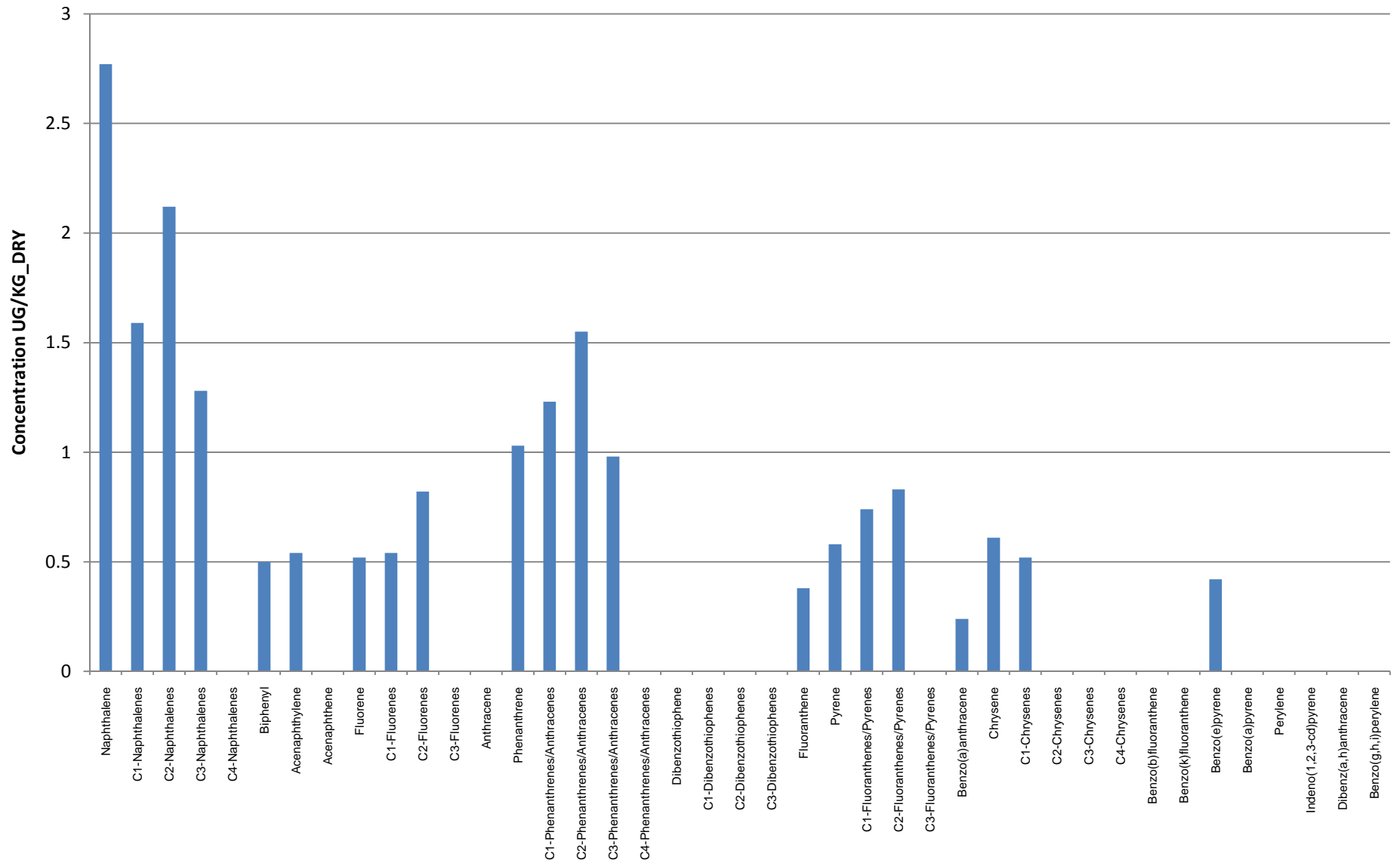
## Analyte Profile Histogram

### 09-04-BF019-FC-01 - Q8752-P



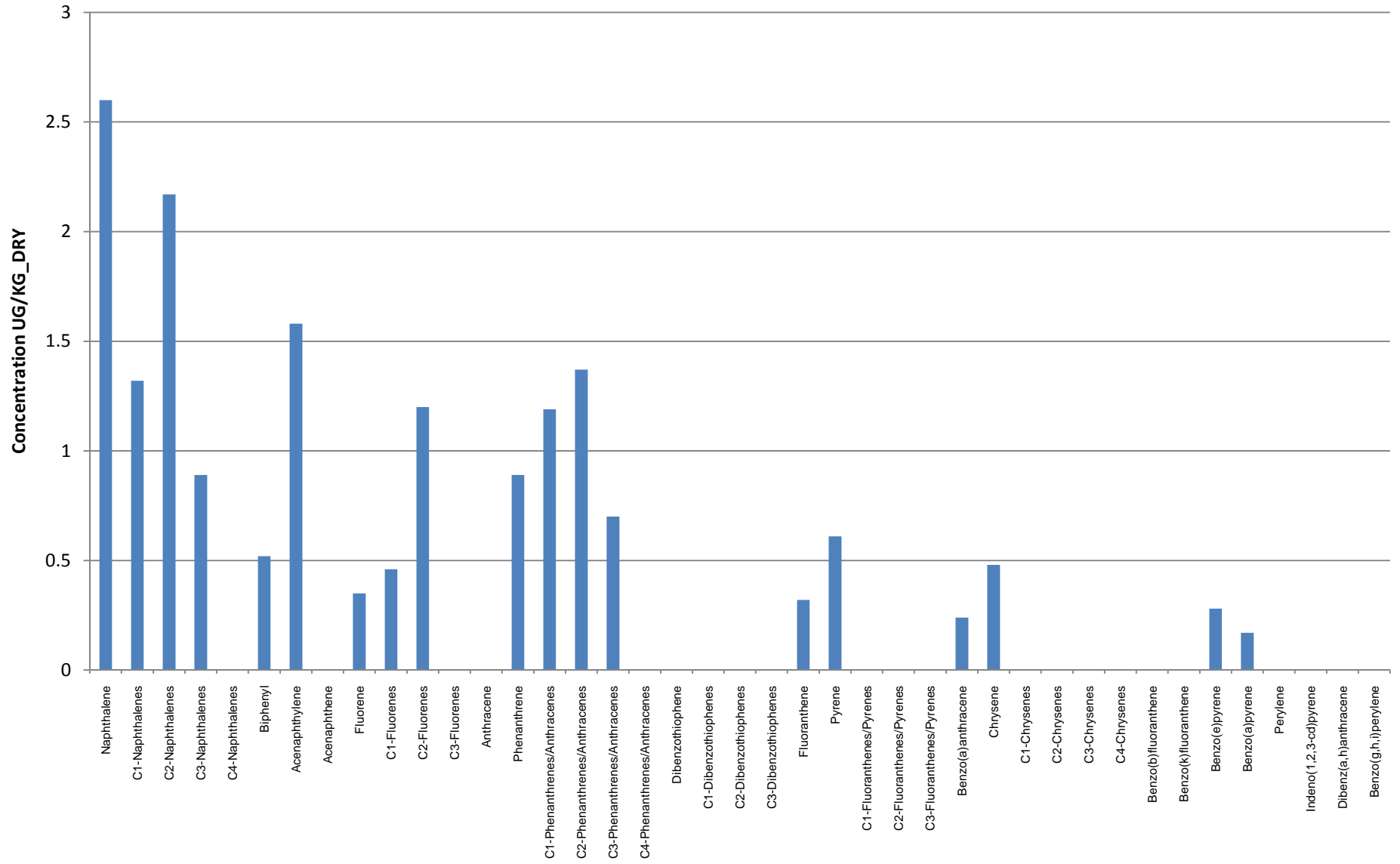
# Analyte Profile Histogram

## 09-04-BF021-FC-01 - Q8753-P



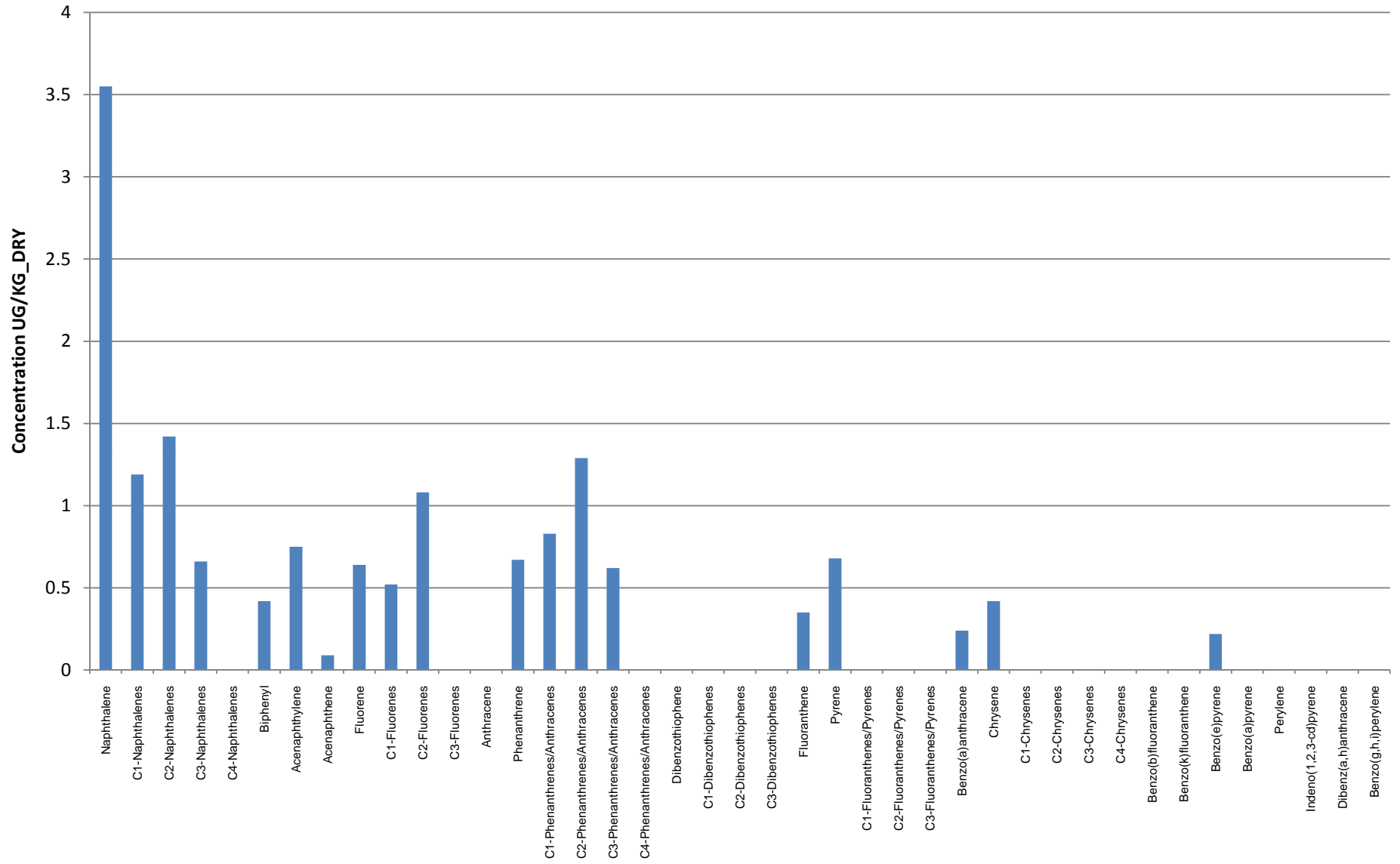
## Analyte Profile Histogram

### 09-04-BF023-FC-01 - Q8754-P



## Analyte Profile Histogram

### 09-04-BF025-FC-01 - Q8755-P



## **Appendix B**

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### **Metals Data and Quality Control Results**

Water content and trace metal concentrations (dry weight) in fish tissue samples.

Sample Identification	Water Content (%)	Ag (µg/g)	As (µg/g)	Ba (µg/g)	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Fe (µg/g)	Hg (µg/g)	Mn (µg/g)	Pb (µg/g)	Se (µg/g)	Zn (µg/g)
<b>Burger Stations</b>													
09-BF001-FC-01	78.4	0.14	10.8	15.9	0.38	2.08	4.1	1130	0.056	16.7	0.703	2.93	84.9
09-BF003-FC-01	78.7	0.12	11.3	7.75	0.25	1.28	3.7	525	0.037	9.6	0.402	3.07	80.2
09-BF007-FC-01	78.6	0.14	7.62	8.01	0.24	1.26	4.6	692	0.045	10.6	0.982	2.73	75.8
09-BF009-FC-01	79.3	0.084	8.65	48.4	0.22	1.08	4.2	326	0.034	10.6	0.694	2.72	81.5
09-BF011-FC-01*	79.9	0.13	9.28	9.58	0.26	1.41	4.9	1040	0.055	10.6	0.748	2.71	83.2
09-BF011-FC-01*	79.9	0.13	9.46	9.73	0.27	1.38	4.9	1030	0.057	10.5	0.732	2.76	82.4
09-BF013-FC-01	81.3	0.072	13.3	10.0	0.29	1.36	4.0	918	0.049	13.6	0.558	2.81	82.3
09-BF015-FC-01	77.0	0.060	10.5	7.99	0.17	1.43	2.9	604	0.072	9.4	0.544	3.05	83.0
09-BF017-FC-01	77.8	0.087	7.94	15.1	0.20	1.60	3.8	774	0.046	12.8	0.664	2.76	71.6
09-BF019-FC-01	77.7	0.089	8.04	7.99	0.12	1.19	3.2	675	0.051	7.4	0.610	2.83	71.2
09-BF021-FC-01	79.4	0.11	7.24	10.0	0.25	1.79	3.4	1040	0.034	13.1	0.555	3.05	77.2
09-BF023-FC-01	79.7	0.092	8.08	3.85	0.23	1.20	4.1	386	0.042	9.1	0.759	3.59	83.8
09-BF025-FC-01	81.2	0.069	7.39	2.35	0.25	0.53	4.5	124	0.023	9.3	0.520	3.39	91.3
<b>Klondike Stations</b>													
09-KF001-FC-01	80.2	0.091	10.9	1.66	0.91	0.30	4.4	153	0.041	8.2	2.29	3.42	87.5
09-KF003-FC-01	81.0	0.081	7.44	4.59	0.58	0.67	4.2	344	0.026	8.4	1.51	3.05	83.7
09-KF005-FC-01	79.0	0.10	10.0	3.22	0.46	0.39	5.1	207	0.037	7.3	1.75	3.04	86.0
09-KF007-FC-01	80.1	0.064	8.82	2.60	0.82	0.38	4.8	217	0.049	8.0	1.53	3.42	92.3
09-KF009-FC-01**	80.1	0.089	9.10	4.44	1.02	0.71	4.4	472	0.033	12.8	1.34	3.52	94.7
09-KF009-FC-02**	81.2	0.12	7.92	3.78	1.09	0.61	4.7	365	0.031	9.9	1.34	3.27	93.4
09-KF011-FC-01	80.0	0.16	11.5	1.28	0.52	0.25	4.0	131	0.036	7.5	0.557	3.60	104
09-KF013-FC-01*	81.2	0.12	10.0	2.10	0.87	0.40	4.1	216	0.036	6.6	0.740	3.51	89.9
09-KF013-FC-01*	81.1	0.12	10.0	2.33	0.87	0.40	4.2	209	0.034	6.4	0.688	3.55	89.1
09-KF015-FC-01	79.5	0.074	10.7	2.77	0.44	0.44	4.2	241	0.043	10.8	0.995	3.21	97.9
09-KF017-FC-01	79.0	0.096	14.4	1.76	0.34	0.26	4.1	144	0.040	6.0	1.20	3.44	99.8
09-KF019-FC-01	82.6	0.16	7.10	2.00	1.83	0.45	5.4	187	0.026	9.4	0.607	3.11	101
09-KF023-FC-01	79.6	0.14	8.96	4.29	0.52	0.79	4.0	218	0.041	7.7	1.13	3.20	81.4
09-KF025-FC-01	77.3	0.18	7.78	7.99	0.38	1.43	4.2	588	0.073	12.0	1.12	3.10	85.5

\*Laboratory duplicate.

\*\*Field duplicate.



Summary statistics for trace metals (dry weight) and water content in fish tissue samples. Values for lab and field duplicates were averaged prior to statistical analysis.

Sample Identification	Statistic	Water Content (%)	Ag (µg/g)	As (µg/g)	Ba (µg/g)	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Fe (µg/g)	Hg (µg/g)	Mn (µg/g)	Pb (µg/g)	Se (µg/g)	Zn (µg/g)
Burger Fixed Stations	Mean	79.1	0.099	9.19	12.2	0.24	1.35	4.0	686	0.045	11.1	0.644	2.97	80.5
	Std. Dev.	1.3	0.028	1.90	12.0	0.06	0.38	0.6	312	0.013	2.5	0.149	0.28	5.7
	n	12	12	12	12	12	12	12	12	12	12	12	12	12
	Maximum	81.3	0.14	13.3	48.4	0.38	2.08	4.9	1130	0.072	16.7	0.982	3.59	91.3
	Minimum	77.0	0.060	7.24	2.35	0.12	0.53	2.9	124	0.023	7.4	0.402	2.72	71.2
Klondike Fixed Stations	Mean	80.0	0.114	9.68	3.21	0.73	0.54	4.4	255	0.040	8.6	1.23	3.29	91.9
	Std. Dev.	1.3	0.038	2.05	1.86	0.42	0.33	0.5	133	0.012	1.9	0.50	0.20	7.5
	n	12	12	12	12	12	12	12	12	12	12	12	12	12
	Maximum	82.6	0.18	14.4	7.99	1.83	1.43	5.4	588	0.073	12.0	2.29	3.60	104
	Minimum	77.3	0.064	7.10	1.28	0.34	0.25	4.0	131	0.026	6.0	0.557	3.04	81.4
Cumulative for Burger and Klondike stations	Mean	79.5	0.107	9.43	7.73	0.48	0.94	4.2	470	0.043	9.8	0.936	3.13	86.2
	Std. Dev.	1.4	0.033	1.95	9.59	0.38	0.54	0.6	322	0.013	2.5	0.469	0.29	8.7
	RSD (%)*	1.8	31	21	124	79	57	14	68	30	26	50	9	10
	n	24	24	24	24	24	24	24	24	24	24	24	24	24
	Maximum	82.6	0.18	14.4	48.4	1.83	2.08	5.4	1130	0.073	16.7	2.29	3.60	104
Minimum	77.0	0.060	7.10	1.28	0.12	0.25	2.9	124	0.023	6.0	0.402	2.72	71.2	

\* RSD = Relative Standard Deviation = (Standard deviation/mean) x 100%.

Results for reference materials analyzed during this study. Reference materials included Dogfish muscle (DORM-2), certified by the National Research Council (NRC) of Canada and Oyster Tissue #1566, certified by the U.S. National Institute of Standards and Technology (NIST).

Reference Material	Ag (µg/g)	As (µg/g)	Ba (µg/g)	Cd (µg/g)	Cr (µg/g)	Cu (µg/g)	Fe (µg/g)	Hg (µg/g)	Mn (µg/g)	Pb (µg/g)	Se (µg/g)	Zn (µg/g)
CRM DORM-2 (This Study)	0.035	18.4	2.64	0.037	31.4	2.2	141	4.64	3.6	0.065	1.34	26.5
CRM DORM-2 (NRC Certified Values)	0.041 ±0.013	18.0 ± 1.1	- -	0.043 ±0.008	34.7 ± 5.5	2.34 ± 0.16	142 ± 10	4.64 ± 0.26	3.66 ± 0.34	0.065 ± 0.007	1.40 ± 0.09	25.6 ± 2.3
SRM #1566b (This Study)	0.658	7.65	8.51	2.42	0.33	70.4	200	0.037	18.3	0.301	1.93	1435
SRM #1566b (NIST Certified Values)	0.666 ±0.009	7.65 ± 0.65	8.6* ± 0.3	2.48 ± 0.08	- -	71.6 ± 1.6	205.8 ± 6.8	0.0371 ± 0.0013	18.5 ± 0.2	0.308 ± 0.009	2.06 ± 0.15	1424 ± 46
SRM #1640 (This Study)	-	-	(µg/L) 146.5	-	(µg/L) 38.1	-	-	-	-	-	-	-
SRM #1640 (NIST Certified Values)	-	-	148.0 ± 2.2	-	38.6 ± 1.6	-	-	-	-	-	-	-
Analytical Precision from two sets of lab replicates**	0.0 0.0	1.4 0.0	1.1 7.3	2.7 0.0	1.5 0.0	0.0 1.7	0.7 2.3	2.5 4.0	0.7 2.2	1.5 5.1	1.3 0.8	0.7 0.6

\* Reference Value, not a Certified Value.

\*Precision presented as % relative standard deviation = (SD/mean) x 100%.

## **Appendix C**

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### **Chains-of-Custody**

Aldrich Offshore Services, AOS

P.O. Box 568  
Seward, AK

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PAGE 1 of 3  
in 3 COOLERS

Cooler #1

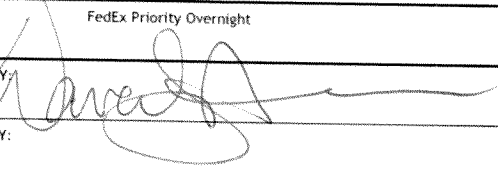
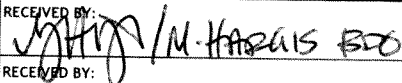
AOS Sampling Contact:
W. Thorsen
SHIP TO: BATTELLE C/O Jeannine Seyfert 397 Washington St. Duxbury, MA 02332
ATTN: Jeannine Seyfert 781.934.0571

CHAIN OF CUSTODY RECORD/SAMPLE ANALYSIS REQUEST FORM

PROJECT NAME: CHUKCHI SEA ENVIRONMENTAL STUDIES BASELINE PROGRAM  
CRUISE ID: WWW0904

SAMPLERS: WAT

STATION ID	DATE	TIME (24 HR)	Unique Sample ID	Preservative	Analysis required	# Cont	Container Size (mL)/Type	REMARKS/HANDLING REQ
09-04-KF001-FC-01	9/26/2009	7:15	78206	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	Please contact John Brown at 978.461.1221 for analysis questions. Aliquots for metals analysis should be sent to John Trefry at FIT.
09-04-KF001-FC-01	9/26/2009	7:15	78205	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF003-FC-01	9/27/2009	7:00	78214	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	Aliquots for metals analysis should be sent to John Trefry at FIT.
09-04-KF003-FC-01	9/27/2009	7:00	78213	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF005-FC-01	9/29/2009	2:45	78232	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF007-FC-01	9/27/2009	3:14	78211	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF007-FC-01	9/27/2009	3:14	78212	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF009-FC-01	9/28/2009	21:20	78223	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF009-FC-01	9/28/2009	21:20	78224	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF011-FC-01	9/26/2009	2:30	78203	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF011-FC-01	9/26/2009	2:30	78204	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF013-FC-01	9/28/2009	2:30	78218	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF013-FC-01	9/28/2009	2:30	78219	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF013-FC-01	9/28/2009	2:30	78222	Frozen	Metals (FIT); Hydrocarbons	1	1 Qt. Ziplock Glass Jar	
09-04-KF015-FC-01	9/29/2009	6:00	78233	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF015-FC-01	9/29/2009	6:00	78234	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF017-FC-01	9/26/2009	23:00	78209	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF017-FC-01	9/26/2009	23:00	78210	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF019-FC-01	9/28/2009	6:00	78221	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF019-FC-01	9/28/2009	6:00	78220	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF023-FC-01	9/27/2009	22:40	78216	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF023-FC-01	9/27/2009	22:40	78217	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF025-FC-01	9/29/2009	21:26	78235	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF025-FC-01	9/29/2009	21:26	78236	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	

Shipped Via: FedEx Priority Overnight	Condition of Samples	Custody Seal Intact:
Tracking No:	Upon Receipt: <b>INTACT</b>	Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
RELINQUISHED BY: 	DATE/TIME: 10/10/09 0814	RECEIVED BY:  M. HARRIS BOO
RELINQUISHED BY:	DATE/TIME:	RECEIVED BY:
RELINQUISHED BY:	DATE/TIME:	RECEIVED BY:

Aldrich Offshore Services, AOS  
P.O. Box 568  
Seward, AK

AOS Sampling Contact:  
W. Thorsen  
SHIP TO: BATTELLE C/O Jeannine Seyfert  
397 Washington St.  
Duxbury, MA 02332  
ATTN: Jeannine Seyfert  
781.934.0571

CHAIN OF CUSTODY RECORD/SAMPLE ANALYSIS REQUEST FORM

PROJECT NAME: CHUKCHI SEA ENVIRONMENTAL STUDIES BASELINE PROGRAM  
CRUISE ID: WWW0904  
SAMPLERS: WAT

STATION ID	DATE	TIME (24 HR)	Unique Sample ID	Preservative	Analysis required	# Cont	Container Size (mL)/Type	REMARKS/HANDLING REQ
09-04-KR045-FC-01	9/30/2009	3:40	78237	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	Please contact John Brown at 978.461.1221 for analysis questions. Aliquots for metals analysis should be sent to John Trefry at FIT.
09-04-KR045-FC-01	9/30/2009	3:40	78238	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KR056-FC-01	9/30/2009	4:44	78240	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KR056-FC-01	9/30/2009	4:44	78239	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF066-FC-01	9/30/2009	6:00	78241	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF066-FC-01	9/30/2009	6:00	78242	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF009-FB-01	9/28/2009	21:20	78225	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF009-FB-01	9/28/2009	21:20	78226	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF005-EB1-01	9/29/2009	2:30	78229	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF005-EB2-01	9/29/2009	2:30	78230	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF017-EB-01	9/26/2009	22:30	78208	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF017-EB-01	9/26/2009	22:30	78207	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF009-FC-02	9/28/2009	21:20	78227	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-KF009-FC-02	9/28/2009	21:20	78228	Frozen	Metals (FIT); Hydrocarbons	1	125 Glass Jar	
09-04-KF023-EB-01	9/27/2009	23:30	78215	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	

Shipped Via: FedEx Priority Overnight	Condition of Samples Upon Receipt: <u>INTACT</u>	Custody Seal Intact: Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
Tracking No:	RECEIVED BY: <u>M. HARRIS BDO</u>	DATE/TIME: <u>1/27/10 0845</u>
RELINQUISHED BY: <u>[Signature]</u>	DATE/TIME: <u>10/10/09 0827</u>	RECEIVED BY: <u>[Signature]</u>
RELINQUISHED BY:	DATE/TIME:	DATE/TIME:

**Aldrich Offshore Services, AOS**

P.O. Box 568  
Seward, AK

PAGE 3 of 3

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*in cooler #3*

SHIP TO:

AOS Sampling Contact:
W. Thorsen
BATTELLE C/O Jeannine Seyfert 397 Washington St. Duxbury, MA 02332
ATTN: Jeannine Seyfert 781.934.0571

**CHAIN OF CUSTODY RECORD/SAMPLE ANALYSIS REQUEST FORM**

PROJECT NAME: CHUKCHI SEA ENVIRONMENTAL STUDIES BASELINE PROGRAM  
CRUISE ID: WWW0904

SAMPLERS: WAT

STATION ID	DATE	TIME (24 HR)	Unique Sample ID	Preservative	Analysis required	# Cont	Container Size (mL)/Type	REMARKS/HANDLING REQ
09-04-BF001-FC-01	10/1/2009	22:00	78248 <i>Q8744</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	Please contact John Brown at 978.461.1221 for analysis questions. Aliquots for metals analysis should be sent to John Trefry at FIT.
09-04-BF003-FC-01	10/10/2009	1:40	78104 <i>Q8745</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF003-FC-01	10/10/2009	1:40	78107 <i>Q8774</i>	Frozen	Metals (FIT); Hydrocarbons	1	1 Qt. Ziplock Glass Jar	
09-04-BF007-FC-01	10/6/2009	1:09	78101 <i>Q8746</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF009-FC-01	10/9/2009	23:00	78106 <i>Q8747</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF011-FC-01	10/1/2009	6:00	78246 <i>Q8748</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF013-FC-01	10/6/2009	8:40	78103 <i>Q8749</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF015-FC-01	10/7/2009	6:00	78256 <i>Q8750</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF017-FC-01	10/6/2009	4:44	78102 <i>Q8751</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF019-FC-01	10/7/2009	1:40	78254 <i>Q8752</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF019-FC-01	10/7/2009	1:40	78255 <i>Q8775</i>	Frozen	Metals (FIT); Hydrocarbons	1	1 Qt. Ziplock Glass Jar	
09-04-BF021-FC-01	10/1/2009	2:44	78244 <i>Q8753</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF023-FC-01	10/6/2009	21:00	78251 <i>Q8754</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF025-FC-01	10/7/2009	10:00	78257 <i>Q8755</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF023-FB-01	10/6/2009	21:00	78250 <i>Q8756</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF023-EB1-01	10/6/2009	21:00	78252 <i>Q8757</i>	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	
09-04-BF023-EB1-01	10/6/2009	21:00	78253	Frozen	Metals (FIT); Hydrocarbons	1	250 Glass Jar	

Shipped Via: FedEx Priority Overnight	Condition of Samples: <i>INTACT</i>	Custody Seal Intact: Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
Tracking No: <i>[Signature]</i>	Upon Receipt: <i>INTACT</i>	
RELINQUISHED BY: <i>[Signature]</i>	DATE/TIME: <i>10/10/09 0836</i>	RECEIVED BY: <i>[Signature]</i>
RELINQUISHED BY:	DATE/TIME:	DATE/TIME: <i>1/27/10 0845</i>