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Environmental Effects-Based Concentrations for Total Petroleum Hydrocarbons (TPH)

Toxicity in Marine Water and Freshwater

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Environmental Effects-Based Concentrations for Total Petroleum Hydrocarbons (TPH)

Toxicity in Marine Water and Freshwater

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Abstract

The Washington State Department of Ecology (Ecology) Toxics Cleanup Program (TCP) is responsible for identifying and remediating sites impacted by hazardous substances. Using Washington's Model Toxics Control Act authority, Ecology undertakes cleanup of contaminated sites. At many contaminated sites, Ecology needs to establish surface water concentrations for petroleum contaminants that are protective of aquatic life in both the marine water and freshwater environment. Currently, there are no cleanup standards within state regulations that are based on dose-response relationships or effects-based concentrations. The goal of this study is to determine petroleum concentrations that are protective of marine and freshwater organisms.

Chronic exposure toxicity testing was carried out on one fish species and one invertebrate species that represent both marine and freshwater aquatic ecosystems. The test organisms included marine: topsmelt (*Atherinops affinis*) and sea urchin (*Strongylocentrotus purpuratus*); and freshwater: fathead minnow (*Pimephales promelas*) and daphnia (*Ceriodaphnia dubia*). Organisms were exposed to concentrations of total petroleum hydrocarbons (referred to as *Northwest TPH* or *NWTPH* after the lab method). Separate tests were carried out using the gasoline fraction (NWTPH-Gx) and the diesel fraction (NWTPH-Dx). A series of mixing experiments and range-finding toxicity tests were conducted before final testing. A final dilution series of six concentrations was used to establish a dose-response relationship for NWTPH with each organism. Clear lethal and sublethal effects were observed for each organism.

The lowest-observed effect concentrations (LOEC) established during this study were:

- NWTPH-Gx: >1.7 mg/L in marine water and 2.1 mg/L in freshwater
- NWTPH-Dx: 0.05 mg/L in marine water and 0.22 mg/L in freshwater.

The no-observed effect concentrations (NOEC), which are relevant to TCP in guidance on surface water cleanup standards, are:

- NWTPH-Gx: 1.7 mg/L in marine water and 1.0 mg/L in freshwater
- NWTPH-Dx: <0.05 mg/L (the limit of analytical reporting) in marine water and 0.15 mg/L in freshwater.

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TCP: Toxics Cleanup Program
HQ: Headquarters
SWRO: Southwest Regional Office
NWRO: Northwest Regional Office
WQP: Water Quality Program
EAP: Environmental Assessment Program

Introduction

The Washington State Department of Ecology (Ecology) Toxics Cleanup Program (TCP) is responsible for identifying and remediating sites impacted by hazardous substances. Using Washington's Model Toxics Control Act (MTCA; WAC 173-340) authority, Ecology sometimes undertakes cleanup of contaminated sites for the federal Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) program. TCP has identified a number of tasks for fiscal years 2017 and 2018 to improve the program's ability to participate in the CERCLA response program. One of the main tasks is the development of standards for aquatic organisms.

At many contaminated sites, Ecology must establish surface water concentrations for petroleum contaminants that are protective of aquatic life in both the marine (salt) water and freshwater environment. Petroleum hydrocarbon contamination in waters of the state is often broadly classified using the analytical methodology for total petroleum hydrocarbon concentrations (TPH; Ecology, 1997). The approach to evaluate TPH includes two methods: NWTPH – gasoline range organics (Gx) and NWTPH – diesel range organics (Dx)[1]. Currently, there are no environmental effects-based concentrations under state or federal regulations for these TPH fractions.

The goal of this study was to use a laboratory-based toxicity test dilution series for NWTPH-Dx and -Gx to determine the no-observed effect concentration (NOEC) and lowest-observed effect concentration (LOEC) for two marine and two freshwater organisms. These effects levels would be directly applicable to whole effluent testing (WET) that is carried out under WAC 173-205. WET refers to the aggregate toxicity of pollutants contained in wastewater effluent. It represents the total exposure of aquatic life to pollutants in a controlled lab environment. Once the effects levels have been established TCP's Policy and Technical Support Unit will then write an implementation memorandum, recommending protective values under WAC-173-340-730(3)(b)(ii) (Environmental effects) – Surface Water Cleanup Standards.

Washington's WAC 173-205, section 050, states that effluent samples must be tested using multiple species, including at a minimum one fish and one invertebrate. Therefore, the toxicity tests in this study were carried out using both a marine and freshwater fish and an invertebrate that have demonstrated sensitivity to hydrocarbons. The organisms included:

Marine water

- Topsmelt (*Atherinops affinis*) – EPA/600/R-95/136, method 1006.0
- Sea urchin (*Strongylocentrotus purpuratus*) – EPA/600/R-95/136

Freshwater

- Fathead minnow (*Pimephales promelas*) – EPA-821-R-02-013, method 1000.0
- Daphnia (*Ceriodaphnia dubia*) – EPA-821-R-02-013, method 1002.0

[1] NWTPH: Northwest total petroleum hydrocarbons, where NWTPH-Gx is in the carbon range C7-C12 and NWTPH-Dx is in the range C10-C24.

Hydrocarbon Composition and Toxicity

The fractions of petroleum hydrocarbons that are of interest in this study are broadly defined as a volatile petroleum hydrocarbon (NWTPH-Gx) and a semi-volatile petroleum hydrocarbons (NWTPH-Dx). Analytically, these two fractions are operationally defined by the extraction methods (Ecology, 1997) and weight of carbon compounds within the fraction (Table 1). In the environment, the carbon ranges within the diesel and gasoline fraction can include a number of products (Table 1). The methods published by Ecology (1997) detailing the quantification of NWTPH-Dx and -Gx also include the chromatograms for each of the products listed in Table 1.

Table 1: Summary of NWTPH fractions.

NWTPH-Gx (C7-C12)	NWTPH-Dx (C10-C24)
Gasoline	#2 Diesel Oil
Weathered gasoline	#2 Diesel Oil/Motor Oil
Naphtha	#2 Fuel Oil (38% Aromatic)
Mineral spirits #1, #2, and #3	Kerosene (Deodorized)
	Jet Fuel A
	Bunker C #1 and #2
	Motor Oil 30 weight
	Hydraulic Oil (USP)
	Transformer Oil
	Gas Oil

Within the gasoline fraction, monocyclic aromatic hydrocarbons (MAHs) are thought to contribute significantly to aquatic toxicity because they are relatively water-soluble in comparison to other petroleum hydrocarbons (McGrath and Di Toro, 2009). MAHs contain one benzene ring and are comprised mainly of benzene, toluene, ethylbenzene, and three xylene isomers (collectively referred to as BTEX). The BTEX concentrations of the toxicity test chambers were also assessed during this project.

The diesel fraction is a more complex mixture of hydrocarbons. Many of the polycyclic aromatic hydrocarbons (PAHs) are present in the diesel fraction. Aquatic toxicity of individual PAHs have been investigated in the past, and there is a strong relationship between the partition coefficient of the compounds (K_{ow}) and the acute toxicity wherein lighter compounds are more acutely toxic (McGrath and Di Toro, 2009; Redman and Parkerton, 2015).

Regulatory Criteria or Standards

This study was designed to inform the use of Surface Water Cleanup Standards (WAC-173-340-730) under the MTCA. In particular, section 3(b)(ii) of this regulation pertains to the use of WET testing under the federal National Toxics Rule (40 C.F.R. Part 131) which states:

> ***(ii) Environmental effects.*** *For hazardous substances for which environmental effects-based concentrations have not been established under applicable state or federal laws,*

concentrations that are estimated to result in no adverse effects on the protection and propagation of wildlife, fish, and other aquatic life. Whole effluent toxicity testing using the protocols described in chapter ***173-205*** *WAC may be used to make this demonstration for fish and aquatic life.*

There are currently no numeric environmental-effects criteria or standards for surface water in Washington State for NWTPH-Dx and -Gx. The Washington State Water Quality Standards (WAC 173- 201A), section 260(2)(b) under the Clean Water Act (CWA), state that the "Aesthetic values must not be impaired by the presence of materials or their effects, excluding those of natural origin, which offend the senses of sight, smell, touch, or taste...". This would include oily sheens from hydrocarbon contamination. It is important to note that this project is intended to inform surface water quality under the MTCA (i.e. applies within a designated site) and not under the CWA, which applies more broadly to waters of the state.

Ecology's Toxics Cleanup Program (TCP) has guidance on the remediation of petroleum-contaminated sites (Ecology, 2016). Part of remediating a contaminated site is establishing a level or concentration of petroleum hydrocarbons as a "cleanup standard." For waters of the state, WAC 173-340-730 (3)(b)(iii)(C) states that Method A groundwater TPH cleanup levels may be used as surface water Method B (site-specific) petroleum cleanup levels protective of human health. The cleanup levels for diesel-range organics by NWTPH-Dx and gasoline-range organics by NWTPH-Gx are shown in Table 2.

Site-specific surface water Method B petroleum concentrations can be derived using Equations 730-1 and 730-2 (WAC 173-340-730 pg. 164). However, if the calculated protective value is lower than the PQL for the specific contaminant the concentration would then default to the practical quantitation limits (PQLs) for the NWTPH-Dx and -Gx (Table 2). The goal of this study was to assist TCP by providing reliable effects-based concentrations for NWTPH-Gx and –Dx to refine site-specific cleanup standards.

Table 2: Cleanup concentrations for petroleum hydrocarbons in surface waters (Ecology, 2016).

Petroleum Hydrocarbons	Method A†	Method B (site-specific)*
NWTPH-Dx	250 µg/L	250 µg/L
NWTPH-Gx (benzene present)	800 µg/L	250 µg/L
NWTPH-Gx (no detectable benzene)	1000 µg/L	250 µg/L

† Table 720-1 WAC 173-340-730.

* The lowest concentration of either equation 730-1 or 730-2 (WAC 173-340-730 pg. 164) or the practical quantitation limits listed in Table 2.

Methods

Detailed descriptions of the methods used and associated quality objectives can be found in Hobbs (2017) and Marshall (2016). The toxicity tests were carried out by Nautilus Environmental (Nautilus; Burnaby, BC). All water chemistry samples were taken by Nautilus and shipped to Ecology's Manchester Environmental Laboratory (MEL) for analysis. Further details on the toxicity tests by Nautilus can be found in dedicated reports for NWTPH-Gx (Appendix A) and NWTPH-Dx (Appendix B).

Water Chemistry

Certified reference materials from an independent provider (Restek) were used for both Unleaded gasoline (NWTPG-Gx) and Diesel #2 (NWTPH-Dx) (Appendix C). The reference materials were composed of a blend from multiple refineries. These reference materials were used for both the calibration of analytical equipment at MEL and the dilution series for the toxicity tests at Nautilus.

Stock solutions for both Gx (10 mg/L) and Dx (50mg/L) were used to prepare the test-chamber dilutions. The different dilutions for the test chambers were then made from the concentrated stock solution. This approach is commonly called a serial dilution (Aurand and Coelho, 2005). Five liters of stock solution were mixed in one aspirator bottle for each test species in respective control water (salt water or freshwater). Aspirator bottles were capped with rubber stoppers to avoid losses, stirred overnight, and allowed to settle for 1 hour prior to preparing the dilution series. The length of mixing is to ensure the hydrocarbons used in the test chambers are dissolved in solution, otherwise known as the water-accommodated fraction (WAF; Redman and Parkerton, 2015). Aspirator bottles were drained via a port at the bottom to avoid undissolved fractions.

All test chambers had a daily water renewal schedule, as per EPA methods for toxicity testing, and water samples for chemistry were taken at the time of renewal to establish the measured concentrations of Gx and Dx in the test chambers. Samples for Gx were collected directly into 40ml vials with no headspace, while Dx samples were collected in amber glass jars. In addition, some test chambers were sampled at the end of the daily test period, just prior to renewal, to establish the concentrations of the stale solutions. Stale water from the test chamber replicates were composited into one water sample.

The laboratory methods for the water chemistry are described in Table 3. An additional objective of this study was to test whether there is a significant difference in the results of NWTPH-Dx when using a silica gel cleanup in the sample preparation methods. A subset of NWTPH-Dx samples from the stale test chambers were split, one going through cleanup while the other did not.

Table 3: Laboratory methods and reporting limits.

Analyte	Expected Reporting Limit (µg/L)	Actual Reporting Limits (µg/L)	Sample Preparation Method	Analytical Method
NWTPH-Dx	150	50	SCP; EPA SW 3535A	NWTPH-Dx
NWTPH-Gx	70	70	EPA SW 5030B	NWTPH-Gx
benzene	1.0	1.0	SW5030B	SW8021B
toluene	1.0	1.0	SW5030B	SW8021B
ethylbenzene	1.0	1.0	SW5030B	SW8021B
xylenes	1.0 for each isomer	1.0	SW5030B	SW8021B

SCP: silica gel cleanup

Toxicity Testing

The project consisted of four chronic toxicity tests (Table 4) in both marine water and freshwater, based on a dilution series using a stock solution mixed from hydrocarbon standards. The hydrocarbon standards were supplied in methanol (Gx) and acetone (Dx), which also meant that the negative control test chambers in the toxicity tests needed to match the highest % methanol or acetone in the hydrocarbon solutions. The methanol and acetone controls were then used to calculate test endpoints. These controls were in addition to laboratory controls for clean test waters and the reference toxicant tests confirming the sensitivity of the organisms.

Table 4: Description of chronic toxicity test methods.

Test Organism and EPA Method	Test Type	Chamber Size (Minimum)	Solution Volume	# Organisms Per Chamber	# Replicates (Minimum)	Age	Temperature	Aeration	Feeding	Endpoints
Ceriodaphnia dubia EPA-821-R-02-013, method 1002.0	7-day static renewal (80% renewal daily)	30 mL	15 mL	1 from a female with ≥ 8 neonates in the 3rd or subsequent broods	10	< 24 hrs and within an 8-hr age range	25° ± 1°C	if DO < 2.0 mg/L	0.1 mL YCT and 0.1 mL algal suspension daily	Number of survivors at 7 days and number of neonates per female at 3 broods.
Pimephales promelas EPA-821-R-02-013, method 1000.0	7-day static renewal (80% renewal daily)	500 mL	250 mL	minimum 10	4	< 24 hrs (< 48 hrs if shipped)	25° ± 1°C	if DO < 4.0 mg/L	0.1 g wet weight per container 3 times daily at 4-hour intervals or 0.15 g wet weight per container twice daily at 6-hour intervals: no food in final 12 hours	Survival rate; Total weight of survivors divided by the initial count (biomass); Total weight of survivors divided by the final count (weight).
Atherinops affinis EPA/600/R-95/136, method 1006.0	7-day static renewal (80% renewal daily)	600 mL	200 mL	minimum 5	5	9 - 15 days post-hatch	20° ± 1°C	if DO < 4.0 mg/L	Twice daily (40 Artemia nauplii/fish at each feeding) morning and afternoon; no food on day 7	Survival rate; Total weight of survivors divided by the initial count (biomass); Total weight of survivors divided by the final count (weight).
Strongylocen-trotus purpuratus EPA/600/R-95/136	24-hr static	20 mL	5 mL	about 5 X 10^7 sperm/mL and about 2000 eggs/mL	4	< 4 hrs after collection of gametes	20° ± 1°C	if DO < 4.0 mg/L	NA	Fertilization of eggs.

DO: dissolved oxygen

YCT: yeast-cerophyl-trout mixture

Before the toxicity testing, Nautilus tested the mixing protocol and submitted triplicate samples to MEL from different dilutions in order to look at accuracy and precision of the test solutions compared with the nominal concentrations. Four nominal concentrations were analyzed for NWTPH-Gx (0, 1, 56, and 200 mg/L), and five nominal concentrations were analyzed for NWTPH-Dx (0, 0.1, 1, 10, and 100mg/L). In addition, a series of range-finding tests were completed prior to the definitive or final chronic toxicity tests to establish a realistic sensitivity of the organism. For NWTPH-Gx, the range-finding nominal concentrations were 1, 10, and 100 mg/L; for NWTPH-Dx the range-finding nominal concentrations were 0.5, 5, and 50 mg/L.

Nominal concentrations for the NWTPH-Gx and -Dx tests should be viewed as a rough guide for the dilution series, because measured concentrations of the WAF were a fraction of this desired nominal concentration. For instance, the initial mixing of the diesel standard into the stock solutions proved difficult to get a WAF that represented greater than ~10% of the desired nominal concentrations. All effects-based concentrations are based on the measured concentrations of the dilution series.

Based on the results from the range-finding tests, the definitive chronic tests for NWTPH-Gx used a nominal dilution series of 0.16, 0.31, 0.63, 1.25, 2.5, and 5.0 mg/L. Following the range-finding tests for NWTPH-Dx, it was decided that different dilution series would be mixed for the organisms. For the topsmelt and echinoderm tests (marine water), nominal concentrations of 0.5, 1.0, 2.0, 4.0, 8.0, and 16.0 mg/L were used, while for freshwater species (fathead minnows and daphnia), the nominal concentrations were 0.045, 0.09, 0.19, 0.38, 0.75, 1.5, 3.0, and 6.0 mg/L.

The organism test chambers used during the NWTPH-Gx tests were maintained with no headspace to decrease the loss of volatiles during the test, which can impact the exposure concentrations (Redman and Parkerton, 2015). The test chambers used for NWTPH-Dx had a consistent headspace, which would have been unlikely to impact the exposure concentrations because the heavier constituents in Dx would not partition to the air (i.e., volatilize).

All toxicity tests were carried out in dedicated climate-controlled rooms. All tests, with the exception of the echinoderm tests because of test duration, were conducted under full-spectrum lighting. All toxicity tests required monitoring for temperature, pH, dissolved oxygen, salinity (if applicable), and conductivity. The EPA methods describe the optimal conditions for these parameters, which were documented by Nautilus along with any deviations (Appendices A and B).

The general conditions of the bioassays met the following (as per Marshall, 2016):

- The approved chronic test manual is EPA-821-R-02-012 (USEPA, 2002).
- Dual endpoint tests must meet conditions in the chronic manual to have a valid chronic result.
- Illumination must be for 16 hours at 10 - 20 $\mu E/m^2/s$ (50 - 100 ft-c) followed by 8 hours of darkness.
- The performance criteria (survival, growth, and reproduction) of the control samples were met for all the bioassays conducted.

Numerical Methods

Following the final chronic toxicity testing on each organism using NWTPH-Gx and -Dx, a dose-response relationship can be established based on the biological endpoints of survival, growth, reproduction, and fertilization. From this dose-response relationship, effects concentrations (NOEC and LOEC) and point estimates of toxicity can be established. The point estimates calculated in this study include: lethal concentration where 50% of the organisms die (LC50), inhibitory concentration where the growth of 50% of the organisms is impeded (IC50), and inhibitory concentration where the growth of 25% of the organisms is impeded (IC25). All calculations were made using the software Comprehensive Environmental Toxicity Information System (CETIS^{TM}), Tidepool Scientific Software.

NOECs and LOECs are determined using hypothesis testing where significance tests are used to establish independence or a difference between the "effect" concentration and a control or the "effect" concentration and the "safe" concentration. The type of test used depends on the variability of the data and how independent the individual concentrations are in the dilution series. The methods used to establish LC50, IC50, and IC25 point estimates are different. These estimates are derived from a model of the dose-response relationship established during each chronic toxicity test.

The fundamental difference between the NOEC and LOEC and the point estimates is the effects concentration is established directly from biological endpoints, whereas the point estimates are estimated from the dose-response relationships for the biological endpoints (survival and growth). For point estimates, the model fit is relatively straightforward and is judged based on the ability to describe the dose-response relationship using the simplest model. The effects concentrations are first dependent on whether the data have homogenous or heterogeneous variance by Bartlett's test. Then, depending on the number of replicates, a test of independence is used (Table 5). The decisions on the appropriate statistical tests are built into the CETIS software and largely follow USEPA guidance (2002). Each of the tests used in this study are described in Table 5.

Table 5: Statistical tests used during this study and the appropriate use.

Statistical test	Use
Point estimates (modeling data)	
Nonlinear regression	An interpolation method from a nonlinear regression (e.g., exponential function).
Spearman-Kärber	A nonparametric method for estimating the LC50.
Linear interpolation	Simple linear interpolation from a linear regression.
NOEC/LOEC (significance tests of independence between test populations)	
Steel Many-one Rank Sum test	Used when the data have heterogeneous variance and an equal number of replicates.
Dunnett multiple comparison	Used when the data has homogenous variance and an equal number of replicates.
Fisher Exact/Bonferroni-Holm test	Used when the data has homogenous variance and an unequal number of replicates. The Bonferroni-Holm correction is used when making multiple comparisons and reduces the chances of a Type 1 error (false positive).

Data Quality

Blanks

All lab blanks analyzed were below the method detection limit. All clean test waters were submitted as blanks during the mixing, range-finding, and final chronic testing phases of the project. All test blanks were below method detection limits (Appendices D and E).

Precision

Precision is a measure of variability between results of replicate measurements that is due to random error. Precision is measured using the relative percent difference (RPD) between replicate samples. Laboratory replicate precision for the lab control standard and duplicates did not meet the project measurement quality objectives (MQOs; <40% RPD) for two out of eleven laboratory control standard duplicates for NWTPH-Dx (Appendix E, Table E-4). This was due to low recoveries of the spikes and attributed to a problem with the extraction of the sample. As a result of this, all samples associated with the batch QC (MEL# 1707056) were qualified as estimates ("J" qualifier). These data are useable and did not impact our ability to quantify the effects-based concentration for the entire 7-day chronic test. The remaining lab replicates had a median RPD of 7%. All laboratory replicates for NWTPH-Gx and BTEX were within the project MQOs (<40% for Gx and <50% for BTEX). The median RPD between NWTPH-Gx lab replicates was 4%.

Replicate samples collected during the toxicity tests for NWTPH-Dx were generally well below the project MQO (<40%). The exceptions were three samples from batches with high laboratory variability in matrix spike recoveries. These samples were qualified, as described earlier. No further corrective action was taken. Excluding the samples that did meet the project MQOs, the replicates for NWTPH-Dx had a median RPD of 5%. Replication of the NWTPH-Gx samples collected during the toxicity tests was excellent and were well within the MQOs for the project. Replicates had a median RPD of 1.6%, while BTEX parameters had RPDs of 1.2%, 4.7%, 1.6%, and 1.4% respectively.

Precision for the toxicity tests is measured and controlled through the use of reference toxicants. In comparison to an inter-laboratory study by the United States Environmental Protection Agency (USEPA, 2000), the coefficient of variations (CV) for precision around the toxicity tests met the median results and, in most cases, were lower than the 25th percentile (Table 6). The echinoderm (sea urchin) fertilization test had the lowest precision and was slightly above the median CV for the EPA study.

Table 6: Percentiles of the coefficient of variation (CV) for the reference toxicants (USEPA, 2000).

Test Organism	Method	EPA Percentiles			NWTPH-Gx		NWTPH-Dx	
		25th	50th (median)	75th	CV	Number of tests†	CV	Number of tests†
Fathead minnow larval survival	1000.0	0.26	0.39	0.48	0.15	118	0.16	124
Fathead minnow larval growth	1000.0	0.22	0.37	0.53	0.25	118	0.27	124
Ceriodaphnia survival	1002.0	0.21	0.30	0.43	0.05	160	0.05	161
Ceriodaphnia reproduction	1002.0	0.25	0.33	0.49	0.17	160	0.18	161
Topsmelt larval survival*	1010.0	0.42	0.42	0.42	0.26	27	0.25	30
Topsmelt larval growth*	1010.0	0.31	0.31	0.31	0.27	27	0.26	30
Echinoderm fertilization	EPA/600/ R-95/136	0.40	0.50	0.69	0.52	4	0.56	5

* One lab participated using this method.
†Number of tests refers to the history of tests run by Nautilus across multiple projects.

All toxicity tests required daily renewal of solutions and fresh mixtures. It is desirable to have the concentrations of the stock solutions remain consistent during the tests. The CV among the daily stock solutions for each test can be viewed as a measure of precision, which is affected by our ability to dissolve and dilute the chemical standard. In general the CV for Gx in the final chronic tests was 10 to 30%, with the greatest variability among the *Ceriodaphnia dubia* tests (Table 7). The CV for the Dx tests were higher, generally falling somewhere between 20 and 50%, with some higher values in the *Ceriodaphnia dubia* and topsmelt tests.

There is no defined threshold for assessing the CV among daily test solutions, rather the variability among these daily solutions reflects the reality of mixing and diluting the stock solutions combined with our ability to accurately measure the parameters (laboratory precision and bias). The number of samples that the CV among daily test solutions represents varies with the length of the test (in days). Generally, tests run with higher concentrations do not last as long, due to lethal effects, and therefore the number of samples is less (Table 7).

Table 7: Summary of the variability among daily test chamber solutions.
Differences in the number of samples is attributable to the duration (days) of the test. Replicate samples are also included in the total sample numbers (n).

Organism	Range-finding					Final chronic test				
	Nominal concentration (mg/L)	Measured concentration (mg/L)				Nominal concentration (mg/L)	Measured concentration (mg/L)			
		n	min	max	CV		n	min	max	CV
NWPTH-Gx										
Fathead minnow	1.0	5	0.59	0.89	0.16	0.16	7	0.07	0.16	0.31
	10.0	3	6.49	9.45	0.19	0.63	11	0.44	0.57	0.10
						2.5	6	1.69	2.18	0.09
Ceriodaphnia dubia						0.16	7	0.07	0.14	0.33
						0.63	12	0.07	0.52	0.46
						2.5	6	0.50	1.80	0.57
Topsmelt and Echinoderm	1.0	6	0.61	0.86	0.13	0.16	7	0.08	0.17	0.33
						0.31	12	0.17	0.32	0.22
						0.63	7	0.38	0.63	0.20
						2.5	7	1.10	2.16	0.23
NWTPH-Dx										
Fathead minnow	5.0	11	0.09	0.44	0.47	0.9	7	0.08	0.14	0.18
	50.0	6	0.73	24.0	1.52	1.9	7	0.12	0.21	0.21
						7.5	12	0.29	1.05	0.43
						30.0	7	1.75	3.68	0.27
Ceriodaphnia dubia	0.5	6	0.14	0.30	0.30	0.19	5	0.05	0.14	0.38
	5.0	7	0.39	0.95	0.34	0.38	7	0.08	0.24	0.37
						0.75	7	0.11	0.52	0.54
						1.5	4	0.29	1.22	0.71
Topsmelt and Echinoderm	5.0	13	0.07	0.39	0.39	0.5	7	0.05	0.14	0.43
						1.0	7	0.05	0.14	0.43
						4.0	12	0.07	0.4	0.37
						16.0	4	0.62	3.31	0.70

Bias

Bias is the difference between the sample mean and the true value. Laboratory bias was addressed by analyzing lab control samples, matrix spikes, and/or standard reference materials. The recoveries of the laboratory control samples and matrix spikes for NWTPH-Gx were all within the MQOs and had very little variability, generally being near 100% (Table 8). Likewise, most of the sample surrogate recoveries for NWTPH-Dx were all within MQOs, however as discussed earlier, the laboratory control samples had poor replication because of low recovery. Two samples had recovery of the laboratory control sample below the MQOs, which led to sample results being qualified as estimates.

Table 8: Laboratory recovery of sample surrogates and control samples.

Test	Sample surrogate recovery (%)						Laboratory control sample recovery (%)					
	n	median	mean	sd	min	max	n	median	mean	sd	min	max
NWTPH-Gx	117	102	102	4	92	124	20	100	99	4	88	105
BTEX	117	98	98	3	88	107	20	101-106	101-104	4-6	88-95	108-113
NWTPH-Dx	119	102	102	18	68	140	21	94	96	20	55	141
NWTPH-Dx (SCP)	26	98	98	13	78	121	na	na	na	na	na	na

SCP = silica gel cleanup; no additional laboratory control samples run with the NWTPH-Dx SCP, only method blanks.

Comparison of the stock solutions for the toxicity tests to the desired nominal concentrations was assessed prior to the tests beginning and is discussed in the *Results and Discussion* section.

Sensitivity

Sensitivity is a measure of the capability of a method to detect a substance. For each parameter, MEL was able to achieve the desired method detection limit (MDL) and reporting limit (RL) (Table 9). In the case of NWTPH-Dx, MEL was able to lower the RL by an order of magnitude and for NWTPH-Gx, MEL was able to confidently report at the MDL.

Table 9: Method detection and reporting limits for the study.

Parameter	Units	Desired MDL	Study MDL	Desired RL	Study RL
NWTPH-Dx	mg/L	NA	NA	0.15	0.05
NWTPH-Gx	mg/L	NA	NA	0.07	0.07
Benzene	µg/L	0.26	0.26	1.00	1.00
Ethylbenzene	µg/L	0.11	0.11	1.00	1.00
Toluene	µg/L	0.15	0.15	1.00	1.00
m,p-Xylenes	µg/L	0.24	0.24	2.00	2.00
o-Xylenes	µg/L	0.24	0.18	1.00	1.00

MDL=method detection limit; RL=reporting limit; NA = not applicable (the NWTPH method does not have MDLs).

The sensitivity of the toxicity tests is dependent on the number of replicates per concentration. The sensitivity is assessed by comparing against the control tests that are run concurrently. There is a recommended minimum significant difference (MSD) for each method (USEPA, 2000). The MSD is the smallest difference between the control and another test treatment that can be determined as statistically significant. The MSD is often expressed as the %MSD of the mean control value. In Washington State, WAC 173-205 defines a “Chronic statistical power standard” that represents the maximum %MSD of the test:control. The chronic statistical power standard is 39%, meaning the percent difference in a statistically significant response (i.e., %MSD) must be less than or equal to 39% to be acceptable. All final toxicity tests had an MSD below 39% (Table 10).

Table 10: The minimum significant difference (MSD) between the toxicity tests and controls.

In accordance with WAC-173-205 the MSD should be below 39%.

Organism	**NWTPH-Gx**				**NWTPH-Dx**			
	Lab Water Control		Solvent Control		Lab Water Control		Solvent Control	
	Survival	Growth	Survival	Growth	Survival	Growth	Survival	Growth
fathead minnow	11.6	14.8	10.6	16.7	NA	NA	11.0	17.3
topsmelt	28.1	28.7	30.3	30.3	NA	NA	21.4	31.2
	Survival	Reproduction	Survival	Reproduction	Survival	Reproduction	Survival	Reproduction
Ceriodaphnia dubia	NA	33.5	NA	26.9	NA	13.2	NA	22.6
		Fertilization		Fertilization		Fertilization		Fertilization
Echinoderm (purple sea urchin)		6.0		3.4		3.6		9.2

NA=not applicable due to no adverse effect on survival or the lab water control was not used.

Results and Discussion

Water Chemistry

Mixing and range-finding

The accuracy of the desired nominal concentrations for both NWTPH-Gx and -Dx was dependent on the solubility of the certified standard material (Unleaded gasoline and Diesel #2) and our ability to dissolve the standard into the stock solution. The initial mixing of the Gx stock solutions produced measured concentrations of NWTPH-Gx that were ~ 40% of the nominal concentrations (Table 11). While this was below the desired accuracy in the QAPP (Hobbs, 2017), the mixing method was precise as described by the CV among triplicate samples (Appendix D, Table D-1; 1 to 7%) and fairly consistent through the range of concentrations.

Table 11: Summary of nominal and measured concentrations of NWTPH-Gx during the mixing and range-finding tests.

Concentrations are mean ± standard deviation; range-finding concentrations are summarized over the 7-day test.

Nominal concentrations (mg/L)	Mixing		Range-finding			
	Marine (mg/L)	% of Nominal	Freshwater (mg/L)	% of Nominal	Marine (mg/L)	% of Nominal
200	88.70 ± 2.51	44%				
100			59.25 ± 7.99	59%	59.10*	59%
56	20.43 ± 1.39	36%				
10			8.50 ± 1.14	85%	7.51*	75%
1	0.41 ± 0.005	41%	0.74 ± 0.11	74%	0.77 ± 0.10	77%

* Based on one sample

During the range-finding tests, our WAF increased, and we were able to achieve concentrations that were ~60-85% of the nominal concentrations. In addition, there was also very little difference between the concentrations measured in marine water compared to freshwater for a specific nominal concentration (Table 11).

In the initial mixing of the diesel standard into the stock solutions, it proved difficult to get a WAF that represented greater than 10% of the desired nominal concentrations (Table 12). However, the variability among triplicate samples was low, suggesting a fairly homogenous mixture of dissolved Dx. We therefore made the decision to continue with the same mixing regime, knowing that we were getting precise measurements of the WAF.

Table 12: Summary of nominal and measured concentrations of NWTPH-Dx during the mixing and range-finding tests.

Concentrations are mean ± standard deviation; range-finding concentrations are summarized over the 7-day test.

Nominal concentrations (mg/L)	Mixing		Range-finding			
	Marine (mg/L)	% of Nominal	Freshwater (mg/L)	% of Nominal	Marine (mg/L)	% of Nominal
100	4.24 ± 0.15	4%				
50			7.37 ± 8.79	15%	1.64*	3%
10	0.28 ± 0.02	3%				
5			0.53 ± 0.58	11%	0.23 ± 0.10	5%
1	0.15 ± 0.00	15%				
0.5			0.15 ± 0.10	30%	0.09 ± 0.06	19%
0.1	0.05 (<MDL)	NA				

* Based on one sample

During the range-finding tests we achieved a marginally higher percent of the nominal concentration in the WAF. We were also able to dissolve more of the Dx into freshwater compared to marine water (Table 12). Following this result we established different dilution ranges for the final toxicity tests between freshwater and marine organisms.

Gasoline (Gx) fraction

During the final toxicity tests the measured concentrations were around 60 to 80% of the desired nominal concentrations in freshwater and around 80% in marine water (Table D-3). In order to establish the estimated concentrations for those solutions without measured NWTPH-Gx, we analyzed the relationship between nominal (explanatory variable) and measured (dependent variable) concentrations for each of the organisms (Figure 1). Linear regression was then used on untransformed data to determine the least squares regression line and equation, which was then used to interpolate and extrapolate estimated concentrations (Figure 1). Each of the linear regressions was statistically significant ($p<0.001$).

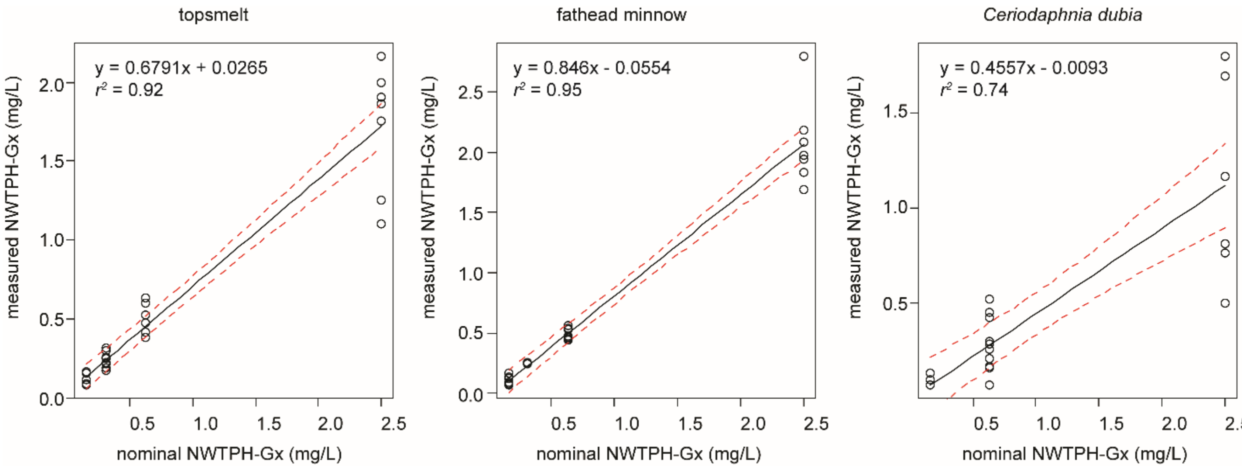

Figure 1: Linear regressions of measured vs. nominal NWTPH-Gx with 95% confidence limits.

Linear regression formulas were used for interpolation of results; r-squared is adjusted for Bonferroni correction

The Echinoderm fertilization test runs for 24 hours, and the measured concentrations for the dilution series were less than 10% relative percent difference of the nominal concentrations (Table 13). Therefore, the nominal concentrations were used as the final concentrations in the toxicity test.

Table 13: Measured and nominal NWTPH-Gx concentrations for the echinoderm (urchin) fertilization test.

Nominal concentration (mg/L)	Measured concentration (mg/L)	RPD (nominal/ measured)
	t=0	
2.5	2.31	8%
0.63	0.61	3%
0.31	0.32	4%
0.31	0.32	2%
0.16	0.16	1%

RPD = relative percent difference between measured and nominal concentrations

The final dilution series for the chronic tests using Gx was 5, 2.5, 1.25, 0.63, 0.31, and 0.16 mg/L for both freshwater and marine organisms (Tables 14 through 16). Concentrations in the test chamber solutions were measured at 2.5, 0.63 and 0.16 mg/L in freshwater and at 2.5, 0.63, 0.31 and 0.16 mg/L for marine water. All concentrations not measured in the dilution series were estimated using the relationships between nominal and measured. The mean of the measured concentrations in the fresh solutions were used as the final concentrations to calculate the toxicity endpoints.

Table 14: Measured and estimated NWTPH-Gx concentrations in fresh and stale solutions for topsmelt.

Final concentrations that were not measured were interpolated or extrapolated based on linear regression.

Nominal concentration (mg/L)	t=0	t=24		t=48	t=72	t=96		t=120	t=144	t=168	Final concentration (mg/L)
	fresh	stale	fresh	fresh	fresh	stale	fresh	fresh	fresh	stale	
5											***3.42***
2.5	1.86	0.635	1.75	2.16	1.1	0.208	1.25	2	1.9	0.061	1.72
1.25											***0.88***
0.63	0.522	0.171	0.416	0.384	0.472	0.07	0.417	0.599	0.633	0.07	0.45
0.31	0.253	0.077	0.188	0.174	0.220	0.07	0.188	0.297	0.314	0.07	0.24
0.16	0.119	0.055	0.082	0.082	0.092	0.07	0.083	0.159	0.167	0.07	0.14

Bold italic are estimated concentrations.

Table 15: Measured and estimated NWTPH-Gx concentrations in fresh and stale solutions for fathead minnow.

Final concentrations that were not measured were interpolated or extrapolated based on linear regression.

Nominal Concentration (mg/L)	t=0	t=24		t=48	t=72	t=96		t=120	t=144	t=168	Final Concentration (mg/L)
	fresh	stale	fresh	fresh	fresh	stale	fresh	fresh	fresh	stale	
5											***4.17***
2.5	1.97	18.1*	2.18	2.79	1.83	0.07	1.94	2.08	1.69	0.437	2.06
1.25											***1.00***
0.63	0.531	0.226	0.443	0.445	0.447	0.13	0.467	0.567	0.533	0.042	0.48
0.31	0.259										***0.21***
0.16	0.12	0.051	0.07	0.089	0.081	0.07	0.094	0.164	0.13	0.07	0.08

* Value is an outlier; re-analysis was beyond the hold time

Bold italic are estimated concentrations.

t = time in hours

Table 16: Measured and estimated NWTPH-Gx concentrations in fresh and stale solutions for *Ceriodaphnia dubia*.

Final concentrations that were not measured were interpolated or extrapolated based on linear regression.

Nominal Concentration (mg/L)	t=0	t=24		t=48	t=72	t=96		t=120	t=144	t=168	Final Concentration (mg/L)
	fresh	stale	fresh	fresh	fresh	stale	fresh	fresh	fresh	stale	
5											***2.27***
2.5	6.84*	0.252	1.17	0.817	0.504	0.343	0.769	1.8	1.7	1.19	1.13
1.25											***0.56***
0.63	0.07	0.07	0.209	0.26	0.158	0.091	0.284	0.524	0.425	0.526	0.28
0.31											***0.13***
0.16	0.07	0.07	0.066	0.065	0.04	0.036	0.1	0.135	0.132	0.126	0.06

* Value is an outlier; re-analysis was beyond the hold time; ***bold italic*** are estimated concentrations

We did not explicitly test the toxicity of BTEX compounds on the organisms, and the standards used for spiking did not have certified concentrations of BTEX. However, we did analyze the BTEX composition of the Gx solutions. Therefore, we can summarize the general range of BTEX present in the dilution series for NWTPH-Gx (Table 17). The complete dataset of BTEX compounds in the range-finding and final toxicity test solutions can be found in Appendix D.

Table 17: Composition of BTEX compounds in NWTPH-Gx dilution series.

Note the concentrations are in µg/L.

Nominal Concentration (µg/L)	NWTPH-Gx (µg/L)		Benzene (µg/L)		Ethylbenzene (µg/L)		Toluene (µg/L)		m.p-Xylenes (µg/L)		o-Xylenes (µg/L)	
	mean	sd	mean	sd	mean	sd	mean	sd	mean	sd	mean	sd
160	106.27	34.23	1.15	0.34	1.34	0.45	5.29	2.42	4.43	1.7	1.82	0.64
310	246.25	53.6	2.42	0.54	2.96	0.73	13.33	3.11	10.64	2.36	4.28	0.86
630	404.03	155.79	4.13	1.46	4.78	1.84	22.81	9.07	17.67	7.07	7.15	2.65
2500	1927.73	1227.87	19.28	6.37	25.94	19.25	111.18	48.64	92.19	71.2	38.35	30.15

Concentrations of the solutions following the daily exposure (stale solutions) were measured on day 1, 4 and 7 (Tables 13 through 15). This data provides information on the changes in NWTPH-Gx over the course of the daily exposure due to uptake by the organism or volatilization. In general, we found that the fish toxicity tests lost between ~70 and 80% of the Gx over the course of the daily exposure period in the fish chambers (Table 18). We also found that there was a comparable loss of the Gx among the different dilution concentrations. The zooplankton chambers had much smaller loss rates (~ 50%) compared with the fish chambers. The lower loss rates in the zooplankton chambers is perhaps due to the lower water volume and different containers.

The physical properties of the BTEX compounds would suggest that benzene would be lost faster from the water, based on lighter molecular weight and higher vapor pressure (MacKay et al., 1992). Over the short 24-hour exposure period we do not see much difference in loss among the BTEX compounds. Overall, the BTEX compounds showed comparable percent losses to the Gx solutions (Table 18).

Table 18: Percent loss from water of monoaromatic hydrocarbons during the daily toxicity test exposures.

Organism		NWTPH-Gx	Benzene	Ethylbenzene	Toluene	m.p-Xylenes	o-Xylenes
Topsmelt	median	0.80	0.78	0.79	0.92	0.82	0.80
	mean	0.79	0.77	0.80	0.86	0.81	0.79
	sd	0.12	0.14	0.14	0.13	0.14	0.15
Fathead minnow	median	0.72	0.67	0.67	0.83	0.74	0.70
	mean	0.74	0.68	0.68	0.79	0.76	0.70
	sd	0.15	0.20	0.18	0.17	0.16	0.19
Ceriodaphnia dubia	median	0.49	0.41	0.45	0.45	0.42	0.39
	mean	0.50	0.41	0.44	0.47	0.44	0.44
	sd	0.22	0.25	0.28	0.24	0.29	0.29

sd = standard deviation

Diesel (Dx) fraction

The amount of NWTPH-Dx dissolved in the stock solutions compared with our desired nominal concentrations was much lower during the final chronic toxicity test compared to the Gx tests. As described previously, this was due to the lower solubility of the diesel standard. In freshwater, we were able to achieve around 40 - 50% of the nominal concentration in solutions that were near the lower end of our dilution series (< 1.5mg/L). In solutions that were above 1.5 mg/L, we achieved around 8 - 15% of the nominal concentration. In marine water, the measured concentration as a percent of the nominal concentration was around 7 - 20%.

Similar to the Gx tests, we modeled the relationships between measured and nominal NWTPH-Dx concentrations using regressions in order to interpolate and extrapolate the concentrations that were not measured (Figures 2 and 3). Nonlinear polynomial regression was used for the topsmelt results to attain a better model fit (Figure 2). Likewise, the best fit for the *Ceriodaphnia dubia* results was a nonlinear power model (Figure 3). The echinoderm fertilization test relied on the same dilution series (t = 0) as the topsmelt.

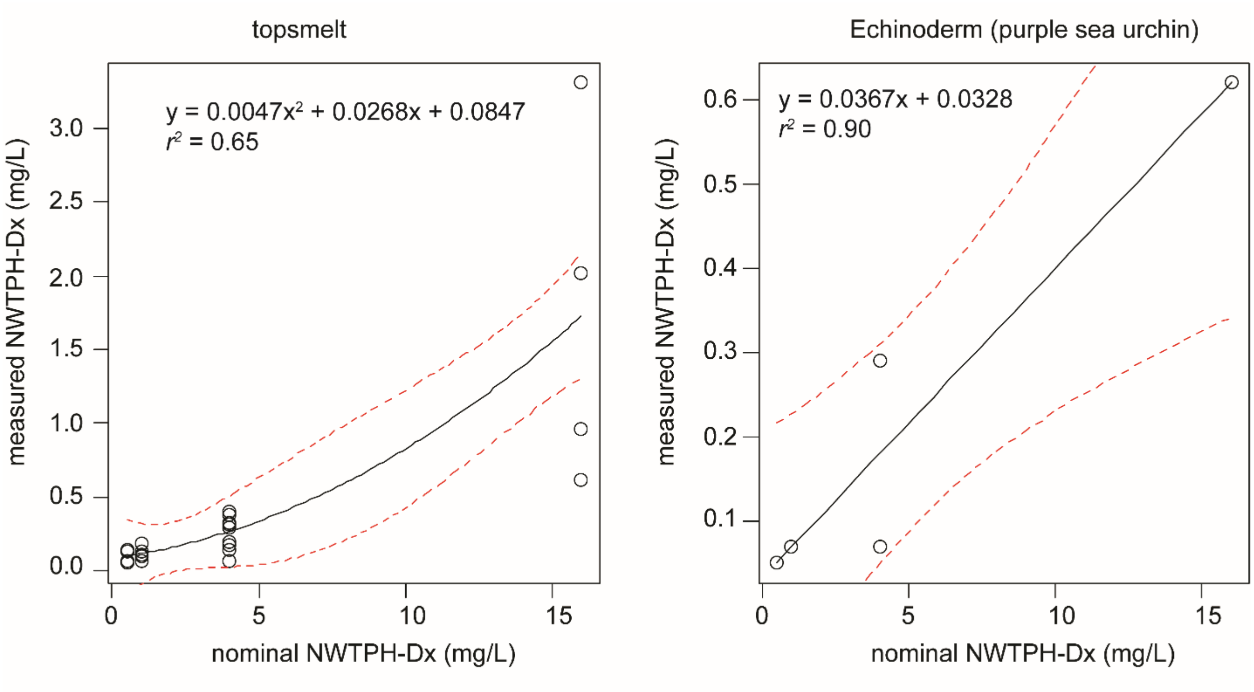

Figure 2: Regressions of measured vs. nominal NWTPH-Dx with 95% confidence limits for marine organisms.

Regression formulas were used for interpolation of results; r-squared is adjusted for Bonferroni correction.

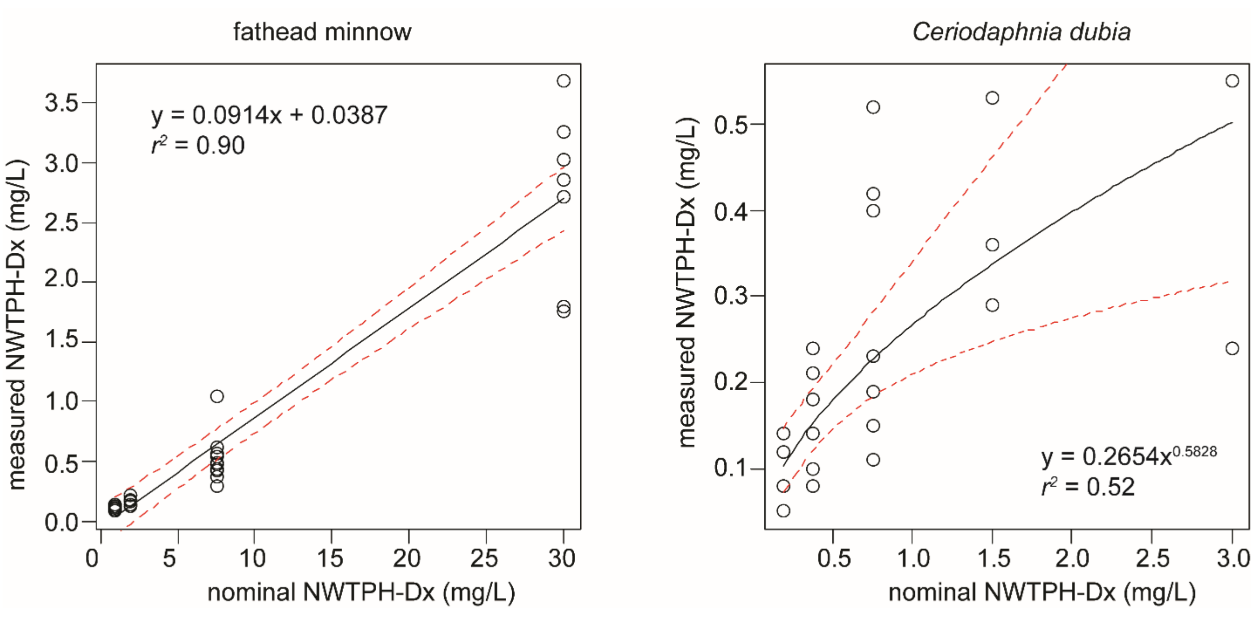

Figure 3: Regressions of measured vs. nominal NWTPH-Dx with 95% confidence limits for freshwater organisms.

Regression formulas were used for interpolation of results; r-squared is adjusted for Bonferroni correction.

The final dilution series for the chronic tests using Dx on marine organisms was 16, 8, 4, 2, 1, and 0.5 mg/L (Tables 19 and 20). For the freshwater organisms, the dilutions series was 30, 15, 7.5, 3.8, 1.9, and 0.9 mg/L for the fathead minnow and 6, 3, 1.5, 0.75, 0.38, 0.19, 0.09, and 0.045 mg/L for the *Ceriodaphnia dubia* (Tables 21 and 22). All concentrations not measured in the dilution series were estimated using the relationships between nominal and measured. Different dilution series were chosen because of observed differences in the sensitivity of the organisms and differences in our ability to dissolve the Dx in marine water and freshwater during the range-finding tests.

Table 19: Measured and estimated NWTPH-Dx concentrations in fresh and stale solutions for topsmelt (Nautilus, 2017b).

Final concentrations that were not measured were interpolated or extrapolated based on non-linear regression.

Nominal Concentration (mg/L)	t=0	t=24		t=48	t=72	t=96		t=120	t=144	t=168	Final Concentration (mg/L)
	fresh	stale	fresh	fresh	fresh	stale	fresh	fresh	fresh	stale	
16	0.62	0.58	2.02	3.31	0.96						1.6
8											***0.57***
4	0.29	0.14	0.38	0.31	0.32	0.15	0.17	0.2	0.29	0.2	0.26
2											***0.16***
1	0.07	*0.05*	0.13	0.1	0.11	0.16	0.11	0.18	0.18	0.28	0.12
0.5	*0.05*	*0.05*	0.07	0.06	0.06	0.12	0.13	0.14	0.13	0.17	0.10

Italic concentrations are below the reporting limit; ***bold italic*** are estimated concentrations.

Table 20: Measured and estimated NWTPH-Dx concentrations in fresh and stale solutions for echinoderm fertilization (Nautilus, 2017b).

Final concentrations that were not measured were interpolated or extrapolated based on linear regression.

Nominal Concentration (mg/L)	t=0	Final Concentration (mg/L)
16	0.62	0.62
8		***0.33***
4	0.29	0.18
4 duplicate	0.07	
2		***0.11***
1	0.07	0.07
0.5	*0.05*	0.05

Table 21: Measured and estimated NWTPH-Dx concentrations in fresh and stale solutions for fathead minnow (Nautilus, 2017b).

Final concentrations that were not measured were interpolated or extrapolated based on linear regression.

Nominal Concentration (mg/L)	t=0	t=24		t=48	t=72	t=96		t=120	t=144	t=168	Final Concentration (mg/L)
	fresh	stale	fresh	fresh	fresh	stale	fresh	fresh	fresh	stale	
30	3.03	0.59	3.26	1.75	1.79	0.59	2.86	2.72	3.68	1.37	2.7
15											***1.3***
7.5	0.49	0.18	0.48	0.37	0.42	0.19	0.57	0.62	1.05	0.42	0.65
3.8											***0.31***
1.9	0.13	0.06	0.12	0.13	0.18	0.18	0.17	0.14	0.21	0.28	0.13
0.9	0.08	0.06	0.11	0.14	0.1	0.15	0.1	0.1	0.12	0.29	0.04

Bold italic are estimated concentrations

Table 22: Measured and estimated NWTPH-Dx concentrations in fresh and stale solutions for *Ceriodaphnia dubia* (Nautilus, 2017b).

Final concentrations that were not measured were interpolated or extrapolated based on non-linear regression.

Nominal Concentration (mg/L)	t=0	t=24	t=48	t=72	t=96	t=120	t=144	Final Concentration (mg/L)
	fresh	fresh	fresh	fresh	fresh	fresh	fresh	
6	1.19							***0.75***
3	0.55							***0.50***
1.5	0.29	1.22	0.36	0.53				0.34
0.75	0.11	0.52	0.23	0.15	0.4	0.19	0.42	0.22
0.38	0.08	0.24	0.18	0.14	0.21	0.1	0.21	0.15
0.19	*0.05*		0.08		0.12		0.14	0.10
0.09								***0.07***
0.045								***0.04***

Italic concentrations are below the reporting limit
Bold italic are estimated concentrations

Concentrations of the solutions after the daily exposures (stale solutions) in the fish chambers lost somewhere between 30 and 55% of the NWTPH-Dx (Table 23). However, about 25% of the solutions at the lower end of the dilution series had higher measured concentrations at the end of the daily exposure. These samples were approximately 0.05 mg/L higher after the daily exposure (Tables 19 and 21). Based on our observations and QC data, the variability in the loss of Dx from the chambers seems more attributable to variability in mixing the daily solutions and variability in lab analytical bias and precision. It is likely that at the higher concentrations there was some measureable loss due to uptake by the fish.

Table 23: Percent loss from water of NWTPH-Dx during the daily toxicity test exposures.

	Topsmelt	Fathead Minnow
Mean	33%	49%
Median	31%	55%
Standard deviation	19%	24%
Number of samples	10	12
Percent of chambers gaining Dx	30%	25%

Effects of Silica Gel Cleanup

As a secondary objective in this project, we tested a subset of the NWTPH-Dx samples using silica cleanup and no cleanup for comparison. Unlike the methods for extractable petroleum hydrocarbons (EPHs), the preparation of the samples for the NWTPH-Dx method does not include cleaning the media for naturally-occurring organics that can interfere with the quantification, unless it can be shown that naturally occurring organic matter is a significant component of the TPH being detected in the samples (Ecology, 2016). The silica gel cleanup of the sample can lead to a loss of degradation products and polar organics, possibly biasing the measured concentration low.

Stale solutions were sampled from the fish chambers where sufficient sample was available and there was a decent likelihood of the organism contributing additional organics. Samples taken during the toxicity tests had some noticeable particulate organics in them, and the color of the sample water following extraction was brown, which indicates the presence of dissolved organic matter (DOM). The particulates and DOM would have been contributed by the fish and the fish food in the test chamber. Following the silica cleanup, the extracts were colorless indicating that the DOM had been removed (*pers. comm*. D. Montgomery).

A total of 26 samples were selected for silica gel cleanup of NWTPH-Dx[2]. There was no statistical difference between the pre-silica gel cleanup and post silica gel cleanup concentration. A t-test on log-transformed data yielded significance *p*-value of 0.83. Indeed, when comparing the concentrations of Dx in the original samples against the concentrations following cleanup, all of the samples were very close to a 1:1 line (Figure 4). There was also no observed difference of silica cleanup between marine water and freshwater.

[2] Cleanup included only silica gel and not the sulfuric acid treatment as per the method described in Ecology, 1997.

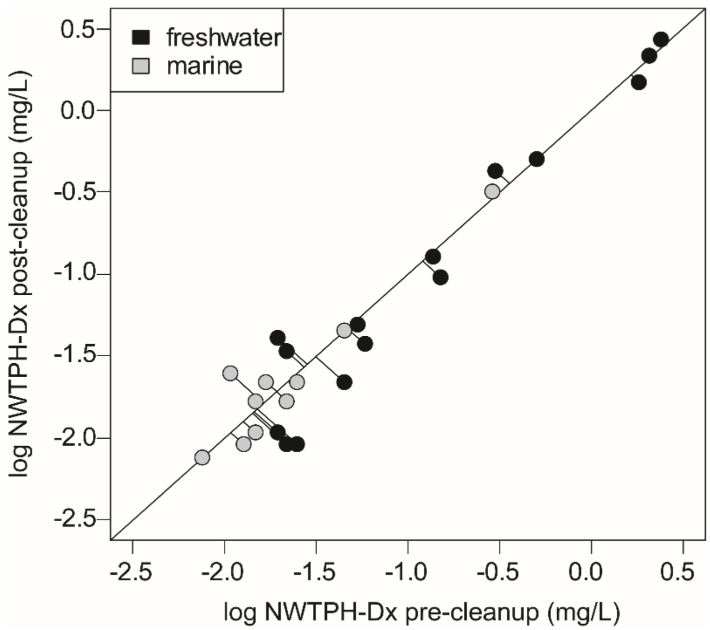

Figure 4: Scatterplot of NWTPH-Dx post-cleanup vs. pre-cleanup.

Data are plotted on a log-log plot. Departure from the 1:1 line for each sample is shown. Black dots are freshwater samples, and gray dots are marine water.

Toxicity Testing

In both the NWTPH-Gx and –Dx tests we were able to establish a reasonable NOEC and LOEC for marine water and freshwater; these values are established directly from the biological endpoints (i.e. the measured concentrations that cause biological effects). In addition, point estimates of lethal (median lethal concentration; LC50) and sublethal (25% inhibitive concentration; IC25) conditions were established for all test organisms. Point estimates are calculated using statistical regression methods based on all available data on dose-response for a specific organism, not simply a linear interpolation between two test concentrations.

Gasoline (Gx) fraction

The toxicity testing on the topsmelt in marine water produced clear lethal and sublethal effects data (Figure 5). A point estimate of the LC50 – where 50% of the population dies – was calculated at 1.7 mg/L. The IC25 was calculated based on biomass results and was also 1.7 mg/L. The LC50 was calculated using the Spearman-Kärber method, while the IC25 was calculated using a linear interpolation.

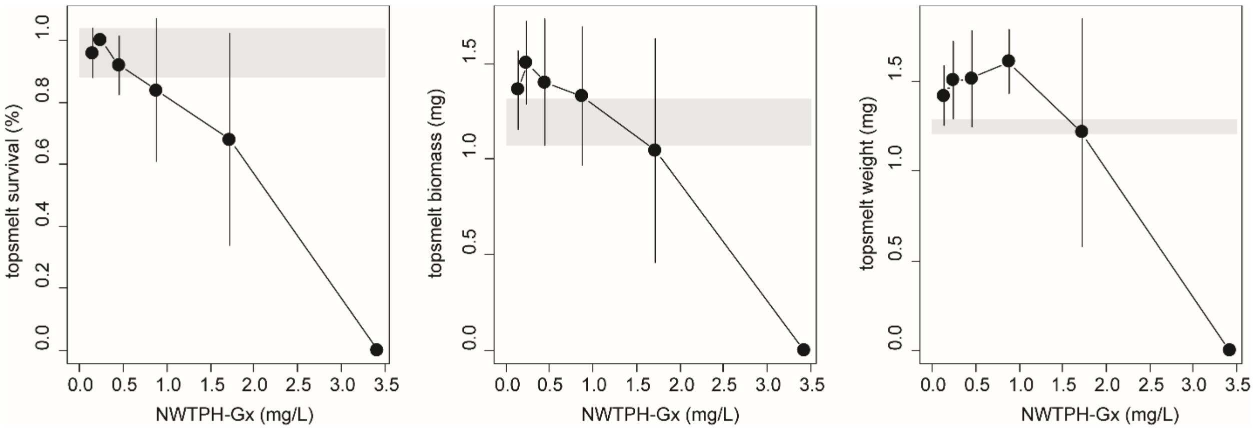

Figure 5: Toxicity results for effects on topsmelt survival and growth from NWTPH-Gx.
Points are the mean of five replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test; left – survival endpoint; center - growth endpoint (biomass); right – growth endpoint (weight)

The echinoderm (purple sea urchin) fertilization test had very good precision around the solvent control, which gave tighter confidence limits (Figure 6). The effect on fertilization (IC25) was discernible at 3.0 mg/L using a linear interpolation.

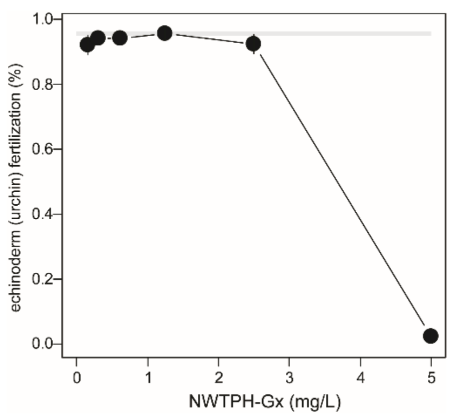

Figure 6: Toxicity results for the effects on echinoderm (sea urchin) fertilization from NWTPH-Gx.
Points are the mean of four replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test.

In freshwater, the fathead minnow tests were successful at demonstrating a clear lethal and sublethal effect (Figure 7). The LC50 was estimated at 2.5 mg/L using the Spearman-Kärber method, while the IC25 was estimated at 1.5 mg/L using an interpolation on a nonlinear regression.

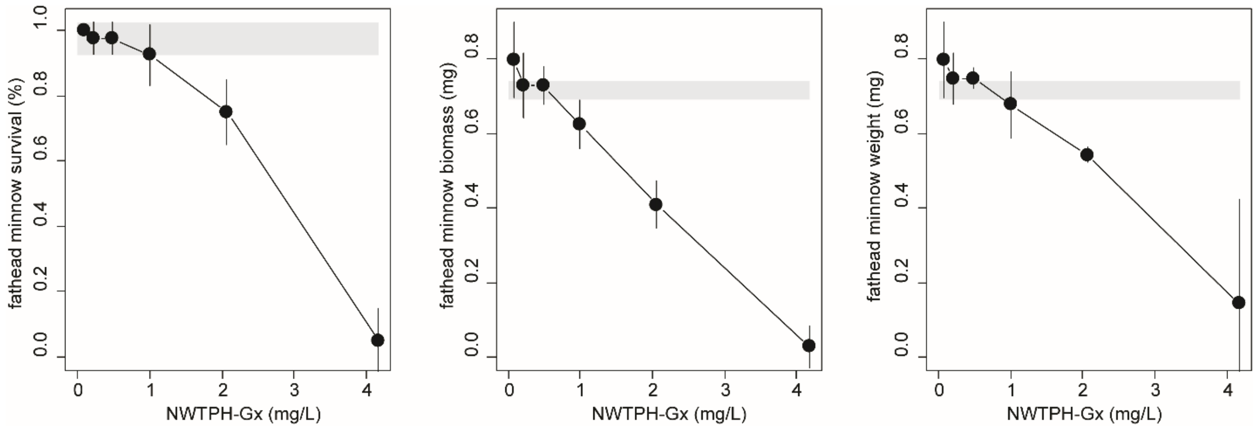

Figure 7: Toxicity results for the effects on fathead minnow survival and growth from NWTPH-Gx.

Points are the mean of four replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test; left – survival endpoint; center - growth endpoint (biomass); right – growth endpoint (weight).

The *Ceriodaphnia dubia* tests were not successful at showing clear lethal effects, which resulted in an LC50 > 2.3 mg/L (Figure 8). However, there was an inhibitory effect observed on reproduction which resulted in an IC25 estimate of 1.7 mg/L.

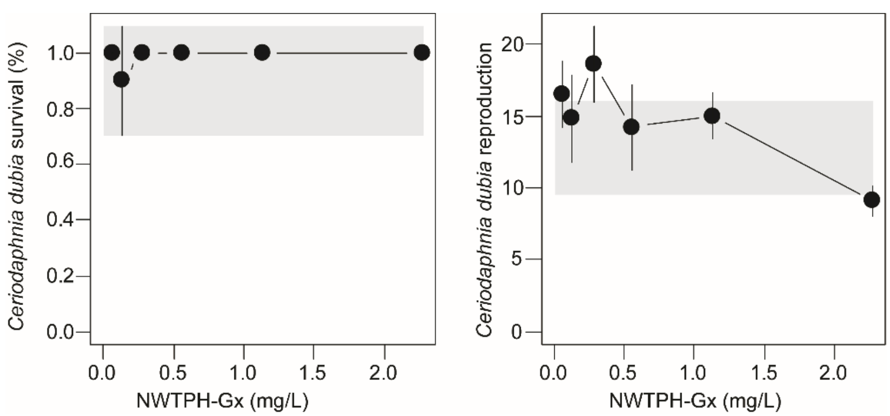

Figure 8: Toxicity results for the effects on *Ceriodaphnia dubia* survival and reproduction from NWTPH-Gx.

Points are the mean of ten replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test; left – survival endpoint; right – reproduction endpoint.

The main goal of this study was to establish defendable NOEC and LOEC concentrations for marine water and freshwater. NOEC and LOEC estimates based on comparisons with the negative controls — solvent controls in our tests — are summarized in Table 24, with the appropriate statistical test used. Based on NOEC values for NWTPH-Gx, we recommend using 1.0 mg/L in freshwater and 1.7 mg/L in marine water. These recommended concentrations are higher than the practical quantitation limit (PQL) of 0.8 mg/L used under Method A of WAC 173-340-730 (see *Introduction – Regulatory Criteria or Standards*).

Table 24: Summary of NWPTH-Gx toxicity point estimates, NOEC and LOEC.

<table>
<tr><th colspan="2">Endpoint *</th><th>Point Estimate</th><th>mg/L Gasoline (95% CL)</th><th>Statistical Test</th><th>NOEC mg/L Gasoline</th><th>LOEC mg/L Gasoline</th><th>Statistical Test</th></tr>
<tr><td rowspan="7">Marine water</td><td colspan="7">Topsmelt</td></tr>
<tr><td>survival</td><td>LC50</td><td>1.7 (1.5–2.1)</td><td>Spearman-Kärber</td><td>1.7</td><td>>1.7</td><td>Steel Many-one Rank Sum test</td></tr>
<tr><td>biomass</td><td>IC25</td><td>1.7 (0.4–2.2)</td><td rowspan="2">Linear interpolation</td><td rowspan="2">1.7</td><td rowspan="2">>1.7</td><td rowspan="2">Dunnett multiple comparison</td></tr>
<tr><td>biomass</td><td>IC50</td><td>2.2 (1.1–2.6)</td></tr>
<tr><td colspan="7">Echinoderm</td></tr>
<tr><td>fertilization</td><td>IC25</td><td>3 (2.9–3.0)</td><td rowspan="2">Linear interpolation</td><td rowspan="2">2.5</td><td rowspan="2">5.0</td><td rowspan="2">Dunnett multiple comparison</td></tr>
<tr><td>fertilization</td><td>IC50</td><td>3.6 (3.5–3.6)</td></tr>
<tr><td rowspan="8">Freshwater</td><td colspan="7">Fathead minnow</td></tr>
<tr><td>survival</td><td>LC50</td><td>2.5 (2.2–2.8)</td><td>Spearman-Kärber</td><td>1.0</td><td>2.1</td><td>Dunnett multiple comparison</td></tr>
<tr><td>biomass</td><td>IC25</td><td>1.5 (1.2–1.7)</td><td rowspan="2">Nonlinear regression</td><td rowspan="2">1.0</td><td rowspan="2">2.1</td><td rowspan="2">Dunnett multiple comparison</td></tr>
<tr><td>biomass</td><td>IC50</td><td>2.1 (1.9–2.3)</td></tr>
<tr><td colspan="7">Ceriodaphnia dubia</td></tr>
<tr><td>survival</td><td>LC50</td><td>>2.3</td><td rowspan="3">Linear interpolation</td><td>2.3</td><td>>2.3</td><td>Fisher Exact/Bonferroni-Holm test</td></tr>
<tr><td>reproduction</td><td>IC25</td><td>1.7 (1.3–1.9)</td><td rowspan="2">1.1</td><td rowspan="2">2.3</td><td rowspan="2">Steel Many-one Rank Sum test</td></tr>
<tr><td>reproduction</td><td>IC50</td><td>>2.3</td></tr>
</table>

LC = Lethal Concentration, IC = Inhibition Concentration, NOEC = No Observed Effect Concentration, LOEC = Lowest Observed Effect Concentration.
* Result was calculated using the solvent control as the negative control.

Although we did not test the organisms for effects of individual BTEX compounds, we can report the BTEX concentrations close to the NWTPH-Gx NOEC concentrations (Table 25). It is important to recognize that the gasoline in these samples is unweathered.

Table 25: Measured BTEX concentrations close to the calculated NOEC values.

	NWTPH-Gx (µg/L)		**Benzene** (µg/L)		**Ethylbenzene** (µg/L)		**Toluene** (µg/L)		**m.p-Xylenes** (µg/L)		**o-Xylenes** (µg/L)	
	mean	sd	mean	sd	mean	sd	mean	sd	mean	sd	mean	sd
Fresh	893.6	159.2	9.7	1.9	11.5	2.4	53.0	9.4	40.0	7.6	16.6	4.3
Marine	1620.0	325.1	22.8	8.9	20.4	2.2	101.9	5.7	74.6	9.1	30.8	1.9

Comparison of our findings to other studies found in EPA's EcoTox database[3] is difficult because we used an unweathered gasoline standard and studies in EcoTox generally use either weathered hydrocarbons or specific BTEX compounds. Furthermore, different organisms are often used. A marine fish similar to topsmelt, atlantic silverside (*Menidia menidia*), has been tested using ethylbenzene and found to have a NOEC of 3,300 µg/L (Masten et al., 1994). In freshwater, concentrations for NOEC of BTEX on fathead minnows range from 5,400 to 10,200 µg/L (Marchini et al., 1992). These results are significantly higher than the range of BTEX compounds found in the NWTPH-Gx NOEC for our study. This observation is also true when comparing the point estimate, LC50, to previous studies (Pickering and Henderson, 1966; Devlin et al., 1982; Slooff, 1982; Brooke, 1987; Geiger et al., 1990; Marchini et al., 1992).

Diesel (Dx) fraction

The toxicity testing on the topsmelt in marine water produced clear lethal and sublethal effects data (Figure 9). The LC50 point estimate was calculated at 0.68 mg/L, while the IC25 based on biomass results, was 0.74 mg/L. The LC50 was calculated using the Spearman-Kärber method, while the IC25 was calculated using a linear interpolation.

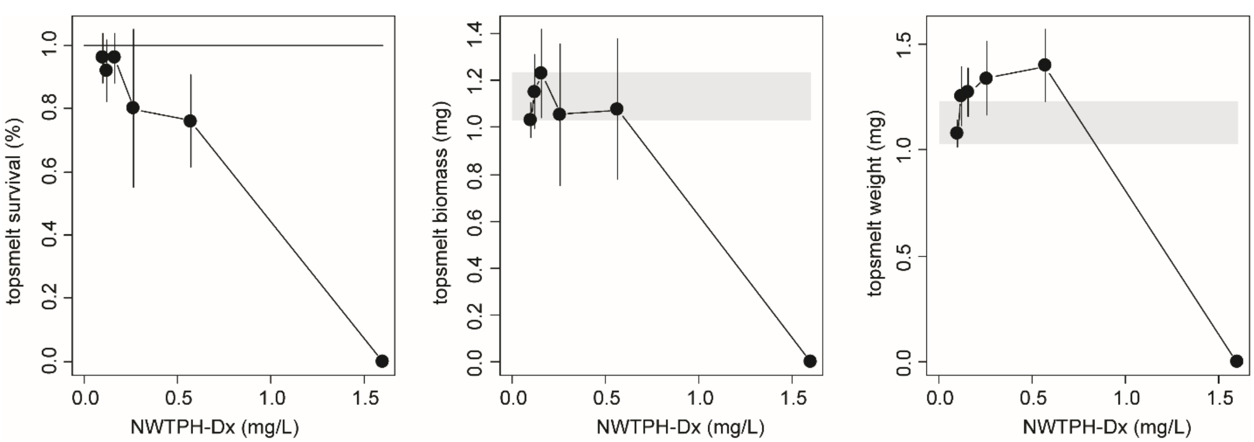

Figure 9: Toxicity results for effects on topsmelt survival and growth from NWTPH-Dx.
Points are the mean of five replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test; left – survival endpoint; center - growth endpoint (biomass); right – growth endpoint (weight)

[3] https://cfpub.epa.gov/ecotox/index.html

The 24-hr toxicity test on echinoderm fertilization produced very precise and clear results (Figure 10). The IC25 calculated from the linear interpolation modeled results was 0.19 mg/L NWTPH-Dx.

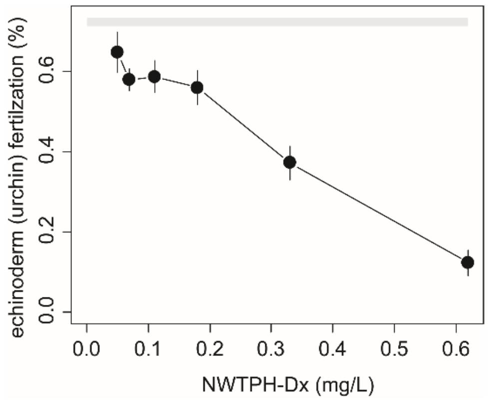

Figure 10: Toxicity results for the effects on echinoderm (sea urchin) fertilization from NWTPH-Dx.
Points are the mean of four replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test.

In the freshwater toxicity tests on the fathead minnows produced clear results for lethal and sublethal effects (Figure 11). Point estimates of the LC50 and IC 25 were calculated at 1.87 mg/L and 0.87 mg/L NWTPH-Dx, respectively. These were based on a linear interpolation method, respectively.

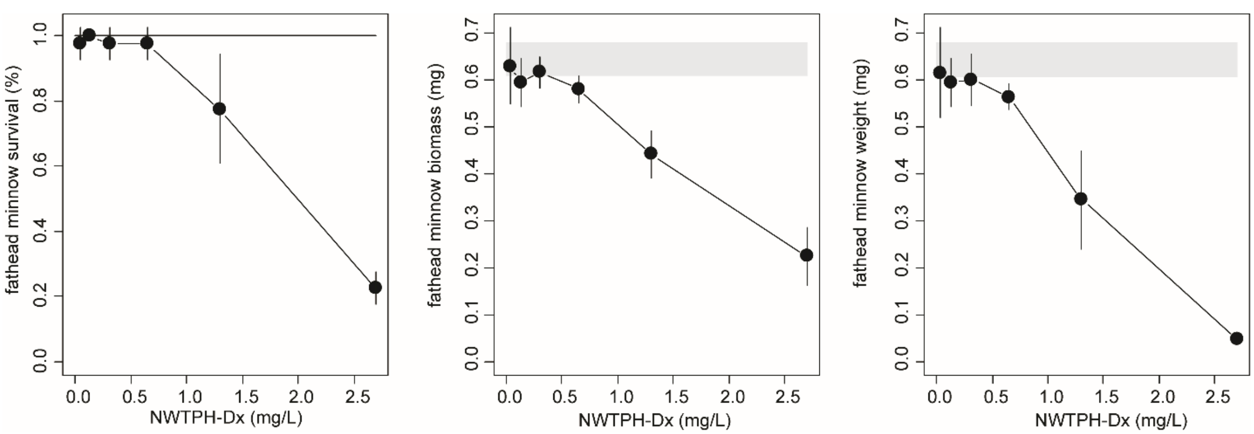

Figure 11: Toxicity results for the effects on fathead minnow survival and growth from NWTPH-Dx.
Points are the mean of four replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test; left – survival endpoint; center - growth endpoint (biomass); right – growth endpoint (weight).

Lastly, the *Ceriodaphnia dubia* tests showed clear adverse effects on survival and sublethal effects (Figure 12). Point estimates of survival (LC50) were calculated at 0.23 mg/L NWTPH-Dx, while an IC25 was calculated at 0.17 mg/L NWPTH-Dx.

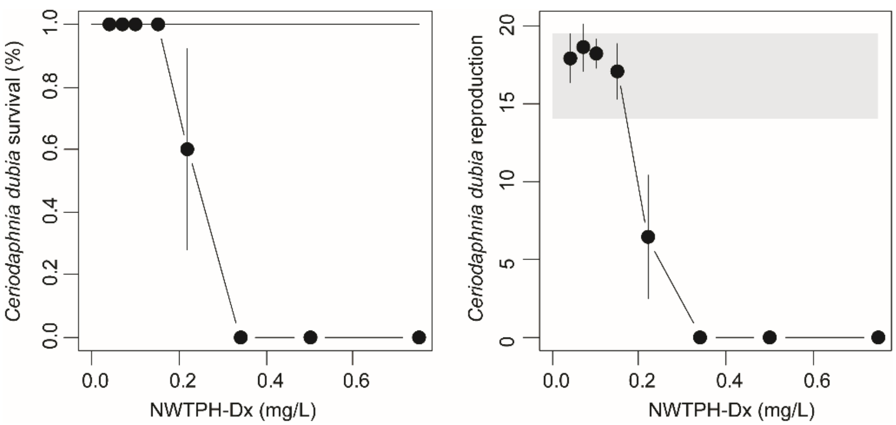

Figure 12: Toxicity results for the effects on *Ceriodaphnia dubia* survival and reproduction from NWTPH-Dx.

Points are the mean of ten replicates with 95% confidence limits; gray shaded area represents the 95% confidence interval of the control test; left – survival endpoint; right – reproduction endpoint..

Based on the toxicity tests we were able to estimate defendable NOEC and LOEC values for marine water and freshwater. A summary of each of the tests effects concentrations are found in Table 26. Based on the NOEC values for NWTPH-Dx, we recommend using a concentration of 0.15 mg/L in freshwater and <0.05mg/L for marine waters. The concentration of <0.05 mg/L represents the method detection limit for the NWTPH-Dx analysis. This concentration is an order of magnitude lower than the PQL of 0.5 mg/L used under Method A of WAC 173-340-730 (see *Introduction – Regulatory Criteria or Standards*).

Direct comparisons with EPA's EcoTox database and similar studies is difficult for diesel fuel because the majority of previous toxicity testing on heavier hydrocarbons has taken place on unrefined mixtures or crude oil in most cases. In addition, there is a lack of comparable test organisms to our study. However, the inland silverside (*Menidia beryllina*) is somewhat comparable to the topsmelt based on life history and habitat. In a study by Little et al. (2000) it was tested for similar endpoints (growth and survival by weight) to this study. The concentrations of 700 µg/L for the NOEC and 1500 µg/L for the LOEC were observed, which is higher than our findings.

The effects of a petroleum mixture (CAS#:8002059) also has been tested on marine invertebrates. For an urchin, the LOEC for general damage to the organism was found at 60 µg/L (Taban et al., 2004). In a study with effects endpoints similar to our study, O'Clair and Rice (1985) found an LOEC of 200 µg/L for a seastar and an NOEC of 120 µg/L for growth effects. These published effects concentrations are slightly higher than what we found for echinoderm fertilization (NOEC < 50 µg/L).

Table 26: Summary of NWPTH-Dx toxicity point estimates, NOEC and LOEC.

Endpoint *		Point Estimates	mg/L Diesel (95% CL)	Statistical Test	NOEC (mg/L)	LOEC (mg/L)	Statistical Test
Marine water	**Topsmelt**						
	survival	LC50	0.68 (0.55 – 0.83)	Spearman-Kärber	0.26	0.57	Dunnett multiple comparison
	biomass	IC25	0.74 (0.37 –0.80)	Linear interpolation	0.57	1.60	
	biomass	IC50	0.99 (0.76 – 1.04)				
	Echinoderm						
	fertilization	IC25	0.19 (0.12 – 0.22)	Linear interpolation	<0.05	0.05	Dunnett multiple comparison
	fertilization	IC50	0.34 (0.29 – 0.38)				
Freshwater	**Fathead minnow**						
	survival	LC50	1.87 (1.43 – 2.45)	Spearman-Kärber	1.30	2.70	Steel Many-one Rank Sum test
	biomass	IC25	0.87 (0.70 – 1.23)	Linear interpolation	0.65	1.30	Dunnett multiple comparison
	biomass	IC50	1.39 (1.03 – 1.84)				
	Ceriodaphnia dubia						
	survival	LC50	0.23 (0.20 – 0.26)	Spearman-Kärber	0.22	0.34	Fisher Exact/Bonferroni-Holm test
	reproduction	IC25	0.17 (0.16 – 0.19)	Linear interpolation	0.15	0.22	Steel Many-one Rank Sum test
	reproduction	IC50	0.20 (0.19 – 0.24)				

LC = Lethal Concentration, IC = Inhibition Concentration, NOEC = No Observed Effect Concentration, LOEC = Lowest Observed Effect Concentration.
* Result was calculated using the solvent control as the negative control.

In freshwater, there are no EcoTox data for the chronic toxicity of petroleum mixtures on fathead minnows; however, acute testing has been carried out on slimy sculpin, dolly varden, and threespine stickleback using crude oil (Moles et al., 1979). Results for the LC50 ranged from 1250 to 6890 µg/L. The lower end of this published range is compatible with our LC50 findings for fathead minnows (Table 26). We were unable to find data on the impacts of a similar petroleum mixture on freshwater invertebrates.

Conclusions

We conducted chronic toxicity tests using NWTPH-Gx and -Dx fractions on aquatic organisms in marine water and freshwater. The following conclusions can be drawn from this study:

- The water-accommodated fraction (WAF) of the test solutions was generally between 60-85% of the desired nominal concentration for Gx. In the Dx test solutions the WAF was generally < 20% except for low concentrations in freshwater, where the WAF increased to 40 - 50% of the desired nominal concentrations.
- The measured concentrations of the Gx test solutions during the 7-day renewal had a relative standard deviation (or coefficient of variation) of approximately 20 - 30%. Among the Dx samples it was approximately 20 - 40%.
- Stale solutions were sampled during the fish toxicity tests. The Gx test solutions lost approximately 70 - 80% over the 24-hour period. Losses were likely due to volatilization, uptake by the organism and analytical variability. The Dx test chambers lost between 30 and 55%. Differences between fresh and stale solutions were likely due to analytical variability and uptake by the organism.
- A subset (26 samples) of samples analyzed for NWTPH-Dx were re-analyzed with an additional silica gel cleanup method. Comparison of the original results with results following silica gel cleanup showed no significant difference. For this subset of samples, the silica gel cleanup did not influence the NWTPH-Dx concentration.
- The toxicity test results from this study were comparable to literature values or in the case of NWTPH-Gx provided greater detail of inhibitory effects at much lower concentrations.
- For both NWTPH-Gx and NWTPH-Dx tests, clear lethal and sublethal effects were observed. Conservative point estimates of the LC50 and IC25 and LOEC and NOEC values are summarized below (Table 27).

Table 27: Overall summary of toxicity point estimates and effects-concentrations for NWTPH in marine water and freshwater.

		Point Estimates (mg/L)		**LOEC** (mg/L)	**NOEC** (mg/L)
		LC50	IC25		
NWTPH-Gx (mg/L)	Marine water	1.7	1.7	>1.7	1.7
	Freshwater	2.5	1.5	2.1	1.0
NWTPH-Dx (mg/L)	Marine water	0.68	0.19	0.05	<0.05
	Freshwater	0.23	0.17	0.22	0.15

Recommendations

Based on the findings from this study and the goals of the study, the following recommendations can be made:

- Conservative NOEC values for NWTPH in marine water and freshwater have been derived. These values represent the "no-effects" levels for NWTPH in surface waters. Use the NOEC values to inform appropriate guidance under WAC-173-340-730(3)(b)(ii) (Environmental effects) – Surface Water Cleanup Standards.
- This study is based on unweathered NWTPH, a companion field study using contaminated groundwater and weathered NWTPH in toxicity tests would be a logical follow-up.

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Personal communication

Dolores Montgomery, Chemist 4, Organics Section, Manchester Environmental Lab, Washington State Department of Ecology, Manchester, WA. 11/28/17 by email.

Citations in text

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Appendices

Appendix A. Toxicity Testing on NWTPH-Gx (Nautilus Environmental)

Appendix A is linked to the report on the web at:

https://fortress.wa.gov/ecy/publications/SummaryPages/1803002.html

Appendix B. Toxicity Testing on NWTPH-Dx (Nautilus Environmental)

Appendix B is linked to the report on the web at:

https://fortress.wa.gov/ecy/publications/SummaryPages/1803002.html

Appendix C. Certificates for Standard Reference Materials

RESTEK
110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

www.restek.com

CERTIFIED REFERENCE MATERIAL

Certificate of Analysis

7010330

FOR LABORATORY USE ONLY-READ SDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed

Catalog No. : 30206 **Lot No.:** A0111959

Description : Unleaded Gasoline Composite Standard

Unleaded Gas Composite 50,000µg/mL P&T Methanol, 5ml ampul

Container Size : 5 mL **Pkg Amt:** > 5 mL

Expiration Date : July 31, 2022 **Storage:** 0°C or colder

CERTIFIED VALUES

Elution Order	Compound		Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)	
1	Unleaded Gasoline Composite CAS # 8006-61-9 Purity —%	(Lot [illegible])	50,500.0 µg/mL	+/- 295.5960 µg/mL +/- 4,541.0586 µg/mL +/- 4,541.0586 µg/mL	Gravimetric Unstressed Stressed

Solvent: P&T Methanol
CAS # 67-56-1
Purity 99%

RESTEK
110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

www.restek.com

CERTIFIED REFERENCE MATERIAL

Certificate of Analysis

ACCREDITED
ISO Guide 34 Accredited Reference Material Producer

ilac-MRA

ACCREDITED
ISO/IEC 17025 Accredited Testing Laboratory

FOR LABORATORY USE ONLY-READ SDS PRIOR TO USE.

This Reference Material is intended for Laboratory Use Only as a standard for the qualitative and/or quantitative determination of the analyte(s) listed.

Catalog No. : 31259 **Lot No.:** A0126761

Description : Diesel Fuel #2 Composite Standard

Diesel Fuel #2 Composite Standard 50,000µg/mL, Methylene Chloride, 5mL/ampul

Container Size : 5 mL **Pkg Amt:** > 5 mL

Expiration Date : May 31, 2024 **Storage:** 25°C nominal

CERTIFIED VALUES

Elution Order	Compound		Grav. Conc. (weight/volume)	Expanded Uncertainty (95% C.L.; K=2)		
1	Diesel Fuel #2 Composite		50,028.9 µg/mL	+/- 290.8571	µg/mL	Gravimetric
	CAS # 68334-30-5.A	(Lot A0125454)		+/- 1,489.5176	µg/mL	Unstressed
	Purity ---%			+/- 1,588.6217	µg/mL	Stressed

Solvent: Methylene Chloride
CAS # 75-09-2
Purity 99%

Appendix D. NWTPH-Gx Analytical Results

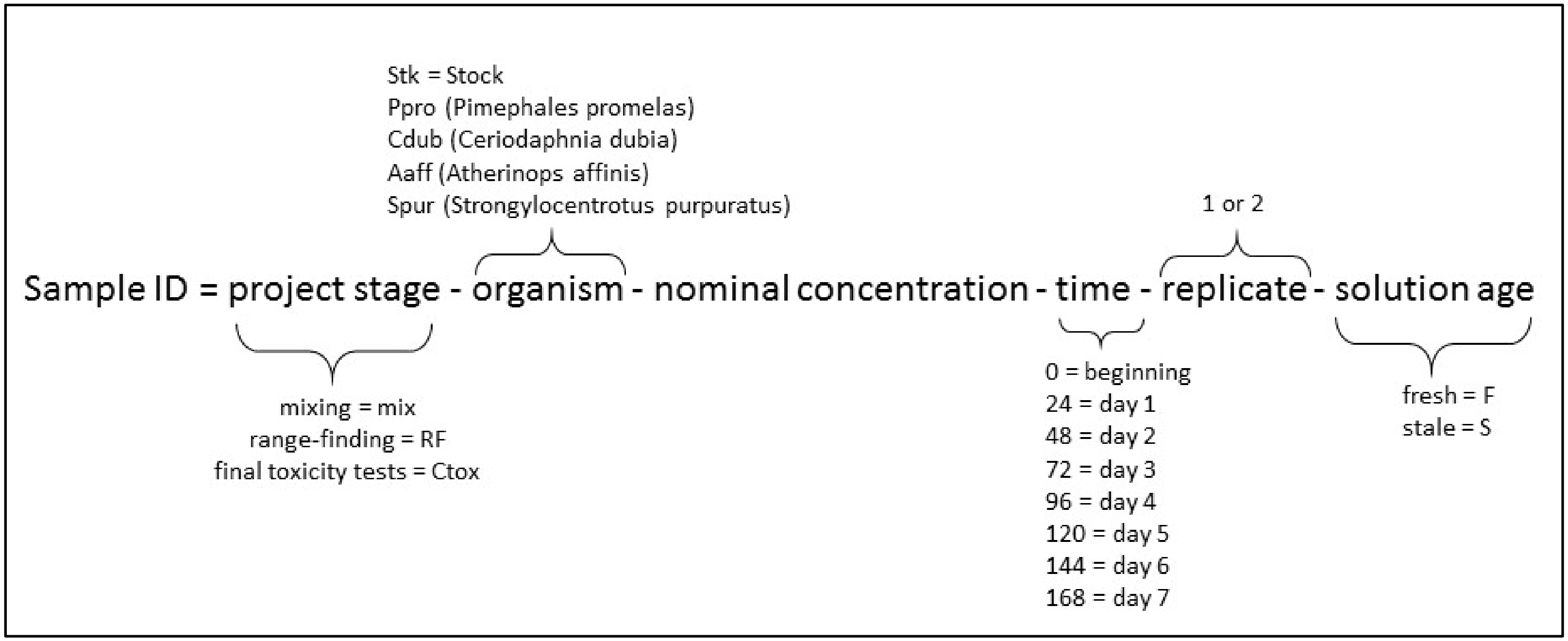

Table D-1: Results of the NWTPH-Gx mixing trials.

Sample ID	MEL ID	sample date	analysis date	nominal Gx	TPH-Gx (mg/L)		benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)	
mix-Stk-200-0-1	1704045-1	4/19/2017	4/25/2017	200	91.6		1210		1120		6650		4410		1840	
mix-Stk-200-0-2	1704045-2	4/19/2017	4/25/2017	200	87.2		1160		1140		6360		4180		1780	
mix-Stk-200-0-3	1704045-3	4/19/2017	4/25/2017	200	87.3		1170		1150		6380		4180		1780	
mix-Stk-56-0-1	1704045-4	4/19/2017	4/25/2017	56	18.9		239		234		1300		873		390	
mix-Stk-56-0-2	1704045-5	4/19/2017	4/25/2017	56	21.6		259		246		1440		990		437	
mix-Stk-56-0-3	1704045-6	4/19/2017	4/25/2017	56	20.8		270		264		1480		972		432	
mix-Stk-1-0-1	1704045-7	4/19/2017	4/24/2017	1	0.411		4.38		4.68		23.6		17.6		7.47	
mix-Stk-1-0-2	1704045-8	4/19/2017	4/24/2017	1	0.402		4.27		4.91		23		17		7.28	
mix-Stk-1-0-3	1704045-9	4/19/2017	4/24/2017	1	0.403		4.27		4.35		23		17.5		7.3	
mix-Stk-0-0-1	1704045-10	4/19/2017	4/24/2017	0	0.07	U	1	U	1	U	1	U	2	U	1	U
mix-Stk-0-0-2	1704045-11	4/19/2017	4/24/2017	0	0.07	U	1	U	1	U	1	U	2	U	1	U
mix-Stk-0-0-3	1704045-12	4/19/2017	4/24/2017	0	0.07	U	1	U	1	U	1	U	2	U	1	U

Table D-2: Results of the NWTPH-Gx range-finding tests.

Sample ID	MEL ID	sample date	Analysis date	nominal Gx	TPH-Gx (mg/L)		matrix recovery* (%)	benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)		matrix recovery** (%)
RF-Marine-BLNK-1	1705051-01	4/27/2017	5/2/2017	0	0.07	U	103	1	U	1	U	1	U	2	U	1	U	103
RF-Marine-BLNK-2	1705051-02	4/27/2017	5/2/2017	0	0.07	U	104	1	U	1	U	1	U	2	U	1	U	104
RF-Marine-BLNK-3	1705051-03	4/27/2017	5/2/2017	0	0.07	U	101	1	U	1	U	1	U	2	U	1	U	101
RF-Aaff-1-0-1-F	1705051-04	4/27/2017	5/3/2017	1	0.606		102	5.66		7.14		33.40		26.80		10.5		101
RF-Aaff-10-0-1-F	1705051-05	4/27/2017	5/3/2017	10	7.51		99	82.80		98.20		461.00		357.00		14100		99
RF-Aaff-100-0-1-F	1705051-06	4/27/2017	5/3/2017	100	59.1		102	881.00		792.00		4770.00		2890.00		1220		100
RF-FRESH-BLNK-1	1705051-07	4/27/2017	5/3/2017	0	0.07	U	99	1	U	1	U	1	U	2	U	1	U	100
RF-FRESH-BLNK-2	1705051-08	4/27/2017	5/3/2017	0	0.07	U	102	1	U	1	U	1	U	2	U	1	U	103
RF-FRESH-BLNK-3	1705051-09	4/27/2017	5/3/2017	0	0.07	U	99	1	U	1	U	1	U	2	U	1	U	99
RF-Ppro-1-0-1-F	1705051-10	4/27/2017	5/3/2017	1	0.594		100	5.99		6.80		33.40		26.20		10.3		100
RF-Ppro-10-0-1-F	1705051-11	4/27/2017	5/3/2017	10	6.49		102`	70.00		77.40		374.00		283.00		113		101
RF-Ppro-100-0-1-F	1705051-12	4/27/2017	5/3/2017	100	64.9		96	894.00		868.00		4980.00		3170.00		1320		95
RF-Aaff-1-24-1-F	1705051-13	4/28/2017	5/3/2017	1	0.812		99	7.70		9.17		44.70		36.10		13.7		98
RF-Aaff-1-24-1-S	1705051-14	4/28/2017	5/3/2017	1	0.241		103	2.08		2.60		11.90		9.99		4.37		101
RF-Aaff-10-24-1-S	1705051-15	4/28/2017	5/3/2017	10	5.18		95	57.80		61.40		316.00		250.00		100		95
RF-Aaff-100-24-1-S	1705051-16	4/28/2017	5/3/2017	100	29.1		100	415.00		395.00		2240.00		1380.00		609		98
RF-Ppro-1-24-1-F	1705051-17	4/28/2017	5/3/2017	1	0.696		99	6.94		8.44		39.20		30.10		12		98
RF-Ppro-10-24-1-F	1705051-18	4/28/2017	5/3/2017	10	8.96		95	93.60		107.00		518.00		404.00		152		95
RF-Ppro-10-24-2-F	1705051-19	4/28/2017	5/3/2017	10	7.96		100	81.80		95.00		453.00		353.00		135		98
RF-Ppro-100-24-1-F	1705051-20	4/28/2017	5/3/2017	100	53.6		100	730.00		702.00		4060.00		2600.00		1060		99
RF-Ppro-1-24-1-S	1705051-21	4/28/2017	5/3/2017	1	0.33		99	3.4		4.24		18.4		14		6.37		97
RF-Ppro-10-24-1-S	1705051-22	4/28/2017	5/3/2017	10	4.6		99	46.4		54.6		256		200		81.4		98
RF-Ppro-100-24-1-S	1705051-23	4/28/2017	5/3/2017	100	26.6		99	359		345		1970		1270		552		99
RF-Cdub-1-24-1-S	1705051-24	4/28/2017	5/9/2017	1	0.332		96	4.04		3.97		19.8		14.1		6		95
RF-Cdub-10-24-1-S	1705051-25	4/28/2017	5/8/2017	10	4.35		97	48.8		55.4		247		186		76.2		94

Sample ID	MEL ID	sample date	Analysis date	nominal Gx	TPH-Gx (mg/L)		matrix recovery* (%)	benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)		matrix recovery** (%)
RF-Cdub-10-24-2-S	1705051-26	4/28/2017	5/8/2017	10	4.03		97	48.4		48.8		240		175		72		95
RF-Cdub-100-24-1-S	1705051-27	4/28/2017	5/8/2017	100	27.8		98	450		360		2230		1300		569		95
RF-Aaff-1-48-1-F	1705051-28	4/29/2017	5/8/2017	1	0.842		99	7.92		9.65		46.70		38.10		14.7		106
RF-Ppro-1-48-1-F	1705051-29	4/29/2017	5/8/2017	1	0.794		96	8.10		9.93		45.10		35.90		13.9		94
RF-Ppro-10-48-1-F	1705051-30	4/29/2017	5/8/2017	10	9.12		94	96.80		117.00		533.00		413.00		158		93
RF-Aaff-1-72-1-F	1705051-31	4/30/2017	5/8/2017	1	0.839		95	8.79		10.20		48.40		38.30		14.7		94
RF-Ppro-1-72-1-F	1705051-32	4/30/2017	5/8/2017	1	0.674		97	6.31		7.95		37.30		29.90		11.8		96
RF-Ppro-10-72-1-F	1705051-33	4/30/2017	5/9/2017	10	9.45		96	97.80		124.00		543.00		422.00		162		94
RF-Ppro-10-72-2-F	1705051-34	4/30/2017	5/9/2017	10	9.3		96	96.60		114.00		536.00		423.00		160		95
Gas standard	1705051-35	5/2/2017	5/9/2017	50000	48500		95	501000		603000		2640000	J	2110000		788000		
Gas standard	1705051-36	5/2/2017	5/9/2017	50000	48800		92	512000		590000		2700000	J	2150000		796000		
RF-Aaff-1-96-1-F	1705040-1	5/1/2017	5/10/2017	1	0.679		93	6.42		7.88		38		30.30		12.00		95
RF-Aaff-1-96-1-S	1705040-2	5/1/2017	5/10/2017	1	0.178		97	1.85		1	U	7.49		9.66		4.61		99
RF-Ppro-1-96-1-F	1705040-3	5/1/2017	5/10/2017	1	0.889		90	8.79		11.10		50.10		39.20		15.3		91
RF-Ppro-1-96-1-S	1705040-4	5/1/2017	5/10/2017	1	0.379		95	3.79		5.21		19.6		16.9		7.42		96
RF-Ppro-10-96-1-S	1705040-5	5/1/2017	5/10/2017	10	5.77		93	63.60		73.20		333	J	252.00		101		94
RF-Cdub-1-96-1-S	1705040-6	5/1/2017	5/10/2017	1	0.43		93	5.26		5.18		26.4		18.6		7.85		95
RF-Aaff-1-120-1-F	1705040-7	5/2/2017	5/10/2017	1	0.785		96	7.70		9.39		44.40	J	35.40		13.6		98
RF-Ppro-1-120-1-F	1705040-8	5/2/2017	5/10/2017	1	**120**		93	1380		1510		7950	J	5700		2330		95
RF-Aaff-1-144-1-F	1705040-9	5/3/2017	5/10/2017	1	0.855		96	8.39		9.97		48.30		37.90		15		98
RF-Ppro-1-144-1-F	1705040-10	5/3/2017	5/10/2017	1	0.798		94	8.15		9.87		46.00		37.00		13.9		95
RF-Aaff-1-168-1-S	1705040-11	5/4/2017	5/10/2017	1	0.102		98	1.35		1.00	U	4.90		5.82		3.21		99
RF-Ppro-1-168-1-S	1705040-12	5/4/2017	5/10/2017	1	0.216		88	1.73		4.14		5.89	J	14.00		5.9		89

U = not detected
* surrogate recovery for Benzene, 1,4-dibromo-2-methyl-

Table D-3: Results of the NWTPH-Gx final chronic toxicity tests.

Sample ID	MEL ID	sample date	analysis date	nominal	TPH-Gx (mg/L)		matrix recovery** (%)	benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)		matrix recovery** (%)
Ctox-MARINE-BLNK-1	1705066-1	5/10/2017	5/15/2017	0	0.07	U	101	1	U	1	U	1	U	2	U	1	U	100
Ctox-FRESH-BLNK-2	1705066-10	5/10/2017	5/15/2017	0	0.07	U	101	1	U	1	U	1	U	2	U	1	U	99
Ctox-FRESH-BLNK-3	1705066-11	5/10/2017	5/15/2017	0	0.07	U	101	1	U	1	U	1	U	2	U	1	U	100
Ctox-Ppro-2.5-0-1-F	1705066-12	5/10/2017	5/16/2017	2.5	1.97		103	33.1		23.2		110		85.1		33.6		102
Ctox-Ppro-0.63-0-1-F	1705066-13	5/10/2017	5/16/2017	0.63	0.531		98	5.54		5.99		29.6		23.2		9.21		98
Ctox-Ppro-0.31-0-1-F	1705066-14	5/10/2017	5/16/2017	0.31	0.259		98	2.73		2.94		14.2		11.3		4.51		98
Ctox-Ppro-0.31-0-2-F	1705066-15	5/10/2017	5/16/2017	0.31	0.249		98	2.59		2.95		13.6		10.7		4.34		98
Ctox-Ppro-0.16-0-1-F	1705066-16	5/10/2017	5/16/2017	0.16	0.12		98	1.38		1.55		6.32		5.37		2.13		97
Ctox-MARINE-BLNK-2	1705066-2	5/10/2017	5/15/2017	0	0.07	U	100	1	U	1	U	1	U	2	U	1	U	99
Ctox-MARINE-BLNK-3	1705066-3	5/10/2017	5/15/2017	0	0.07	U	101	1	U	1	U	1	U	2	U	1	U	100
Ctox-Aaff-2.5-0-1-F	1705066-4	5/10/2017	5/16/2017	2.5	1.86		101	33.1		22.8		108		83.2		33		98
Ctox-Aaff-0.63-0-1-F	1705066-5	5/10/2017	5/16/2017	0.63	0.522		100	5.17		6.16		28.8		23		9		98
Ctox-Aaff-0.31-0-1-F	1705066-6	5/10/2017	5/16/2017	0.31	0.253		99	2.59		3.03		13.7		11.1		4.36		96
Ctox-Aaff-0.31-0-2-F	1705066-7	5/10/2017	5/16/2017	0.31	0.252		100	2.53		3.14		13.7		10.9		4.36		96
Ctox-Aaff-0.16-0-1-F	1705066-8	5/10/2017	5/16/2017	0.16	0.119		101	1.28		1.51		6.24		5.12		2.06		97
Ctox-FRESH-BLNK-1	1705066-9	5/10/2017	5/15/2017	0	0.07	U	102	1	U	1	U	1	U	2	U	1	U	101
Ctox-Aaff-0.16-24-1-F	1705075-01	5/11/2017	5/16/2017	0.16	0.082		101	0.83	J	0.97	J	3.99		3.42		1.41		101
Ctox-Aaff-0.31-24-1-F	1705075-02	5/11/2017	5/17/2017	0.31	0.188		124	1.76		2.2		9.69		7.88		3.28		103
Ctox-Aaff-0.31-24-2-F	1705075-03	5/11/2017	5/16/2017	0.31	0.186		99	1.76		2.12		9.7		7.88		3.27		99
Ctox-Aaff-0.63-24-1-F	1705075-04	5/11/2017	5/16/2017	0.63	0.416		102	3.78		4.78		22.2		17.6		7.17		101
Ctox-Aaff-2.5-24-1-F	1705075-05	5/11/2017	5/16/2017	2.5	1.75		101	17.6		19.7		96.6		75.6		29.8		101
Ctox-Ppro-0.16-24-1-F	1705075-06	5/11/2017	5/16/2017	0.16	0.07	U	103	1	U	1	U	1	U	2	U	1	U	103
Ctox-Ppro-0.63-24-1-F	1705075-07	5/11/2017	5/16/2017	0.63	0.443		100	4.24		5.25		24.3		18.9		7.64		100
Ctox-Ppro-0.63-24-2-F	1705075-08	5/11/2017	5/16/2017	0.63	0.45		99	4.29		5.65		24.7		18.9		7.77		99
Ctox-Ppro-2.5-24-1-F	1705075-09	5/11/2017	5/16/2017	2.5	2.18		106	20.8		28		120		91.7		36.5		106
Ctox-Aaff-0.16-24-1-S	1705075-10	5/11/2017	5/16/2017	0.16	0.07	J	104	0.55	J	0.64	J	2.4		2.25		0.96	J	103

Sample ID	MEL ID	sample date	analysis date	nominal	TPH-Gx (mg/L)		matrix recovery** (%)	benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)		matrix recovery** (%)
Ctox-Aaff-0.31-24-1-S	1705075-11	5/11/2017	5/16/2017	0.31	0.077		103	0.73	J	0.93	J	3.63		3.24		1.44		103
Ctox-Aaff-0.63-24-1-S	1705075-12	5/11/2017	5/16/2017	0.63	0.171		103	1.61		1.69		8.35		7.5		3.26		103
Ctox-Aaff-2.5-24-1-S	1705075-13	5/11/2017	5/16/2017	2.5	0.635		103	5.9		5.05		30.7		26.5		12.1		101
Ctox-Ppro-0.16-24-1-S	1705075-14	5/11/2017	5/17/2017	0.16	0.07	J	103	0.71	J	0.82	J	2.77		2.44		1.27		102
Ctox-Ppro-0.63-24-1-S	1705075-15	5/11/2017	5/17/2017	0.63	0.226		101	2.37		2.94		12.8		10		4.49		100
Ctox-Ppro-2.5-24-1-S	1705075-16	5/11/2017	5/17/2017	2.5	18.1		100	169		233		1250	E	843	E	342	E	89
Ctox-Ppro-2.5-24-1-S	1705075-16RE1	5/11/2017	6/13/2017	2.5	0.174		102	1.85		2.11		9.78		7.52		3.23		101
Ctox-PERRIER-BLNK-1	1705075-17	5/11/2017	5/17/2017		0.07	U	102	1	U	1	U	1	U	2	U	1	U	96
Ctox-PERRIER-BLNK-2	1705075-18	5/11/2017	5/17/2017		0.07	U	103	1	U	1	U	1	U	2	U	1	U	99
Ctox-PERRIER-BLNK-3	1705075-19	5/11/2017	5/17/2017		0.07	U	102	1	U	1	U	1	U	2	U	1	U	97
Ctox-Cdub-0.16-0-1-F	1705075-20	5/11/2017	5/17/2017	0.16	0.07	U	99	1	U	1	U	1	U	2	U	1	U	94
Ctox-Cdub-0.63-0-1-F	1705075-21	5/11/2017	5/17/2017	0.63	0.07	U	107	1	U	1	U	1	U	2	U	1	U	101
Ctox-Cdub-0.63-0-2-F	1705075-22	5/11/2017	5/17/2017	0.63	0.07	U	104	1	U	1	U	1	U	2	U	1	U	99
Ctox-Cdub-2.5-0-1-F	1705075-23	5/11/2017	5/17/2017	2.5	6.84		102	14.6		106		288		387		165		97
Ctox-Cdub-2.5-0-1-F	1705075-23RE1	5/11/2017	6/14/2017	2.5	0.057		99	0.61		0.44		2.04		1.63		1.48		97
Ctox-Aaff-0.16-48-1-F	1705075-24	5/12/2017	5/17/2017	0.16	0.082		106	0.83	J	0.99	J	4		3.54		1.46		99
Ctox-Aaff-0.31-48-1-F	1705075-25	5/12/2017	5/17/2017	0.31	0.174		104	1.73		2.02		9.28		7.62		3.16		99
Ctox-Aaff-0.63-48-1-F	1705075-26	5/12/2017	5/17/2017	0.63	0.384		104	3.65		4.29		21.2		16.7		6.89		99
Ctox-Aaff-2.5-48-1-F	1705075-27	5/12/2017	5/17/2017	2.5	2.16		103	20		38.2		145		140		59.1		99
Ctox-Ppro-0.16-48-1-F	1705075-28	5/12/2017	5/17/2017	0.16	0.089		103	0.93	J	1.16		4.35		3.87		1.59		98
Ctox-Ppro-0.63-48-1-F	1705075-29	5/12/2017	5/18/2017	0.63	0.445		103	4.36		5.06		24.8		19.7		7.85		98
Ctox-Ppro-2.5-48-1-F	1705075-30	5/12/2017	5/18/2017	2.5	2.79		103	21.6		31.1		132		118		47.4		98
Ctox-Ppro-2.5-48-1-F	1705075-30RE1	5/12/2017	6/13/2017	2.5	0.352		101	3.85		4.47		21.2		15.6		6.47		99
Ctox-Cdub-0.16-24-1-F	1705075-31	5/12/2017	5/18/2017	0.16	0.07	J	104	0.87	J	0.87	J	3.7		2.95		1.24		98
Ctox-Cdub-0.63-24-1-F	1705075-32	5/12/2017	5/18/2017	0.63	0.209		104	2.6		2.69		13.3		9.29		4.07		97
Ctox-Cdub-0.63-24-2-F	1705075-33	5/12/2017	5/18/2017	0.63	0.209		104	2.58		2.55		13.2		9.34		4.03		99
Ctox-Cdub-2.5-24-1-F	1705075-34	5/12/2017	5/18/2017	2.5	1.17		101	12		15.7		68.2		53.2		24.1		94

Sample ID	MEL ID	sample date	analysis date	nominal	TPH-Gx (mg/L)		matrix recovery** (%)	benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)		matrix recovery** (%)
Ctox-Cdub-0.16-24-1-S	1705075-35	5/12/2017	5/18/2017	0.16	0.07	U	105	1	U	1	U	1	U	2	U	1	U	99
Ctox-Cdub-0.63-24-1-S	1705075-36	5/12/2017	5/18/2017	0.63	0.07	U	105	1	U	1	U	1	U	2	U	1	U	98
Ctox-Cdub-2.5-24-1-S	1705075-37	5/12/2017	5/18/2017	2.5	0.35	J	106	2	J	3.65	J	9.6		11.4		4.95	J	99
Ctox-Aaff-0.16-72-1-F	1705075-38	5/13/2017	5/18/2017	0.16	0.092		106	1		1.23		5		4.17		1.69		100
Ctox-Aaff-0.31-72-1-F	1705075-39	5/13/2017	5/18/2017	0.31	0.22		104	2.14		2.62		11.9		9.48		3.9		99
Ctox-Aaff-0.31-72-2-F	1705075-40	5/13/2017	5/18/2017	0.31	0.217		102	2.13		2.5		11.9		9.46		3.83		96
Ctox-Aaff-0.63-72-1-F	1705075-41	5/13/2017	5/18/2017	0.63	0.472		103	4.51		5.45		26.3		20.8		8.31		97
Ctox-Aaff-2.5-72-1-F	1705075-42	5/13/2017	5/18/2017	2.5	1.1		104	13.3		16.6		75.2		55.8		24		97
Ctox-Ppro-0.16-72-1-F	1705075-43	5/13/2017	5/18/2017	0.16	0.081		104	0.86	J	1.09		3.84		3.44		1.45		97
Ctox-Ppro-0.63-72-1-F	1705075-44	5/13/2017	5/18/2017	0.63	0.447		102	4.38		5.11		25		19.8		7.93		96
Ctox-Ppro-0.63-72-2-F	1705075-45	5/13/2017	5/18/2017	0.63	0.44		103	4.28		4.77		24.4		19.6		7.77		97
Ctox-Ppro-2.5-72-1-F	1705075-46	5/13/2017	5/18/2017	2.5	1.83		95	18.8		22.8		110		82.2		33.3		93
Ctox-Cdub-0.16-48-1-F	1705075-47	5/13/2017	5/19/2017	0.16	0.07	J	101	0.85	J	0.88	J	3.69		2.86		1.21		98
Ctox-Cdub-0.63-48-1-F	1705075-48	5/13/2017	5/19/2017	0.63	0.26		100	3.16		3.1		16.4		11.3		4.93		99
Ctox-Cdub-2.5-48-1-F	1705075-49	5/13/2017	5/19/2017	2.5	0.817		101	11.4		10.8		55.4		34.6		16		98
Ctox-Aaff-0.16-96-1-F	1705075-50	5/14/2017	5/19/2017	0.16	0.083		103	0.87	J	1.02		4.04		3.52		1.47		100
Ctox-Aaff-0.31-96-1-F	1705075-51	5/14/2017	5/19/2017	0.31	0.188		102	1.83		2.26		9.86		7.99		3.33		101
Ctox-Aaff-0.31-96-2-F	1705075-52	5/14/2017	5/19/2017	0.31	0.19		101	1.84		2.21		10		8.2		3.4		107
Ctox-Aaff-0.63-96-1-F	1705075-53	5/14/2017	5/19/2017	0.63	0.417		101	3.94		4.6		22.8		18.3		7.4		99
Ctox-Aaff-2.5-96-1-F	1705075-54	5/14/2017	5/19/2017	2.5	1.25		101	17.8		18.6		101		65		29.5		98
Ctox-Ppro-0.16-96-1-F	1705075-55	5/14/2017	5/19/2017	0.16	0.094		102	0.99	J	1.19		4.66		4.04		1.64		99
Ctox-Ppro-0.63-96-1-F	1705075-56	5/14/2017	5/19/2017	0.63	0.467		99	4.72		5.53		26.5		20.9		8.26		94
Ctox-Ppro-0.63-96-2-F	1705075-57	5/14/2017	5/19/2017	0.63	0.476		100	4.8		5.76		27		21.2		8.32		95
Ctox-Ppro-2.5-96-1-F	1705075-58	5/14/2017	5/22/2017	2.5	1.94		100	22.6		26		122		87.3		35		96
Ctox-Aaff-0.16-96-1-S	1705075-59	5/14/2017	5/22/2017	0.16	0.07	U	101	1	U	1	U	1	U	2	U	1	U	98
Ctox-Aaff-0.31-96-1-S	1705075-60	5/14/2017	5/22/2017	0.31	0.07	U	102	1	U	1	U	1	U	2	U	1	U	98
Ctox-Aaff-0.63-96-1-S	1705075-61	5/14/2017	5/22/2017	0.63	0.07	U	101	1	U	1	U	1	U	2	U	1	U	97

Sample ID	MEL ID	sample date	analysis date	nominal	TPH-Gx (mg/L)		matrix recovery** (%)	benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)		matrix recovery** (%)
Ctox-Aaff-2.5-96-1-S	1705075-62	5/14/2017	5/25/2017	2.5	0.208		96	2.62		0.72	J	13.2		12.6		5.62		93
Ctox-Ppro-0.16-96-1-S	1705075-63	5/14/2017	5/22/2017	0.16	0.07	U	100	1	U	1	U	1	U	2	U	1	U	96
Ctox-Ppro-0.63-96-1-S	1705075-64	5/14/2017	5/22/2017	0.63	0.13		99	1.39		1.7		7.61		5.37		2.86		95
Ctox-Ppro-2.5-96-1-S	1705075-65	5/14/2017	5/25/2017	2.5	0.07	U	97	1	U	1	U	1	U	2	U	1	U	94
Ctox-Cdub-0.16-72-1-F	1705075-66	5/14/2017	5/22/2017	0.16	0.07	J	101	0.52	J	0.58	J	1.95		1.77	J	0.79	J	97
Ctox-Cdub-0.63-72-1-F	1705075-67	5/14/2017	5/22/2017	0.63	0.158		99	1.79		1.93		9.38		6.92		3.04		94
Ctox-Cdub-0.63-72-2-F	1705075-68	5/14/2017	5/22/2017	0.63	0.168		98	1.91		2.05		10.1		7.41		3.26		95
Ctox-Cdub-2.5-72-1-F	1705075-69	5/14/2017	5/22/2017	2.5	0.504		100	7.25		7.5		36.2		22.6		10.8		96
Ctox-Aaff-0.16-120-1-F	1705085-01	5/15/2017	5/23/2017	0.16	0.159		109	1.54		2.06		8.48		6.79		2.69		102
Ctox-Aaff-0.31-120-1-F	1705085-02	5/15/2017	5/23/2017	0.31	0.297		106	2.71		3.46		16.1		12.9		5.04		99
Ctox-Aaff-0.63-120-1-F	1705085-03	5/15/2017	5/23/2017	0.63	0.599		105	5.49		6.54		33.2		26.7		10.2		98
Ctox-Aaff-2.5-120-1-F	1705085-04	5/15/2017	5/23/2017	2.5	2		106	21.4		29.4		128		96.5		39.2		101
Ctox-Ppro-0.16-120-1-F	1705085-05	5/15/2017	5/23/2017	0.16	0.164		102	1.72		2.08		8.98		7.18		2.84		95
Ctox-Ppro-0.63-120-1-F	1705085-06	5/15/2017	5/23/2017	0.63	0.567		103	5.73		7.06		32.6		24.9		9.88		96
Ctox-Ppro-2.5-120-1-F	1705085-07	5/15/2017	5/23/2017	2.5	2.08		106	22.8		27.4		125		92		37		99
Ctox-Cdub-0.16-96-1-F	1705085-08	5/15/2017	5/23/2017	0.16	0.1		107	1.19		1.21		5.82		4.35		1.84		100
Ctox-Cdub-0.63-96-1-F	1705085-09	5/15/2017	5/23/2017	0.63	0.284		106	3.28		3.48		17.5		12.3		5.33		99
Ctox-Cdub-0.63-96-2-F	1705085-10	5/15/2017	5/23/2017	0.63	0.297		105	3.46		3.46		18.4		13.1		5.59		98
Ctox-Cdub-2.5-96-1-F	1705085-11	5/15/2017	5/23/2017	2.5	0.769		104	10.9		11.6		55		34		16.2		98
Ctox-Cdub-0.16-96-1-S	1705085-12	5/15/2017	5/23/2017	0.16	0.036	J	108	0.47	J	0.54	J	1.5		1.73	J	0.79	J	102
Ctox-Cdub-0.63-96-1-S	1705085-13	5/15/2017	5/24/2017	0.63	0.091		101	1.16		1.2		5.17		4.03		1.86		98
Ctox-Cdub-2.5-96-1-S	1705085-14	5/15/2017	5/25/2017	2.5	0.343		97	4.26		4.12		21.7		14.8		6.71		93
Ctox-Aaff-0.16-144-1-F	1705085-15	5/16/2017	5/24/2017	0.16	0.167		102	1.72		2.14		9.09		7.36		2.87		99
Ctox-Aaff-0.31-144-1-F	1705085-16	5/16/2017	5/24/2017	0.31	0.314		101	3.14		3.96		17.6		13.8		5.39		97
Ctox-Aaff-0.31-144-2-F	1705085-17	5/16/2017	5/24/2017	0.31	0.315		100	3.12		3.95		17.5		13.8		5.39		98
Ctox-Aaff-0.63-144-1-F	1705085-18	5/16/2017	5/24/2017	0.63	0.633		99	6.29		7.92		36		28.4		11		97
Ctox-Aaff-2.5-144-1-F	1705085-19	5/16/2017	5/24/2017	2.5	1.9		101	22.4		26.9		127		98.4		38.8		99

Sample ID	MEL ID	sample date	analysis date	nominal	TPH-Gx (mg/L)		matrix recovery** (%)	benzene (ug/L)		ethylbenzene (ug/L)		toluene (ug/L)		m,p-xylene (ug/L)		o-xylene (ug/L)		matrix recovery** (%)
Ctox-Ppro-0.16-144-1-F	1705085-20	5/16/2017	5/24/2017	0.16	0.13		101	1.48		1.56		7.16		5.65		2.22		97
Ctox-Ppro-0.63-144-1-F	1705085-21	5/16/2017	5/24/2017	0.63	0.533		97	5.96		6.34		31.8		24		9.26		95
Ctox-Ppro-0.63-144-2-F	1705085-22	5/16/2017	5/24/2017	0.63	0.544		98	6.02		6.49		32.2		24.4		9.43		96
Ctox-Ppro-2.5-144-1-F	1705085-23	5/16/2017	5/24/2017	2.5	1.69		97	21		20.8		107		75.4		30.4		94
Ctox-Cdub-0.16-120-1-F	1705085-24	5/16/2017	5/24/2017	0.16	0.135		98	1.58		1.82		7.8		5.77		2.44		96
Ctox-Cdub-0.63-120-1-F	1705085-25	5/16/2017	5/24/2017	0.63	0.524		95	5.98		6.46		32.1		23		9.42		90
Ctox-Cdub-2.5-120-1-F	1705085-26	5/16/2017	5/24/2017	2.5	1.8		94	21.2		21.6		113		80.2		32.8		90
Ctox-Aaff-0.16-168-1-S	1705085-27	5/17/2017	5/24/2017	0.16	0.07	U	100	1	U	1	U	1	U	2	U	1	U	95
Ctox-Aaff-0.31-168-1-S	1705085-28	5/17/2017	5/24/2017	0.31	0.07	U	101	1	U	1	U	1	U	2	U	1	U	101
Ctox-Aaff-0.63-168-1-S	1705085-29	5/17/2017	5/24/2017	0.63	0.07	J	98	0.27	J	0.29	J	0.56	J	0.84	J	0.33	J	94
Ctox-Aaff-2.5-168-1-S	1705085-30	5/17/2017	5/25/2017	2.5	0.061	J	99	0.57	J	0.55	J	2.88		2.95		1.21		95
Ctox-Ppro-0.16-168-1-S	1705085-31	5/17/2017	5/25/2017	0.16	0.07	U	98	1	U	1	U	1	U	2	U	1	U	94
Ctox-Ppro-0.63-168-1-S	1705085-32	5/17/2017	5/25/2017	0.63	0.042	J	94	0.5	J	0.63	J	1.95		1.84	J	0.9	J	91
Ctox-Ppro-2.5-168-1-S	1705085-33	5/17/2017	5/25/2017	2.5	0.437		98	4.9	J	5.75		23.4		18.1		7.95		94
Ctox-Cdub-0.16-144-1-F	1705085-34	5/17/2017	5/25/2017	0.16	0.132		96	1.35		1.58		6.83		5.51		2.38		93
Ctox-Cdub-0.63-144-1-F	1705085-35	5/17/2017	5/25/2017	0.63	0.425		95	3.98		4.94		22.9		18		7.83		91
Ctox-Cdub-0.63-144-2-F	1705085-36	5/17/2017	5/25/2017	0.63	0.455		95	4.27		5.09		24.6		19.4		8.32		90
Ctox-Cdub-2.5-144-1-F	1705085-37	5/17/2017	5/25/2017	2.5	1.7		92	17		19.4		95.4		72		32.2		88
Ctox-Cdub-0.16-168-1-S	1705086-01	5/18/2017	5/25/2017	0.16	0.126		104	1.46		1.6		7.17		5.38		2.27		98
Ctox-Cdub-0.63-168-1-S	1705086-02	5/18/2017	5/25/2017	0.63	0.526		103	5.42		6.44		31.6		22.9		9.55		97
Ctox-Cdub-2.5-168-1-S	1705086-03	5/18/2017	5/26/2017	2.5	1.19		99	14		14.8		69.6		50.8		21.5		97
Ctox-Spur-0.16-0-1-F	1705086-04	5/18/2017	5/25/2017	0.16	0.159		104	1.59		1.98		8.33		6.73		2.72		98
Ctox-Spur-0.31-0-1-F	1705086-05	5/18/2017	5/25/2017	0.31	0.322		103	3.14		4.27		17.5		13.6		5.51		98
Ctox-Spur-0.31-0-2-F	1705086-06	5/18/2017	5/25/2017	0.31	0.316		104	3.05		3.7		17		13.6		5.4		99
Ctox-Spur-0.63-0-1-F	1705086-07	5/18/2017	5/26/2017	0.63	0.61		104	5.88		7.62		33.9		26.8		10.5		98
Ctox-Spur-2.5-0-1-F	1705086-08	5/18/2017	5/26/2017	2.5	2.31		102	23.6		26.5		128		98.3		39.9		99

U = analyte not detected; J = analyte detected, result is an estimate; * surrogate recovery for Benzene, 1,4-dibromo-2-methyl-

Table D-4: Sampling QC results for NWTPH-Gx.

Sample ID	MEL ID	sample date	analysis date	TPH-Gx (mg/L)		RPD	benzene		RPD	ethylbenzene		RPD	toluene		RPD	m,p-xylene (ug/L)		RPD	o-xylene (ug/L)		RPD
RF-Ppro-10-24-1-F	1705051-18	4/28/2017	5/3/2017	8.96		0.12	93.6		0.13	107		0.12	518		0.13	404		0.13	152		0.12
RF-Ppro-10-24-2-F	1705051-19	4/28/2017	5/3/2017	7.96			81.8			95			453			353			135		
RF-Cdub-10-24-1-S	1705051-25	4/28/2017	5/8/2017	4.35		0.08	48.8		0.01	55.4		0.13	247		0.03	186		0.06	76.2		0.06
RF-Cdub-10-24-2-S	1705051-26	4/28/2017	5/8/2017	4.03			48.4			48.8			240			175			72		
RF-Ppro-10-72-1-F	1705051-33	4/30/2017	5/9/2017	9.45		0.02	97.8		0.01	124		0.08	543		0.01	422		0.00	162		0.01
RF-Ppro-10-72-2-F	1705051-34	4/30/2017	5/9/2017	9.3			96.6			114			536			423			160		
Ctox-Ppro-0.31-0-1-F	1705066-14	5/10/2017	5/16/2017	0.259		0.04	2.73		0.05	2.94		0.00	14.2		0.04	11.3		0.05	4.51		0.04
Ctox-Ppro-0.31-0-2-F	1705066-15	5/10/2017	5/16/2017	0.249			2.59			2.95			13.6			10.7			4.34		
Ctox-Aaff-0.31-0-1-F	1705066-6	5/10/2017	5/16/2017	0.253		0.00	2.59		0.02	3.03		0.04	13.7		0.00	11.1		0.02	4.36		0.00
Ctox-Aaff-0.31-0-2-F	1705066-7	5/10/2017	5/16/2017	0.252			2.53			3.14			13.7			10.9			4.36		
Ctox-Aaff-0.31-24-1-F	1705075-02	5/11/2017	5/17/2017	0.188		0.01	1.76		0.00	2.2		0.04	9.69		0.00	7.88		0.00	3.28		0.00
Ctox-Aaff-0.31-24-2-F	1705075-03	5/11/2017	5/16/2017	0.186			1.76			2.12			9.7			7.88			3.27		
Ctox-Ppro-0.63-24-1-F	1705075-07	5/11/2017	5/16/2017	0.443		0.02	4.24		0.01	5.25		0.07	24.3		0.02	18.9		0.00	7.64		0.02
Ctox-Ppro-0.63-24-2-F	1705075-08	5/11/2017	5/16/2017	0.45			4.29			5.65			24.7			18.9			7.77		
Ctox-Cdub-0.63-0-1-F	1705075-21	5/11/2017	5/17/2017	0.07	U	0.00	1	U	0.00	1	U	0.00	1	U	0.00	2	U	0.00	1	U	0.00
Ctox-Cdub-0.63-0-2-F	1705075-22	5/11/2017	5/17/2017	0.07	U		1	U		1	U		1	U		2	U		1	U	
Ctox-Cdub-0.63-24-1-F	1705075-32	5/12/2017	5/18/2017	0.21		0.00	2.6		0.01	2.69		0.05	13.3		0.01	9.29		0.01	4.07		0.01
Ctox-Cdub-0.63-24-2-F	1705075-33	5/12/2017	5/18/2017	0.21			2.58			2.55			13.2			9.34			4.03		
Ctox-Aaff-0.31-72-1-F	1705075-39	5/13/2017	5/18/2017	0.22		0.01	2.14		0.00	2.62		0.05	11.9		0.00	9.48		0.00	3.9		0.02
Ctox-Aaff-0.31-72-2-F	1705075-40	5/13/2017	5/18/2017	0.22			2.13			2.5			11.9			9.46			3.83		
Ctox-Ppro-0.63-72-1-F	1705075-44	5/13/2017	5/18/2017	0.45		0.02	4.38		0.02	5.11		0.07	25		0.02	19.8		0.01	7.93		0.02
Ctox-Ppro-0.63-72-2-F	1705075-45	5/13/2017	5/18/2017	0.44			4.28			4.77			24.4			19.6			7.77		
Ctox-Aaff-0.31-96-1-F	1705075-51	5/14/2017	5/19/2017	0.19		0.01	1.83		0.01	2.26		0.02	9.86		0.01	7.99		0.03	3.33		0.02
Ctox-Aaff-0.31-96-2-F	1705075-52	5/14/2017	5/19/2017	0.19			1.84			2.21			10			8.2			3.4		
Ctox-Ppro-0.63-96-1-F	1705075-56	5/14/2017	5/19/2017	0.47		0.02	4.72		0.02	5.53		0.04	26.5		0.02	20.9		0.01	8.26		0.01

Sample ID	MEL ID	sample date	analysis date	TPH-Gx (mg/L)		RPD	benzene		RPD	ethylbenzene		RPD	toluene		RPD	m,p-xylene (ug/L)		RPD	o-xylene (ug/L)		RPD
Ctox-Ppro-0.63-96-2-F	1705075-57	5/14/2017	5/19/2017	0.48			4.8			5.76			27			21.2			8.32		
Ctox-Cdub-0.63-72-1-F	1705075-67	5/14/2017	5/22/2017	0.16		0.06	1.79		0.06	1.93		0.06	9.38		0.07	6.92		0.07	3.04		0.07
Ctox-Cdub-0.63-72-2-F	1705075-68	5/14/2017	5/22/2017	0.17			1.91			2.05			10.1			7.41			3.26		
Ctox-Cdub-0.63-96-1-F	1705085-09	5/15/2017	5/23/2017	0.28		0.04	3.28		0.05	3.48		0.01	17.5		0.05	12.3		0.06	5.33		0.05
Ctox-Cdub-0.63-96-2-F	1705085-10	5/15/2017	5/23/2017	0.30			3.46			3.46			18.4			13.1			5.59		
Ctox-Aaff-0.31-144-1-F	1705085-16	5/16/2017	5/24/2017	0.31		0.00	3.14		0.01	3.96		0.00	17.6		0.01	13.8		0.00	5.39		0.00
Ctox-Aaff-0.31-144-2-F	1705085-17	5/16/2017	5/24/2017	0.32			3.12			3.95			17.5			13.8			5.39		
Ctox-Ppro-0.63-144-1-F	1705085-21	5/16/2017	5/24/2017	0.53		0.02	5.96		0.01	6.34		0.02	31.8		0.01	24		0.02	9.26		0.02
Ctox-Ppro-0.63-144-2-F	1705085-22	5/16/2017	5/24/2017	0.54			6.02			6.49			32.2			24.4			9.43		
Ctox-Cdub-0.63-144-1-F	1705085-35	5/17/2017	5/25/2017	0.43		0.07	3.98		0.07	4.94		0.03	22.9		0.07	18		0.07	7.83		0.06
Ctox-Cdub-0.63-144-2-F	1705085-36	5/17/2017	5/25/2017	0.46			4.27			5.09			24.6			19.4			8.32		
Ctox-Spur-0.31-0-1-F	1705086-05	5/18/2017	5/25/2017	0.32		0.02	3.14		0.03	4.27		0.14	17.5		0.03	13.6		0.00	5.51		0.02
Ctox-Spur-0.31-0-2-F	1705086-06	5/18/2017	5/25/2017	0.32			3.05			3.7			17			13.6			5.4		

Table D-5: Laboratory quality control samples.

Sample ID	MEL ID	MEL work order / batch	analysis date	TPH-Gx (mg/L)		benzene		ethylbenzene		toluene		m,p-xylene (ug/L)		o-xylene (ug/L)	
Mixing															
Method Blnk	B17D140-BLK1	1704045	4/24/2017	0.07	U										
LCS	B17D140-BS1	1704045	4/24/2017	91	%										
LCS Dup	B17D140-BSD1	1704045	4/24/2017	92	%										
Method Blnk	B17D153-BLK1	1704045	4/24/2017			1	U	1	U	1	U	2	U	1	U
LCS	B17D153-BS1	1704045	4/24/2017			98	%	92	%	97	%	101	%	97	%
LCS Dup	B17D153-BSD1	1704045	4/24/2017			90	%	93	%	88	%	87	%	89	%
Range Finding															
Method Blnk	B17E053-BLK1	1705051	5/2/2017	0.07	U										
LCS	B17E053-BS1	1705051	5/2/2017	96	%										
LCS Dup	B17E053-BSD1	1705051	5/2/2017	101	%										
Method Blnk	B17E053-BLK2	1705051	5/3/2017	0.07	U										
LCS	B17E053-BS2	1705051	5/3/2017	98	%										
LCS Dup	B17E053-BSD2	1705051	5/3/2017	98	%										
Method Blnk	B17E061-BLK1	1705051	5/8/2017	0.07	U										
LCS	B17E061-BS1	1705051	5/8/2017	97	%										
LCS Dup	B17E061-BSD1	1705051	5/8/2017	102	%										
Method Blnk	B17E071-BLK1	1705051	5/9/2017	0.07	U										
Method Blnk	B17E071-BLK2	1705051	5/9/2017	0.07	U										
LCS	B17E071-BS1	1705051	5/9/2017	101	%										
LCS Dup	B17E071-BSD1	1705051	5/9/2017	95	%										
Method Blnk	B17E076-BLK1	1705051	5/9/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
Method Blnk	B17E076-BLK2	1705051	5/10/2017	0.07	U										
LCS	B17E076-BS1	1705051	5/9/2017	101	%	106	%	105	%	117	%	103	%	103	%
LCS Dup	B17E076-BSD1	1705051	5/9/2017	95	%	102	%	105	%	102	%	101	%	102	%
Method Blnk	B17E086-BLK1	1705040	5/2/2017			1	U	1	U	1	U	2	U	1	U

Sample ID	MEL ID	MEL work order / batch	analysis date	TPH-Gx (mg/L)		benzene		ethylbenzene		toluene		m,p-xylene (ug/L)		o-xylene (ug/L)	
Method Blnk	B17E086-BLK2	1705040	5/3/2017			1	U	1	U	1	U	2	U	1	U
LCS	B17E086-BS1	1705040	5/2/2017			104	%	107	%	104	%	103	%	104	%
LCS Dup	B17E086-BS2	1705040	5/3/2017			96	%	98	%	97	%	97	%	97	%
LCS	B17E086-BSD1	1705040	5/2/2017			98	%	100	%	97	%	97	%	98	%
LCS Dup	B17E086-BSD2	1705040	5/3/2017			100	%	104	%	100	%	100	%	100	%
Method Blnk	B17E091-BLK1	1705040	5/8/2017			1	U	1	U	1	U	2	U	1	U
LCS	B17E091-BS1	1705040	5/8/2017			97	%	97	%	96	%	97	%	99	%
LCS Dup	B17E091-BSD1	1705040	5/8/2017			100	%	104	%	100	%	99	%	101	%
Method Blnk	B17E092-BLK1	1705040	5/9/2017			1	U	1	U	1	U	2	U	1	U
LCS	B17E092-BS1	1705040	5/9/2017			106	%	104	%	117	%	103	%	103	%
LCS Dup	B17E092-BSD1	1705040	5/9/2017			102	%	108	%	102	%	100	%	102	%
Final Toxicity Test															
Method Blnk	B17E117-BLK1	1705066	5/15/2017	0.07	U										
LCS	B17E117-BS1	1705066	5/15/2017	102	%										
LCS Dup	B17E117-BSD1	1705066	5/15/2017	100	%										
Method Blnk	B17E139-BLK1	1705066	5/15/2017			1	U	1	U	1	U	2	U	1	U
LCS	B17E139-BS1	1705066	5/15/2017			100	%	103	%	101	%	103	%	103	%
LCS Dup	B17E139-BSD1	1705066	5/15/2017			104	%	110	%	105	%	105	%	106	%
Method Blnk	B17E193-BLK1	1705075	5/16/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E193-BS1	1705075	5/16/2017	99	%	106	%	113	%	108	%	108	%	109	%
LCS Dup	B17E193-BSD1	1705075	5/16/2017	103	%	101	%	103	%	102	%	104	%	104	%
Method Blnk	B17E194-BLK1	1705075	5/17/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E194-BS1	1705075	5/17/2017	101	%	97	%	105	%	99	%	100	%	102	%
LCS Dup	B17E194-BSD1	1705075	5/17/2017	105	%	104	%	112	%	106	%	107	%	109	%
Method Blnk	B17E197-BLK1	1705075	5/18/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E197-BS1	1705075	5/18/2017	101	%	98	%	104	%	100	%	100	%	102	%
LCS Dup	B17E197-BSD1	1705075	5/18/2017	97	%	103	%	110	%	105	%	106	%	106	%
Method Blnk	B17E201-BLK1	1705075	5/18/2017	0.07	U	1	U	1	U	1	U	2	U	1	U

Sample ID	MEL ID	MEL work order / batch	analysis date	TPH-Gx (mg/L)		benzene		ethylbenzene		toluene		m,p-xylene (ug/L)		o-xylene (ug/L)	
LCS	B17E201-BS1	1705075	5/18/2017	98	%	99	%	108	%	103	%	106	%	107	%
LCS Dup	B17E201-BSD1	1705075	5/18/2017	103	%	95	%	104	%	98	%	100	%	102	%
Method Blnk	B17E202-BLK1	1705075	5/22/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E202-BS1	1705075	5/22/2017	92	%	95	%	90	%	90	%	88	%	92	%
LCS Dup	B17E202-BSD1	1705075	5/22/2017	88	%	96	%	91	%	91	%	89	%	93	%
Method Blnk	B17E214-BLK1	1705075	5/24/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E214-BS1	1705075	5/24/2017	100	%	103	%	105	%	100	%	97	%	100	%
LCS Dup	B17E214-BSD1	1705075	5/24/2017	103	%	108	%	107	%	104	%	103	%	104	%
Method Blnk	B17E203-BLK1	1705085	5/23/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E203-BS1	1705085	5/23/2017	102	%	107	%	100	%	102	%	104	%	102	%
LCS Dup	B17E203-BSD1	1705085	5/23/2017	96	%	106	%	107	%	102	%	99	%	101	%
Method Blnk	B17E205-BLK1	1705085	5/24/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E205-BS1	1705085	5/24/2017	95	%	110	%	108	%	106	%	105	%	105	%
LCS Dup	B17E205-BSD1	1705085	5/24/2017	97	%	107	%	106	%	104	%	101	%	103	%
Method Blnk	B17E207-BLK1	1705085	5/24/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E207-BS1	1705085	5/24/2017	100	%	103	%	101	%	99	%	99	%	99	%
LCS Dup	B17E207-BSD1	1705085	5/24/2017	103	%	108	%	107	%	104	%	104	%	103	%
Method Blnk	B17E212-BLK1	1705086	5/25/2017	0.07	U	1	U	1	U	1	U	2	U	1	U
LCS	B17E212-BS1	1705086	5/25/2017	95	%	104	%	109	%	104	%	101	%	100	%
LCS Dup	B17E212-BSD1	1705086	5/25/2017	98	%	100	%	99	%	97	%	96	%	97	%

Appendix E. NWTPH-Dx Analytical Results

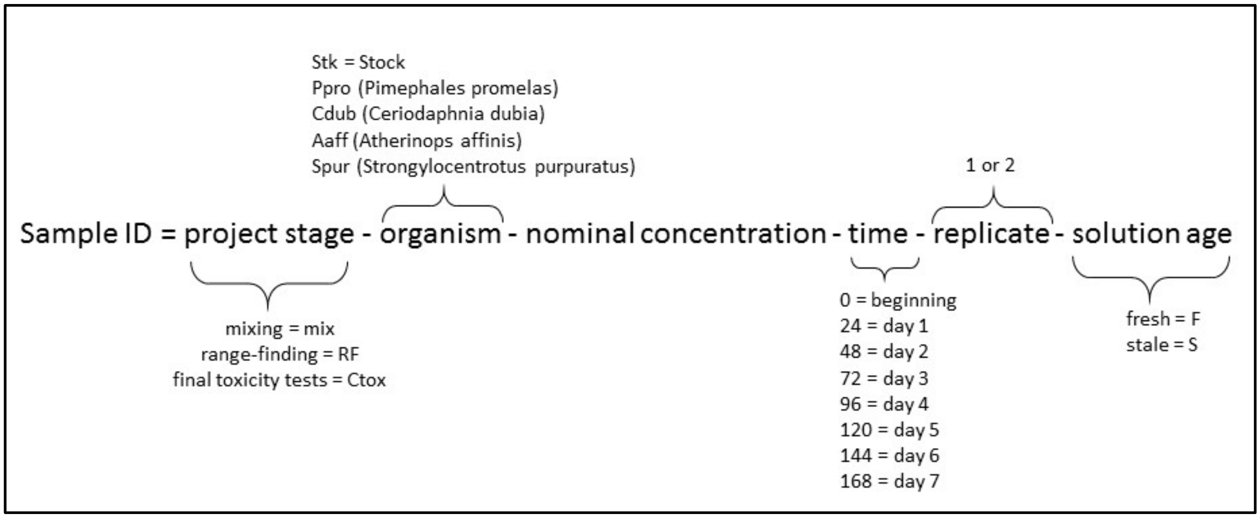

Table E-1: Results of the NWTPH-Dx mixing trials.

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% recovery
Dx-mix-stk-0-0-1	1705087-01	5/25/2017	5/31/2017	0	0.05	U	95
Dx-mix-stk-0-0-2	1705087-02	5/25/2017	5/31/2017	0	0.05	U	91
Dx-mix-stk-0-0-3	1705087-03	5/25/2017	5/31/2017	0	0.05	U	92
Dx-mix-stk-0.1-0-1	1705087-04	5/25/2017	5/31/2017	0.1	0.05	U	98
Dx-mix-stk-0.1-0-2	1705087-05	5/25/2017	5/31/2017	0.1	0.05	U	102
Dx-mix-stk-0.1-0-3	1705087-06	5/25/2017	5/31/2017	0.1	0.05	U	86
Dx-mix-stk-1-0-1	1705087-07	5/25/2017	5/31/2017	1	0.15	U	106
Dx-mix-stk-1-0-2	1705087-08	5/25/2017	5/31/2017	1	0.15	U	91
Dx-mix-stk-1-0-3	1705087-09	5/25/2017	5/31/2017	1	0.15	U	100
Dx-mix-stk-10-0-1	1705087-10	5/25/2017	6/1/2017	10	0.27		96
Dx-mix-stk-10-0-2	1705087-11	5/25/2017	6/1/2017	10	0.31		98
Dx-mix-stk-10-0-3	1705087-12	5/25/2017	6/1/2017	10	0.27		97
Dx-mix-stk-100-0-1	1705087-13	5/25/2017	5/31/2017	100	4.11		94
Dx-mix-stk-100-0-2	1705087-14	5/25/2017	5/31/2017	100	4.21		92
Dx-mix-stk-100-0-3	1705087-15	5/25/2017	5/31/2017	100	4.4		97

Table E-2: Analytical results of the water samples during the range-finding toxicity tests.

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% of nominal	% recovery
Dx-RF-MARINE-BLNK-1	1706044-01	6/8/2017	6/14/2017	0	0.05	U		109
Dx-RF-Aaff-50-0-1-F	1706044-02	6/8/2017	6/14/2017	50	1.64		0.03	120
Dx-RF-Aaff-5-0-1-F	1706044-03	6/8/2017	6/14/2017	5	0.18		0.04	110
Dx-RF-Aaff-5-0-2-F	1706044-04	6/8/2017	6/14/2017	5	0.17		0.03	121
Dx-RF-Aaff-0.5-0-1-F	1706044-05	6/8/2017	6/14/2017	0.5	0.05	U	0.10	117
Dx-RF-FRESH-BLNK-1	1706044-06	6/8/2017	6/14/2017	0	0.07			117
Dx-RF-Ppro-50-0-1-F	1706044-07	6/8/2017	6/14/2017	50	1.32		0.03	104
Dx-RF-Ppro-5-0-1-F	1706044-08	6/8/2017	6/14/2017	5	0.15		0.03	112
Dx-RF-Ppro-5-0-2-F	1706044-09	6/8/2017	6/14/2017	5	0.09		0.02	104
Dx-RF-Ppro-0.5-0-1-F	1706044-10	6/8/2017	6/14/2017	0.5	0.05	U	0.10	109
Dx-RF-PERRIER-BLNK-1	1706044-11	6/8/2017	6/14/2017	0	0.05	U		109
Dx-RF-Cdub-50-0-1-F	1706044-12	6/8/2017	6/14/2017	50	10		0.20	135
Dx-RF-Cdub-5-0-1-F	1706044-13	6/8/2017	6/15/2017	5	0.39		0.08	87
Dx-RF-Cdub-5-0-2-F	1706044-14	6/8/2017	6/15/2017	5	0.46		0.09	98
Dx-RF-Cdub-0.5-0-1-F	1706044-15	6/8/2017	6/15/2017	0.5	0.15		0.30	81
Dx-RF-Aaff-5-24-1-S	1706044-17	6/9/2017	6/15/2017	5	0.26		0.05	93
Dx-RF-Aaff-0.5-24-1-S	1706044-18	6/9/2017	6/15/2017	0.5	0.19		0.38	77
Dx-RF-Ppro-50-24-1-S	1706044-19	6/9/2017	6/15/2017	50	1.29		0.03	92
Dx-RF-Ppro-5-24-1-S	1706044-20	6/9/2017	6/15/2017	5	0.44		0.09	95
Dx-RF-Ppro-0.5-24-1-S	1706044-21	6/9/2017	6/15/2017	0.5	0.26		0.52	89
Dx-RF-Aaff-5-24-1-F	1706044-22	6/9/2017	6/16/2017	5	0.17		0.03	69
Dx-RF-Aaff-5-24-2-F	1706044-23	6/9/2017	6/16/2017	5	0.29		0.06	86
Dx-RF-Aaff-0.5-24-1-F	1706044-24	6/9/2017	6/16/2017	0.5	0.17		0.34	81
Dx-RF-Ppro-50-24-1-F	1706044-25	6/9/2017	6/16/2017	50	24		0.48	91
Dx-RF-Ppro-5-24-1-F	1706044-26	6/9/2017	6/16/2017	5	2.27		0.45	93
Dx-RF-Ppro-5-24-2-F	1706044-27	6/9/2017	6/16/2017	5	1.84		0.37	82
Dx-RF-Ppro-0.5-24-1-F	1706044-28	6/9/2017	6/16/2017	0.5	0.34		0.68	84
Dx-RF-Cdub-5-24-1-F	1706044-29	6/9/2017	6/16/2017	5	0.56		0.11	76
Dx-RF-Cdub-5-24-2-F	1706044-30	6/9/2017	6/16/2017	5	0.6		0.12	83
Dx-RF-Cdub-0.5-24-1-F	1706044-31	6/9/2017	6/16/2017	0.5	0.25		0.50	88
Dx-RF-Aaff-5-48-1-F	1706044-32	6/10/2017	6/16/2017	5	0.36		0.07	74
Dx-RF-Aaff-0.5-48-1-F	1706044-33	6/10/2017	6/16/2017	0.5	0.2		0.40	80
Dx-RF-Ppro-50-48-1-F	1706044-34	6/10/2017	6/16/2017	50	0.73		0.01	73
Dx-RF-Ppro-5-48-1-F	1706044-35	6/10/2017	6/16/2017	5	0.23		0.05	73
Dx-RF-Ppro-0.5-48-1-F	1706044-36	6/10/2017	6/16/2017	0.5	0.12		0.24	80
Dx-RF-Cdub-5-48-1-F	1706044-37	6/10/2017	6/16/2017	5	0.95		0.19	82
Dx-RF-Cdub-0.5-48-1-F	1706044-38	6/10/2017	6/16/2017	0.5	0.18		0.36	86
Dx-RF-Aaff-5-72-1-F	1706044-39	6/12/2017	6/16/2017	5	0.35		0.07	80

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% of nominal	% recovery
Dx-RF-Aaff-5-72-2-F	1706044-40	6/12/2017	6/16/2017	5	0.39		0.08	86
Dx-RF-Aaff-0.5-72-1-F	1706044-41	6/12/2017	6/16/2017	0.5	0.05	U	0.10	80
Dx-RF-Ppro-50-72-1-F	1706044-42	6/12/2017	6/17/2017	50	4.65		0.09	97
Dx-RF-Ppro-5-72-1-F	1706044-43	6/12/2017	6/17/2017	5	0.24		0.05	85
Dx-RF-Ppro-5-72-2-F	1706044-44	6/12/2017	6/17/2017	5	0.23		0.05	82
Dx-RF-Ppro-0.5-72-1-F	1706044-45	6/12/2017	6/17/2017	0.5	0.05	U	0.10	94
Dx-RF-Cdub-5-72-1-F	1706044-46	6/12/2017	6/17/2017	5	0.59		0.12	88
Dx-RF-Cdub-5-72-2-F	1706044-47	6/12/2017	6/17/2017	5	0.39		0.08	79
Dx-RF-Cdub-0.5-72-1-F	1706044-48	6/12/2017	6/17/2017	0.5	0.14		0.28	82
Dx-RF-Aaff-5-96-1-F	1706045-01	6/20/2017	6/21/2017	5	0.19		0.04	98
Dx-RF-Aaff-5-96-2-F	1706045-02	6/20/2017	6/21/2017	5	0.07		0.01	90
Dx-RF-Aaff-0.5-96-1-F	1706045-03	6/20/2017	6/21/2017	0.5	0.05	U	0.10	106
Dx-RF-Ppro-50-96-1-F	1706045-04	6/20/2017	6/21/2017	50	3.52		0.07	122
Dx-RF-Ppro-5-96-1-F	1706045-05	6/20/2017	6/21/2017	5	0.13		0.03	82
Dx-RF-Ppro-5-96-2-F	1706045-06	6/20/2017	6/21/2017	5	0.32		0.06	109
Dx-RF-Ppro-0.5-96-1-F	1706045-07	6/20/2017	6/21/2017	0.5	0.05	U	0.10	94
Dx-RF-Cdub-0.5-96-1-F	1706045-08	6/20/2017	6/21/2017	0.5	0.3		0.60	108
Dx-RF-Aaff-5-96-1-S	1706045-09	6/20/2017	6/21/2017	5	0.08	U	0.02	90
Dx-RF-Aaff-0.5-96-1-S	1706045-10	6/20/2017	6/22/2017	0.5	0.07	U	0.14	94
Dx-RF-Ppro-50-96-1-S	1706045-11	6/20/2017	6/22/2017	50	1.46		0.03	104
Dx-RF-Ppro-5-96-1-S	1706045-12	6/20/2017	6/22/2017	5	0.19		0.04	97
Dx-RF-Ppro-0.5-96-1-S	1706045-13	6/20/2017	6/22/2017	0.5	0.08	U	0.16	96
Dx-RF-Aaff-5-120-1-F	1706045-14	6/20/2017	6/22/2017	5	0.18		0.04	93
Dx-RF-Aaff-0.5-120-1-F	1706045-15	6/20/2017	6/22/2017	0.5	0.05	U	0.10	89
Dx-RF-Ppro-5-120-1-F	1706045-16	6/20/2017	6/22/2017	5	0.28		0.06	98
Dx-RF-Ppro-0.5-120-1-F	1706045-17	6/20/2017	6/22/2017	0.5	0.05	U	0.10	96
Dx-RF-Cdub-0.5-120-1-F	1706045-18	6/20/2017	6/22/2017	0.5	0.21		0.42	95
Dx-RF-Aaff-5-144-1-F	1706045-19	6/20/2017	6/22/2017	5	0.23		0.05	100
Dx-RF-Aaff-5-144-2-F	1706045-20	6/20/2017	6/22/2017	5	0.23		0.05	91
Dx-RF-Aaff-0.5-144-1-F	1706045-21	6/21/2017	6/22/2017	0.5	0.08	J	0.16	43
Dx-RF-Ppro-5-144-1-F	1706045-22	6/21/2017	6/22/2017	5	0.15		0.03	93
Dx-RF-Ppro-5-144-2-F	1706045-23	6/21/2017	6/22/2017	5	0.14		0.03	90
Dx-RF-Ppro-0.5-144-1-F	1706045-24	6/21/2017	6/22/2017	0.5	0.05	U	0.10	89
Dx-RF-Aaff-5-168-1-S	1706045-25	6/21/2017	6/22/2017	5	0.06	U	0.01	93
Dx-RF-Aaff-0.5-168-1-S	1706045-26	6/21/2017	6/22/2017	0.5	0.06	U	0.12	90
Dx-RF-Ppro-5-168-1-S	1706045-27	6/21/2017	6/22/2017	5	0.2		0.04	89
Dx-RF-Ppro-0.5-168-1-S	1706045-28	6/21/2017	6/22/2017	0.5	0.06	U	0.12	91

U = analyte not detected
J = analyte detected; result is an estimate

Table E-3: Analytical results of the toxicity test water samples.

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% recovery
Dx-Ctox-MARINE-BLNK-1	1707045-01	7/6/2017	7/14/2017		0.05	UJ	104
Dx-Ctox-MARINE-BLNK-2	1707045-02	7/6/2017	7/14/2017		0.05	UJ	100
Dx-Ctox-MARINE-BLNK-3	1707045-03	7/6/2017	7/14/2017		0.06	J	111
Dx-Ctox-Aaff-16-0-1-F	1707045-04	7/6/2017	7/14/2017	16	0.62	J	83
Dx-Ctox-Aaff-4-0-1-F	1707045-05	7/6/2017	7/14/2017	4	0.07	J	86
Dx-Ctox-Aaff-4-0-2-F	1707045-06	7/6/2017	7/14/2017	4	0.29	J	118
Dx-Ctox-Aaff-1-0-1-F	1707045-07	7/6/2017	7/14/2017	1	0.07	J	127
Dx-Ctox-Aaff-0.5-0-1-F	1707045-08	7/6/2017	7/14/2017	0.5	0.05	UJ	120
Dx-Ctox-FRESH-BLNK-1	1707045-09	7/6/2017	7/14/2017		0.05	UJ	102
Dx-Ctox-FRESH-BLNK-2	1707045-10	7/6/2017	7/14/2017		0.05	UJ	122
Dx-Ctox-FRESH-BLNK-3	1707045-11	7/6/2017	7/14/2017		0.05	UJ	119
Dx-Ctox-Ppro-30-0-1-F	1707045-12	7/6/2017	7/14/2017	30	3.03	J	132
Dx-Ctox-Ppro-7.5-0-1-F	1707045-13	7/6/2017	7/15/2017	7.5	0.29	J	102
Dx-Ctox-Ppro-7.5-0-2-F	1707045-14	7/6/2017	7/15/2017	7.5	0.49	J	140
Dx-Ctox-Ppro-1.9-0-1-F	1707045-15	7/6/2017	7/15/2017	1.9	0.13	J	136
Dx-Ctox-Ppro-0.9-0-1-F	1707045-16	7/6/2017	7/15/2017	0.9	0.08	J	130
Dx-Ctox-PERRIER-BLNK-1	1707045-17	7/6/2017	7/15/2017		0.05	UJ	131
Dx-Ctox-PERRIER-BLNK-2	1707045-18	7/6/2017	7/15/2017		0.05	UJ	132
Dx-Ctox-PERRIER-BLNK-3	1707045-19	7/6/2017	7/15/2017		0.06	UJ	137
Dx-Ctox-Cdub-6-0-1-F	1707045-20	7/6/2017	7/15/2017	6	1.19	J	139
Dx-Ctox-Cdub-3-0-1-F	1707045-21	7/6/2017	7/17/2017	3	0.55	J	123
Dx-Ctox-Cdub-3-0-2-F	1707045-22	7/6/2017	7/17/2017	3	0.24	J	88
Dx-Ctox-Cdub-1.5-0-1-F	1707045-23	7/6/2017	7/17/2017	1.5	0.29	J	113
Dx-Ctox-Cdub-0.75-0-1-F	1707045-24	7/6/2017	7/17/2017	0.75	0.11	J	120
Dx-Ctox-Cdub-0.38-0-1-F	1707045-25	7/6/2017	7/17/2017	0.38	0.08	J	82
Dx-Ctox-Cdub-0.19-0-1-F	1707045-26	7/6/2017	7/17/2017	0.19	0.05	UJ	122
Dx-Ctox-Aaff-16-24-1-F	1707045-27	7/7/2017	7/17/2017	16	2.02	J	130
Dx-Ctox-Aaff-4-24-1-F	1707045-28	7/7/2017	7/17/2017	4	0.38	J	121
Dx-Ctox-Aaff-4-24-2-F	1707045-29	7/7/2017	7/17/2017	4	0.4	J	121
Dx-Ctox-Aaff-1-24-1-F	1707045-30	7/7/2017	7/17/2017	1	0.13	J	118
Dx-Ctox-Aaff-0.5-24-1-F	1707045-31	7/7/2017	7/17/2017	0.5	0.07	J	117
Dx-Ctox-Ppro-30-24-1-F	1707045-32	7/7/2017	7/17/2017	30	3.26	J	133
Dx-Ctox-Ppro-7.5-24-1-F	1707045-33	7/7/2017	7/17/2017	7.5	0.48	J	124
Dx-Ctox-Ppro-7.5-24-2-F	1707045-34	7/7/2017	7/17/2017	7.5	0.47	J	125
Dx-Ctox-Ppro-1.9-24-1-F	1707045-35	7/7/2017	7/17/2017	1.9	0.12	J	127
Dx-Ctox-Ppro-0.9-24-1-F	1707045-36	7/7/2017	7/17/2017	0.9	0.11	J	124
Dx-Ctox-Cdub-1.5-24-1-F	1707045-37	7/7/2017	7/17/2017	1.5	1.22	J	130
Dx-Ctox-Cdub-0.75-24-1-F	1707045-38	7/7/2017	7/17/2017	0.75	0.52	J	125

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% recovery
Dx-Ctox-Cdub-0.38-24-1-F	1707045-39	7/7/2017	7/17/2017	0.38	0.24	J	123
Dx-Ctox-Aaff-16-24-1-S	1707045-40	7/7/2017	7/17/2017	16	0.58	J	132
Dx-Ctox-Aaff-4-24-1-S	1707045-41	7/7/2017	7/18/2017	4	0.14		99
Dx-Ctox-Aaff-1-24-1-S	1707045-42	7/7/2017	7/18/2017	1	0.05	U	104
Dx-Ctox-Aaff-0.5-24-1-S	1707045-43	7/7/2017	7/18/2017	0.5	0.05	U	103
Dx-Ctox-Ppro-30-24-1-S	1707045-44	7/7/2017	7/18/2017	30	0.74		102
Dx-Ctox-Ppro-7.5-24-1-S	1707045-45	7/7/2017	7/18/2017	7.5	0.18		98
Dx-Ctox-Ppro-1.9-24-1-S	1707045-46	7/7/2017	7/18/2017	1.9	0.06	U	96
Dx-Ctox-Ppro-0.9-24-1-S	1707045-47	7/7/2017	7/18/2017	0.9	0.06	U	104
Dx-Ctox-Cdub-0.38-72-1-F	1707045-48	7/9/2017	7/18/2017	0.38	0.14		98
Dx-Ctox-Aaff-16-48-1-F	1707045-49	7/8/2017	7/18/2017	16	3.31		107
Dx-Ctox-Aaff-4-48-1-F	1707045-50	7/8/2017	7/18/2017	4	0.31		102
Dx-Ctox-Aaff-1-48-1-F	1707045-51	7/8/2017	7/18/2017	1	0.1		109
Dx-Ctox-Aaff-0.5-48-1-F	1707045-52	7/8/2017	7/18/2017	0.5	0.06		111
Dx-Ctox-Ppro-30-48-1-F	1707045-53	7/8/2017	7/18/2017	30	1.75		110
Dx-Ctox-Ppro-7.5-48-1-F	1707045-54	7/8/2017	7/18/2017	7.5	0.37		102
Dx-Ctox-Ppro-1.9-48-1-F	1707045-55	7/8/2017	7/18/2017	1.9	0.13		106
Dx-Ctox-Ppro-0.9-48-1-F	1707045-56	7/8/2017	7/18/2017	0.9	0.14		98
Dx-Ctox-Cdub-1.5-48-1-F	1707045-57	7/8/2017	7/18/2017	1.5	0.36		104
Dx-Ctox-Cdub-0.75-48-1-F	1707045-58	7/8/2017	7/19/2017	0.75	0.23		104
Dx-Ctox-Cdub-0.38-48-1-F	1707045-59	7/8/2017	7/19/2017	0.38	0.18		111
Dx-Ctox-Cdub-0.19-48-1-F	1707045-60	7/8/2017	7/19/2017	0.19	0.08		101
Dx-Ctox-Aaff-16-72-1-F	1707045-61	7/9/2017	7/19/2017	16	0.96	J	74
Dx-Ctox-Aaff-4-72-1-F	1707045-62	7/9/2017	7/19/2017	4	0.32	J	77
Dx-Ctox-Aaff-4-72-2-F	1707045-63	7/9/2017	7/19/2017	4	0.31	J	73
Dx-Ctox-Aaff-1-72-1-F	1707045-64	7/9/2017	7/19/2017	1	0.11	J	81
Dx-Ctox-Aaff-0.5-72-1-F	1707045-65	7/9/2017	7/19/2017	0.5	0.06	J	81
Dx-Ctox-Ppro-30-72-1-F	1707045-66	7/9/2017	7/19/2017	30	1.79	J	83
Dx-Ctox-Ppro-7.5-72-1-F	1707045-67	7/9/2017	7/19/2017	7.5	0.42	J	78
Dx-Ctox-Ppro-7.5-72-2-F	1707045-68	7/9/2017	7/19/2017	7.5	0.43	J	76
Dx-Ctox-Ppro-1.9-72-1-F	1707045-69	7/9/2017	7/19/2017	1.9	0.18	J	78
Dx-Ctox-Ppro-0.9-72-1-F	1707045-70	7/9/2017	7/19/2017	0.9	0.1	J	87
Dx-Ctox-Cdub-1.5-72-1-F	1707045-71	7/9/2017	7/19/2017	1.5	0.53	J	77
Dx-Ctox-Cdub-0.75-72-1-F	1707045-72	7/9/2017	7/19/2017	0.75	0.15	J	71
Dx-Ctox-Aaff-4-96-1-F	1707056-01	7/10/2017	7/21/2017	4	0.17	J	68
Dx-Ctox-Aaff-4-96-2-F	1707056-02	7/10/2017	7/21/2017	4	0.14	J	79
Dx-Ctox-Aaff-1-96-1-F	1707056-03	7/10/2017	7/21/2017	1	0.11	J	82
Dx-Ctox-Aaff-0.5-96-1-F	1707056-04	7/10/2017	7/21/2017	0.5	0.13	J	78
Dx-Ctox-Ppro-30-96-1-F	1707056-05	7/10/2017	7/21/2017	30	2.86	J	89

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% recovery
Dx-Ctox-Ppro-7.5-96-1-F	1707056-06	7/10/2017	7/21/2017	7.5	0.57	J	82
Dx-Ctox-Ppro-7.5-96-2-F	1707056-07	7/10/2017	7/21/2017	7.5	0.54	J	85
Dx-Ctox-Ppro-1.9-96-1-F	1707056-08	7/10/2017	7/21/2017	1.9	0.17	J	94
Dx-Ctox-Ppro-0.9-96-1-F	1707056-09	7/10/2017	7/21/2017	0.9	0.1	J	86
Dx-Ctox-Cdub-0.75-96-1-F	1707056-10	7/10/2017	7/21/2017	0.75	0.4	J	84
Dx-Ctox-Cdub-0.38-96-1-F	1707056-11	7/10/2017	7/21/2017	0.38	0.21	J	83
Dx-Ctox-Cdub-0.19-96-1-F	1707056-12	7/10/2017	7/21/2017	0.19	0.12	J	90
Dx-Ctox-Aaff-4-96-1-S	1707056-13	7/10/2017	7/21/2017	4	0.15	J	81
Dx-Ctox-Aaff-1-96-1-S	1707056-14	7/10/2017	7/22/2017	1	0.16	J	89
Dx-Ctox-Aaff-0.5-96-1-S	1707056-15	7/10/2017	7/22/2017	0.5	0.12	J	80
Dx-Ctox-Ppro-30-96-1-S	1707056-16	7/10/2017	7/22/2017	30	0.59	J	85
Dx-Ctox-Ppro-7.5-96-1-S	1707056-17	7/10/2017	7/22/2017	7.5	0.19	J	84
Dx-Ctox-Ppro-1.9-96-1-S	1707056-18	7/10/2017	7/22/2017	1.9	0.18	J	85
Dx-Ctox-Ppro-0.9-96-1-S	1707056-19	7/10/2017	7/22/2017	0.9	0.15	J	84
Dx-Ctox-Aaff-4-120-1-F	1707056-20	7/11/2017	7/22/2017	4	0.2	J	80
Dx-Ctox-Aaff-1-120-1-F	1707056-21	7/11/2017	7/25/2017	1	0.18		98
Dx-Ctox-Aaff-0.5-120-1-F	1707056-22	7/11/2017	7/25/2017	0.5	0.14		95
Dx-Ctox-Ppro-30-120-1-F	1707056-23	7/11/2017	7/25/2017	30	2.72		104
Dx-Ctox-Ppro-7.5-120-1-F	1707056-24	7/11/2017	7/25/2017	7.5	0.62		100
Dx-Ctox-Ppro-1.9-120-1-F	1707056-25	7/11/2017	7/25/2017	1.9	0.14		101
Dx-Ctox-Ppro-0.9-120-1-F	1707056-26	7/11/2017	7/25/2017	0.9	0.1		103
Dx-Ctox-Cdub-0.75-120-1-F	1707056-27	7/11/2017	7/25/2017	0.75	0.19		100
Dx-Ctox-Cdub-0.38-120-1-F	1707056-28	7/11/2017	7/25/2017	0.38	0.1		102
Dx-Ctox-Aaff-4-144-1-F	1707056-29	7/12/2017	7/25/2017	4	0.29		98
Dx-Ctox-Aaff-4-144-2-F	1707056-30	7/12/2017	7/25/2017	4	0.32		99
Dx-Ctox-Aaff-1-144-1-F	1707056-31	7/12/2017	7/26/2017	1	0.18		105
Dx-Ctox-Aaff-0.5-144-1-F	1707056-32	7/12/2017	7/26/2017	0.5	0.13		104
Dx-Ctox-Ppro-30-144-1-F	1707056-33	7/12/2017	7/26/2017	30	3.68		120
Dx-Ctox-Ppro-7.5-144-1-F	1707056-34	7/12/2017	7/26/2017	7.5	1.05		108
Dx-Ctox-Ppro-7.5-144-2-F	1707056-35	7/12/2017	7/26/2017	7.5	1.05		108
Dx-Ctox-Ppro-1.9-144-1-F	1707056-36	7/12/2017	7/26/2017	1.9	0.21		106
Dx-Ctox-Ppro-0.9-144-1-F	1707056-37	7/12/2017	7/26/2017	0.9	0.12		98
Dx-Ctox-Cdub-0.75-144-1-F	1707056-38	7/12/2017	7/26/2017	0.75	0.42		97
Dx-Ctox-Cdub-0.38-144-1-F	1707056-39	7/12/2017	7/26/2017	0.38	0.21		102
Dx-Ctox-Cdub-0.19-144-1-F	1707056-40	7/12/2017	7/26/2017	0.19	0.14		106
Dx-Ctox-Aaff-4-168-1-S	1707056-41	7/13/2017	8/1/2017	4	0.2		85
Dx-Ctox-Aaff-1-168-1-S	1707056-42	7/13/2017	8/1/2017	1	0.16		75
Dx-Ctox-Aaff-0.5-168-1-S	1707056-43	7/13/2017	8/1/2017	0.5	0.17		86
Dx-Ctox-Ppro-30-168-1-S	1707056-44	7/13/2017	8/1/2017	30	1.37		90

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% recovery
Dx-Ctox-Ppro-7.5-168-1-S	1707056-45	7/13/2017	8/1/2017	7.5	0.42		87
Dx-Ctox-Ppro-1.9-168-1-S	1707056-46	7/13/2017	8/1/2017	1.9	0.28		90
Dx-Ctox-Ppro-0.9-168-1-S	1707056-47	7/13/2017	8/1/2017	0.9	0.29		96

U = analyte not detected
J = analyte detected; result is an estimate

Table E-4: Results of the sampling quality control samples.

Sample ID	MEL ID	sample date	analysis date	TPH-Dx (mg/L)		RPD
RF-Ppro-10-24-1-F	1705051-18	4/28/2017	5/3/2017	8.96		0.12
RF-Ppro-10-24-2-F	1705051-19	4/28/2017	5/3/2017	7.96		
RF-Cdub-10-24-1-S	1705051-25	4/28/2017	5/8/2017	4.35		0.08
RF-Cdub-10-24-2-S	1705051-26	4/28/2017	5/8/2017	4.03		
RF-Ppro-10-72-1-F	1705051-33	4/30/2017	5/9/2017	9.45		0.02
RF-Ppro-10-72-2-F	1705051-34	4/30/2017	5/9/2017	9.3		
Dx-Ctox-Aaff-4-0-1-F	1707045-05	7/6/2017	7/14/2017	0.07	J	**1.22**
Dx-Ctox-Aaff-4-0-2-F	1707045-06	7/6/2017	7/14/2017	0.29	J	
Dx-Ctox-Ppro-7.5-0-1-F	1707045-13	7/6/2017	7/15/2017	0.29	J	**0.51**
Dx-Ctox-Ppro-7.5-0-2-F	1707045-14	7/6/2017	7/15/2017	0.49	J	
Dx-Ctox-Cdub-3-0-1-F	1707045-21	7/6/2017	7/17/2017	0.55	J	**0.78**
Dx-Ctox-Cdub-3-0-2-F	1707045-22	7/6/2017	7/17/2017	0.24	J	
Dx-Ctox-Aaff-4-24-1-F	1707045-28	7/7/2017	7/17/2017	0.38	J	0.05
Dx-Ctox-Aaff-4-24-2-F	1707045-29	7/7/2017	7/17/2017	0.4	J	
Dx-Ctox-Ppro-7.5-24-1-F	1707045-33	7/7/2017	7/17/2017	0.48	J	0.02
Dx-Ctox-Ppro-7.5-24-2-F	1707045-34	7/7/2017	7/17/2017	0.47	J	
Dx-Ctox-Aaff-4-72-1-F	1707045-62	7/9/2017	7/19/2017	0.32	J	0.03
Dx-Ctox-Aaff-4-72-2-F	1707045-63	7/9/2017	7/19/2017	0.31	J	
Dx-Ctox-Ppro-7.5-72-1-F	1707045-67	7/9/2017	7/19/2017	0.42	J	0.02
Dx-Ctox-Ppro-7.5-72-2-F	1707045-68	7/9/2017	7/19/2017	0.43	J	
Dx-Ctox-Aaff-4-96-1-F	1707056-01	7/10/2017	7/21/2017	0.17	J	0.19
Dx-Ctox-Aaff-4-96-2-F	1707056-02	7/10/2017	7/21/2017	0.14	J	
Dx-Ctox-Ppro-7.5-96-1-F	1707056-06	7/10/2017	7/21/2017	0.57	J	0.05
Dx-Ctox-Ppro-7.5-96-2-F	1707056-07	7/10/2017	7/21/2017	0.54	J	
Dx-Ctox-Aaff-4-144-1-F	1707056-29	7/12/2017	7/25/2017	0.29		0.10
Dx-Ctox-Aaff-4-144-2-F	1707056-30	7/12/2017	7/25/2017	0.32		
Dx-Ctox-Ppro-7.5-144-1-F	1707056-34	7/12/2017	7/26/2017	1.05		0.00
Dx-Ctox-Ppro-7.5-144-2-F	1707056-35	7/12/2017	7/26/2017	1.05		

Bold values are in excess of 20% RPD
J = analyte detected; result is an estimate

Table E-5: Results of the laboratory quality control samples.

Sample ID	MEL ID	MEL work order / batch	analysis date	TPH-Dx (mg/L) and LCS Recovery		surrogate recovery (%)
Mixing						
Method Blnk	B17E222-BLK1	1705087	5/31/2017	0.15	U	97
LCS	B17E222-BS1	1705087	5/31/2017	85	%	102
LCS Dup	B17E222-BSD1	1705087	5/31/2017	82	%	101
Range-Finding						
Method Blnk	B17F103-BLK1	1706044	6/15/2017	0.15	U	67
LCS	B17F103-BS1	1706044	6/15/2017	79	%	94
LCS Dup	B17F103-BSD1	1706044	6/15/2017	81	%	103
Method Blnk	B17F106-BLK1	1706044	6/16/2017	0.15	U	91
LCS	B17F106-BS1	1706044	6/16/2017	94	%	95
LCS Dup	B17F106-BSD1	1706044	6/16/2017	77	%	81
Method Blnk	B17F123-BLK1	1706044	6/16/2017	0.15	U	88
LCS	B17F123-BS1	1706044	6/17/2017	80	%	86
LCS Dup	B17F123-BSD1	1706044	6/17/2017	83	%	88
Method Blnk	B17F147-BLK1	1706045	6/22/2017	150	U	100
LCS	B17F147-BS1	1706045	6/22/2017	85	%	101
LCS Dup	B17F147-BSD1	1706045	6/22/2017	85	%	103
Method Blnk	B17F148-BLK1	1706045	6/21/2017	0.15	U	103
LCS	B17F148-BS1	1706045	6/21/2017	83	%	115
LCS Dup	B17F148-BSD1	1706045	6/21/2017	76	%	117
Final Chronic Test						
Method Blnk	B17G054-BLK1	1707045	7/14/2017	0.15	U	116
LCS	B17G054-BS1	1707045	7/14/2017	67	%	117
LCS Dup	B17G054-BSD1	1707045	7/14/2017	78	%	141
Method Blnk	B17G060-BLK1	1707045	7/17/2017	0.15	U	117
LCS	B17G060-BS1	1707045	7/17/2017	57	%	108
LCS Dup	B17G060-BSD1	1707045	7/17/2017	80	%	115
Method Blnk	B17G073-BLK1	1707045	7/18/2017	0.15	U	94
LCS	B17G073-BS1	1707045	7/18/2017	78	%	98
LCS Dup	B17G073-BSD1	1707045	7/18/2017	84	%	113
Method Blnk	B17G085-BLK1	1707045	7/19/2017	0.15	U	88
LCS	B17G085-BS1	1707045	7/19/2017	83	%	85
LCS Dup	B17G085-BSD1	1707045	7/19/2017	48	%	57
Method Blnk	B17G119-BLK1	1707056	7/21/2017	0.15	U	83
LCS	B17G119-BS1	1707056	7/21/2017	77	%	86
LCS Dup	B17G119-BSD1	1707056	7/21/2017	41	%	55
Method Blnk	B17G125-BLK1	1707056	7/25/2017	0.15	U	93

Sample ID	MEL ID	MEL work order / batch	analysis date	TPH-Dx (mg/L) and LCS Recovery		surrogate recovery (%)
LCS	B17G125-BS1	1707056	7/25/2017	NA	%	99
LCS Dup	B17G125-BSD1	1707056	7/25/2017	NA	%	98
Method Blnk	B17G157-BLK1	1707056	8/1/2017	0.15	U	83
LCS	B17G157-BS1	1707056	8/1/2017	NA	%	86
LCS Dup	B17G157-BSD1	1707056	8/1/2017	NA	%	83

U = analyte not detected

Table E-6: Results from the re-analysis of samples with silica gel cleanup.

Sample ID	MEL ID	sample date	analysis date	nominal Dx	TPH-Dx (mg/L)		% recovery	re-analysis date	TPH-Dx Si (mg/L)		% recovery
Dx-RF-Aaff-5-24-1-S	1706044-17	6/9/2017	6/15/2017	5	0.26		93	7/10/2017	0.26		114
Dx-RF-Aaff-0.5-24-1-S	1706044-18	6/9/2017	6/15/2017	0.5	0.19		77	7/10/2017	0.17		92
Dx-RF-Ppro-50-24-1-S	1706044-19	6/9/2017	6/15/2017	50	1.29		92	7/10/2017	1.19		102
Dx-RF-Ppro-5-24-1-S	1706044-20	6/9/2017	6/15/2017	5	0.44		95	7/10/2017	0.36		118
Dx-RF-Ppro-0.5-24-1-S	1706044-21	6/9/2017	6/15/2017	0.5	0.26		89	7/10/2017	0.19		102
Dx-RF-Ppro-50-96-1-S	1706045-11	6/20/2017	6/22/2017	50	1.46		104	7/10/2017	1.54		121
Dx-RF-Ppro-5-96-1-S	1706045-12	6/20/2017	6/22/2017	5	0.19		97	7/10/2017	0.13		114
Dx-RF-Ppro-5-168-1-S	1706045-27	6/21/2017	6/22/2017	5	0.2		89	7/10/2017	0.13		105
Dx-Ctox-Aaff-16-24-1-S	1707045-40	7/7/2017	7/17/2017	16	0.58	J	132	8/18/2017	0.61	J	114
Dx-Ctox-Aaff-4-24-1-S	1707045-41	7/7/2017	7/18/2017	4	0.14		99	8/18/2017	0.2		107
Dx-Ctox-Ppro-30-24-1-S	1707045-44	7/7/2017	7/18/2017	30	0.74		102	8/18/2017	0.74		104
Dx-Ctox-Ppro-7.5-24-1-S	1707045-45	7/7/2017	7/18/2017	7.5	0.18		98	8/18/2017	0.25		106
Dx-Ctox-Aaff-4-96-1-S	1707056-13	7/10/2017	7/21/2017	4	0.15	J	81	8/18/2017	0.13	J	83
Dx-Ctox-Aaff-1-96-1-S	1707056-14	7/10/2017	7/22/2017	1	0.16	J	89	8/18/2017	0.14	J	98
Dx-Ctox-Aaff-0.5-96-1-S	1707056-15	7/10/2017	7/22/2017	0.5	0.12	J	80	8/18/2017	0.12	J	85
Dx-Ctox-Ppro-30-96-1-S	1707056-16	7/10/2017	7/22/2017	30	0.59	J	85	8/18/2017	0.69	J	111
Dx-Ctox-Ppro-7.5-96-1-S	1707056-17	7/10/2017	7/22/2017	7.5	0.19	J	84	8/18/2017	0.23	J	89
Dx-Ctox-Ppro-1.9-96-1-S	1707056-18	7/10/2017	7/22/2017	1.9	0.18	J	85	8/18/2017	0.14	J	80
Dx-Ctox-Ppro-0.9-96-1-S	1707056-19	7/10/2017	7/22/2017	0.9	0.15	J	84	8/18/2017	0.13	J	80
Dx-Ctox-Aaff-4-168-1-S	1707056-41	7/13/2017	8/1/2017	4	0.2		85	8/18/2017	0.19		87
Dx-Ctox-Aaff-1-168-1-S	1707056-42	7/13/2017	8/1/2017	1	0.16		75	8/18/2017	0.17		78
Dx-Ctox-Aaff-0.5-168-1-S	1707056-43	7/13/2017	8/1/2017	0.5	0.17		86	8/19/2017	0.19		95
Dx-Ctox-Ppro-30-168-1-S	1707056-44	7/13/2017	8/1/2017	30	1.37		90	8/19/2017	1.39		95
Dx-Ctox-Ppro-7.5-168-1-S	1707056-45	7/13/2017	8/1/2017	7.5	0.42		87	8/19/2017	0.41		86
Dx-Ctox-Ppro-1.9-168-1-S	1707056-46	7/13/2017	8/1/2017	1.9	0.28		90	8/19/2017	0.27		94
Dx-Ctox-Ppro-0.9-168-1-S	1707056-47	7/13/2017	8/1/2017	0.9	0.29		96	8/19/2017	0.24		97

J = analyte detected; result is an estimate

Appendix F. Glossary, Acronyms, and Abbreviations

Glossary

Clean Water Act: A federal act passed in 1972 that contains provisions to restore and maintain the quality of the nation's waters. Section 303(d) of the Clean Water Act establishes the TMDL program.

Dissolved oxygen (DO): A measure of the amount of oxygen dissolved in water.

Inhibitory concentration (IC): The toxicant concentration that would cause a given percent reduction in a nonquantal biological measurement for the test population. For example, the IC25 is the concentration of toxicant that would cause a 25% reduction in mean young per female or in growth for the test population.

Lowest-observed effect concentration (LOEC): The lowest concentration of toxicant to which organisms are exposed in a life-cycle or partial life-cycle (short-term) test, which causes adverse effects on the test organisms (i.e., where the values for the observed responses are statistically significantly different from the controls).

Lethal concentration (LC): The toxicant concentration that would cause death in a given percent of the test population. Identical to EC when the observable adverse effect is death. For example, the LC50 is the concentration of toxicant that would cause death in 50% of the test population.

Method Detection Limit (MDL): The minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero.

No-observed effects concentration (NOEC): The highest concentration of toxicant to which organisms are exposed in a full life-cycle or partial life-cycle (short-term) test, that causes no observable adverse effects on the test organisms (i.e., the highest concentration of toxicant in which the values for the observed responses are not statistically significantly different from the controls). This value is used, along with other factors, to determine toxicity limits in permits..

Practical Quantitation Limit (PQL): The analyte concentration selected as the lowest non-zero standard in the instrument calibration curve, adjusted for sample specific conditions (e.g.: sample size, percent solids, dilutions, cleanup procedures, etc.). Results below the PQL are considered less accurate and are qualified as estimates.

Water accommodated fraction (WAF): A laboratory-prepared media from the low-energy mixing of a low solubility liquid (e.g. diesel fuel) into water. It is essentially the dissolved portion of the test material which is free of particles.

Whole effluent toxicity (WET) testing: Refers to the aggregate toxicity of pollutants contained in wastewater effluent. It represents the total exposure of aquatic life to pollutants in a controlled lab environment. It is conducted by a qualified lab using EPA methods on test organisms.

Acronyms and Abbreviations

Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
IC25	*see above*
LC50	*see above*
LOEC	Lowest-observed effect concentration
MDL	Method detection limit
MEL	Manchester Environmental Laboratory
NOEC	No-observed effects concentration
NWTPH-Gx	Northwest Total Petroleum Hydrocarbons – Gasoline fraction
NWTPH-Dx	Northwest Total Petroleum Hydrocarbons – Diesel fraction
PQL	Practical quantitation limit
RPD	Relative percent difference
RSD	Relative standard deviation
SRM	Standard reference materials
WAC	Washington Administrative Code

Units of Measurement

°C	degrees centigrade
dw	dry weight
ft	feet
ft-c	foot-candle (measurement of illumination)
g	gram, a unit of mass
mg	milligram
mg/L	milligrams per liter (parts per million)
$\mu E/m^2/s$	microeinsteins per meter squared per second (measurement of illumination)
µg/L	micrograms per liter (parts per billion)