DEPARTMENT OF ECOLOGY
State of Washington

Concentrations of Gasoline and Diesel Range Organics Predicted to be Protective of Aquatic Receptors in Surface Waters

Implementation Memorandum No. 23

Date: August 25, 2021

To: Interested Persons

From: Jeff Johnston, Manager
Information & Policy Section
Toxics Cleanup Program

Contacts: Arthur Buchan, Toxicologist, abuc461@ecy.wa.gov 360-407-7146
Policy & Technical Support Unit, Headquarters, Toxics Cleanup Program

Attachments: A – Response to comments on the January 2019 review draft of this memo

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Acronyms and Abbreviations

Acronym or Abbreviation	Description
BTEX	benzene, toluene, ethylbenzene, and total xylenes
DRO	diesel range organics
EPA	Environmental Protection Agency
µg/L	micrograms per liter
mg/L	milligrams per liter
mL	milliliter
MTCA	Model Toxics Control Act
NOEC	no-observed adverse effect concentration
NWTPH-Dx	Northwest Total Petroleum Hydrocarbons – Diesel Range Organics
NWTPH-Gx	Northwest Total Petroleum Hydrocarbons – Gasoline Range Organics
RCW	Revised Code of Washington
TPH	total petroleum hydrocarbons
WAC	Washington Administrative Code
WAF	water accommodated fraction
WET	whole effluent toxicity

1.0 Purpose and applicability

This memorandum provides gasoline and diesel range organic concentrations that are predicted to be protective for environmental effects in marine and fresh surface waters at any Model Toxics Control Act (MTCA) cleanup site.

The MTCA regulation (also known as the MTCA Cleanup Rule), Chapter 173-340 WAC[1] contains surface water cleanup standards. When you establish Method B or Method C surface water cleanup levels for gasoline or diesel that are protective for environmental effects under WAC 173-340-730(3)(b)(ii) or (4)(b)(ii), you may either:

1. Use the concentrations specified in this document; or
2. Establish concentrations using the protocols described in Chapter 173-205 WAC[2], Whole Effluent Toxicity Testing and Limits.

As an alternative to the values presented in this implementation memorandum, you may use the protocols in Chapter 173-205 WAC, Whole Effluent Toxicity Testing and Limits to establish a site specific protective concentrations.

2.0 Background

Predicted protective concentrations provided in this memorandum were derived from studies published by the Washington State Department of Ecology's Environmental Assessment Program:

1. The 2018 toxicity study: Environmental effects-based concentrations for total petroleum hydrocarbons (TPH): Toxicity in marine water and freshwater[3] (Ecology 2018); and
2. The 2020 toxicity study: Environmental effects-based concentrations for weathered diesel range organics: Toxicity in marine water and freshwater[4] (Ecology 2020).

The purpose of the 2018 toxicity study was to set a baseline for protective concentrations for a relatively fresh, or what could be considered an "unweathered" release of gasoline or diesel range organics. The result was the derivation of protective concentrations of gasoline, diesel and BTEX (benzene, toluene, ethylbenzene, and total xylenes) from fresh-spiked total petroleum hydrocarbon products (based on toxicity point estimates). These toxicity point estimates represent the highest no-observed adverse effect concentration (NOEC) for surface water for aquatic ecological receptors. Therefore, it provides a conservative concentration that we expect to be protective for environmental effects in marine and fresh surface waters at any MTCA cleanup site. The numeric values for benzene, toluene, ethylbenzene, and total xylenes

[1] https://apps.leg.wa.gov/WAC/default.aspx?cite=173-340-730
[2] https://app.leg.wa.gov/WAC/default.aspx?cite=173-205&full=true
[3] https://apps.ecology.wa.gov/publications/SummaryPages/1803002.html
[4] https://apps.ecology.wa.gov/publications/SummaryPages/2003008.html

(BTEX) are the reported concentrations based on those Northwest Total Petroleum Hydrocarbons – Gasoline Range Organics (NWTPH-Gx) NOEC toxicity point estimates.

The purpose of the 2020 toxicity study was to provide additional protective concentrations for a release of diesel range organics that could be considered as an event that had happened in the past (significant time prior to investigation), and natural biologic process have influenced the original chemistry of the product, or what could be considered a "weathered" release of diesel. The result was the derivation of protective concentrations of diesel range organics from a weathered source. Contaminated groundwater was used as the source of weathered DRO, with upgradient uncontaminated groundwater used for comparison to the toxicity testing.

When establishing environmental effects-based protective concentrations for surface water under MTCA, 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. 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. The toxicity tests in both of these studies were conducted using the same marine and freshwater fish and invertebrates (Ecology 2018, 2020). The organisms that were included in the studies were:

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
- Cladoceran (Ceriodaphnia dubia) – EPA-821-R-02-013, method 1002.0

3.0 Results

Table 1 lists the gasoline and diesel surface water concentrations that are predicted to be protective of aquatic receptors in marine and freshwater using the NWTPH-Gx and NWTPH-Dx methods. These concentrations satisfy the requirements for establishing Method B or Method C surface water cleanup levels that are protective of aquatic receptors under WAC 173-340-730 (3)(b)(ii) or 173-340-730(4)(b)(ii) using the NWTPH-Gx and NWTPH-Dx methods.

Table 1: Gasoline and diesel concentrations that are considered protective of aquatic receptors in marine and freshwater using NWTPH (Gx and Dx) methods. Note: "Diesel range organics" includes the sum of diesel fuels plus heavy oils.

Hazardous Substance	Protective Value Freshwater (µg/L)	Protective Value Marine Water (µg/L)
Gasoline Range Organics	1000	1700
Diesel Range Organics "Unweathered"	150	50
Diesel Range Organics "Weathered"	3000	2100
Benzene	10	23
Toluene	53	102
Ethylbenzene	12	21
Total Xylenes	57	106

4.0 When should I use the protective values?

As of the date of this memo, environmental effects-based concentrations for gasoline or diesel in surface water had not been established under applicable state or federal laws. Consequently, if using Method B or C, you must establish cleanup levels as provided in the MTCA cleanup regulation (173-340-730 (3) and (4)). Those cleanup levels must have either *no adverse effect* (under Method B) or *no significant adverse effect* (under Method C) on the protection and propagation of wildlife, fish, and other aquatic life. This implementation memorandum identifies concentrations that are predicted to have *no observed adverse effect.*

You should use these protective values when you need to establish an environmental-effects-based water concentration for sites with gasoline or diesel contamination in surface water or groundwater with the potential to discharge to surface water. If you do not use these values, then you will need to establish site-specific cleanup levels as provided in the regulation (e.g. whole effluent toxicity testing using the protocols described in chapter 173-205 WAC). In short, this implementation memorandum provides an additional off-ramp (screening tool) for sites with gasoline and/or diesel contamination to avoid additional costly environmental studies.

You may use the concentrations presented in Table 1 for gasoline, diesel and BTEX (benzene, toluene, ethylbenzene, and xylenes) compounds. However, if other contaminants are also found at the site you will need to use either:

1. Environmental effects-based concentrations found under applicable state or federal laws (see MTCA's Surface Water Cleanup Standards (WAC 173-340-730); or

2. Concentrations that are estimated to result in no adverse effects on the protection and propagation of wildlife, fish, and other aquatic life for those contaminants (if concentrations have not been established under applicable state or federal laws).

As a reminder, you will also need to establish human-health based concentrations for all site chemicals, in accordance with WAC 173-340-730 (3) or (4). The more stringent of the human health or environmental-effects-based value should then be used to show overall protection (human health and environmental effects) for the surface water or groundwater with the potential to discharge to surface water pathway.

5.0 Further study

Ecology is currently conducting a follow-up study to update the section of the Analytical Methods for Petroleum Hydrocarbons[5] (Ecology 1997) that addresses how to use silica gel cleanup for groundwater sample analysis. Specifically, the intention of the follow-up study is to provide background information and guidelines to site managers and consultants regarding the analytics of the silica gel cleanup method for groundwater samples.

6.0 References

Ecology. (2018). Environmental effects-based concentrations for total petroleum hydrocarbons (TPH): Toxicity in marine water and freshwater. (Ecology Publication No. 18-03-002.) Olympia, WA: Washington State Department of Ecology, Environmental Assessment Program. Retrieved from: https://fortress.wa.gov/ecy/publications/SummaryPages/1803002.html

Ecology. (2020). Environmental effects-based concentrations for weathered diesel-range organics: Toxicity in marine water and freshwater. (Ecology Publication No. 20-03-008.) Olympia, WA: Washington State Department of Ecology, Environmental Assessment Program. Retrieved from: https://fortress.wa.gov/ecy/publications/SummaryPages/2003008.html

Model Toxics Control Act—Cleanup Regulation. WASH. ADMIN CODE § Chapter 173-340 WAC. (2013). Retrieved from: http://apps.leg.wa.gov/wac/default.aspx?cite=173-340 and https://fortress.wa.gov/ecy/publications/summarypages/9406.html

Whole Effluent Toxicity Testing and Limits. WASH. ADMIN CODE § Chapter 173-205 WAC. (1993). Retrieved from:https://apps.leg.wa.gov/WAC/default.aspx?cite=173-205

[5] http://teams/sites/TCP/ResourceLibrary1/Shared%20Documents/97602.pdf

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Attachment A: Response to comments on the January 2019 review draft of this memo

A public comment period was held from March 7, 2019 to May 10, 2019, for the review draft of an earlier version of this document dated January 2019 that had only included protective concentrations for gasoline and fresh diesel. The following comments were received during that period and helped inform updates to this final version dated August 2021.

It appears that the public had particular concerns that Ecology should also provide the protective concentrations that were developed through the "weathered diesel" study. As a result of those concerns, Ecology expanded this memo to include those concentrations.

Comments have been presented verbatim, other than minor editing to fit the format of this document.

Comment No. 1. The fundamental flaw to this approach is that the toxicity tests establishing these values were done using unweathered diesel and gasoline, in the environment, weathering significantly changes the composition of what discharges to surface waters - there must be a mention of this key assumption in that these concentrations are based on fresh samples and they may not apply to weathered.

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 2. Fuels are complex molecular mixtures of thousands of individual compounds comprising various hydrocarbons, small amounts of other compounds such as nitrogen and sulfur, and additives. Although, fuels can generally be described as mixtures of various ranges of hydrocarbons (i.e. gasoline can generally be described as a mixture of C4 to C12 hydrocarbons and diesel can generally be described as a mixture of C10 to C22 hydrocarbons) the actual chemical composition of various gasoline and diesel products can vary widely between brands and grades, and even within brands and grades, based on the source of the crude oil used in the refining process. In the study (Ecology 2018) that formed the basis for the petroleum hydrocarbon surface water protective values (cleanup standards) listed in Implementation Memorandum No. 23 (IM 23), laboratory standards (unleaded gasoline composite standard and Diesel Fuel #2 Composite Standard) sourced from RESTEK were used to "spike" surface water at various concentrations during the whole effluent toxicity (WET)

testing experiment; both standards also contained additional chemicals methanol and methylene chloride, respectively. The composite standards contain fuels from three separate sources, and RESTEK acknowledges on their website that their gasoline and diesel composite standards exhibit lot-to-lot variability. This supports the point that fuels exhibit a high degree of variability with thousands of compounds and the composition of the laboratory fuel standards used in the study may not be chemically or toxicologically similar to fuels that have been or will be released into the environment. Therefore results of the study cannot reliably be extrapolated to understand the toxicity of fuels in the environment.

Additionally, fuels begin to undergo a weathering process immediately upon contact with the environment. Weathering occurs through volatilization, UV degradation, abiotic chemical reactions, and biologically mediated chemical reactions via microbes in soil, groundwater, sediment, and surface water. Importantly, the weathering process causes chemical changes in which hydrocarbons are converted to polar organic and other compounds. Expected polar organic compounds resulting from biodegradation of hydrocarbons fall into various families of compounds (e.g. alcohols, phenols, acids and esters, ketones, and aldehydes) but nearly all of these compounds have lower toxicity than the parent hydrocarbons. The weathering process is also unpredictable both in terms of speed and the resulting mixture of compounds that will be generated. These chemical changes add to the potential variability that may be encountered in a fuel after it is released to the environment.

Because of the high degree of variability in fuels, one study cannot reliably predict the aquatic toxicity of all gasoline and diesel products on the market and most certainly cannot predict the aquatic toxicity of products that have been released to the environment and have undergone weathering processes.

Response. See response to Comment No. 1. Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 3. The toxicity study (Ecology 2018) that forms the basis for the petroleum hydrocarbon surface water protective values (cleanup standards) has not been published in a peer reviewed journal, nor has it been independently repeated. In short, it has not been subjected to the strict review standards by which research is evaluated by the scientific community. Ecology also did not solicit comment or outside review of a work plan or quality assurance project plan prior to or after conducting the study, nor does it even appear to have gone through the appropriate evaluation required by the Model Toxics Control Act (MTCA) [WAC 173-304-702(15)&(16)]. It is inappropriate for Ecology to implement a new cleanup standard (even as guidance) for petroleum hydrocarbons based on one study alone; it is even more inappropriate for Ecology to implement a cleanup standard based on a study that has not undergone peer review and has not been validated by the scientific community.

Response. Ecology presented the project plan and solicited comments for the 2018 toxicity study[6] at the Northwest Environmental Business Council Conference on March 29th (2018), October 23rd (2019), and October 8th (2020). In general, the audience was comprised of 70 to 100 environmentalists, consultants, and representatives from the petroleum industry.
In addition, Ecology is not modifying regulatory requirements. The MTCA cleanup regulation specifies that Method B surface water cleanup levels must be at least as stringent as:

> 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 (WAC 173-340-730(3)(b)(ii)).

The requirement for Method C surface water cleanup levels is the same as for Method B, except that the cleanup levels only need to be as stringent as "concentrations that are estimated to result in no significant adverse effects" (WAC 173-340-730(4)(b)(ii)).

Ecology updated the memo to clarify that you may use these concentrations as a cleanup level with no adverse effect (Method B) or no significant adverse effect (Method C) on the protection and propagation of wildlife, fish, and other aquatic life because state or federal laws for gasoline or diesel have not yet established environmental effect based concentrations. This guidance provides an additional off-ramp (screening tool) for some sites to avoid costly, environmental studies. You may always establish site-specific protective concentrations using the protocols in Chapter 173-205 WAC, Whole Effluent toxicity Testing and Limits.

Comment No. 4. Toxicity assessments are typically conducted by compiling and reviewing numerous relevant studies from the scientific literature. The body of scientific evidence is reviewed by experts and a consensus on relative toxicity is reached. In this case there is only one study and the products in question are inherently variable both in their unaltered and weathered forms; it is scientifically inappropriate to derive toxicity and cleanup standards from a single study.

Response. Numerous studies from relevant scientific literature were reviewed and compiled before, during, and after Ecology published the 2018 toxicity study. We used EPA's Ecotoxicology database (ECOTOX) to gather endpoint information (https://cfpub.epa.gov/ecotox/). For examples of literature reviewed and cited for this study, see pp. 37, 39, 40, and References on pp. 43–45 at https://fortress.wa.gov/ecy/publications/SummaryPages/1803002.html

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[6] Ecology (2018). Environmental effects-based concentrations for total petroleum hydrocarbons (TPH): Toxicity in marine water and freshwater. (Ecology Publication No. 18-03-002.) Olympia, WA: Washington State Department of Ecology, Environmental Assessment Program. Retrieved from: https://fortress.wa.gov/ecy/publications/SummaryPages/1803002.html

Comment No. 5. IM 23 amounts to de facto rulemaking and Ecology has not gone through the appropriate evaluation required by MTCA regulation [WAC 173-304-702(15) & (16)], which requires the department to consult with the science advisory board, the department of health, and the United States Environmental Protection Agency when considering new scientific information in the establishment of cleanup levels. Ecology is not authorized to promulgate cleanup levels in the manner it is attempting in IM 23.

Response. Implementation Memo No. 23 is not an attempt at rulemaking and does not change the MTCA Cleanup Rule. The regulation applicable to the protection of the surface water environment remains the same: WAC 173-340-730(3)(b)(ii) and (4)(b)(ii). See response to Comment No. 3.

Comment No. 6. Of particular concern is Ecology's intent to promulgate a surface water cleanup level for an extremely complex mixture of compounds based on a single study that tested only one mixture of each fuel when there are thousands of variations and mixtures that could produce different results. Additionally, while indicating that the study was done with "fresh-spiked" total petroleum hydrocarbons (TPH), IM 23 does not acknowledge the absence of weathered TPH testing. Ecology's means and methods in this case are ill-conceived and have not been scientifically vetted and validated.

Response. Ecology updated the memo to clarify that you may use these concentrations as a cleanup level with no adverse effect (Method B) or no significant adverse effect (Method C) on the protection and propagation of wildlife, fish, and other aquatic life because state or federal laws for gasoline or diesel have not yet established environmental effect based concentrations. This guidance provides an additional off-ramp (screening tool) for some sites to avoid costly, environmental studies. You may always establish site-specific protective concentrations using the protocols in Chapter 173-205 WAC, Whole Effluent toxicity Testing and Limits.

Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 7. Although the scientific literature using similar methods for testing toxicity of petroleum hydrocarbon mixtures is limited, it does exist. IM 23 does not acknowledge or discuss the results of other scientific studies on this subject, many of which are highly variable and determined NOEC and LOEC values for aquatic organisms that are considerably higher than the values being proposed by Ecology. At a minimum, Ecology should compile and provide an evaluation of related existing research.

Response. Numerous studies from relevant scientific literature were reviewed and compiled before, during, and after Ecology published the 2018 toxicity study. We used EPA's Ecotoxicology database (ECOTOX) to gather endpoint information (https://cfpub.epa.gov/ecotox/). For examples of literature reviewed and cited for this study, see pp. 37, 39, 40, and References on pp. 43–45 at https://fortress.wa.gov/ecy/publications/SummaryPages/1803002.html

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Comment No. 8. Because Ecology's methodology behind the cleanup standards does not meet scientific rigor, there is a significant risk that the standards will be subject to litigation that will require use of tax payer dollars to defend. It is inappropriate of Ecology to "set up" the tax payers of Washington for expensive litigation when those dollars could be better utilized to facilitate site cleanup.

Response. Ecology updated the memo to clarify that you may use these concentrations as a cleanup level with no adverse effect (Method B) or no significant adverse effect (Method C) on the protection and propagation of wildlife, fish, and other aquatic life because state or federal laws for gasoline or diesel have not yet established environmental effect based concentrations. This guidance provides an additional off-ramp (screening tool) for some sites to avoid costly, environmental studies. You may always establish site-specific protective concentrations using the protocols in Chapter 173-205 WAC, Whole Effluent toxicity Testing and Limits.

Comment No. 9. Of concern is Ecology's intent to apply these standard universally to petroleum release sites regardless of the age of the fuel release. Weathered fuel products may exhibit significantly lower toxicity than fresh fuel products and there is no defensible way to extrapolate the concentrations in IM 23 to weathered fuel products. Without a comparable and comprehensive study with fuels that have undergone varying degrees of weathering, Ecology has not adequately shown the range of possible environmental effects that may or may not be observed at similar dosages. If Ecology moves forward with promulgating surface water cleanup levels for TPH in IM 23 (not recommended), the memorandum should acknowledge these facts and provide a caveat that the concentrations provided apply only to fresh fuel releases.

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 10. In IM 23, Ecology offers an alternate means to demonstrate toxicity using Whole Effluent Toxicity (WET) testing. However, this method is extremely expensive and collecting the necessary sample volumes, at the required frequency, from representative locations can range from cumbersome to impossible. In short, WET testing (as the only viable alternative to accepting Ecology's test values – including essentially non-detect values for diesel – see #5 below) can be impractical to impossible at many sites. It also has the potential to add significant time and cost to remedial investigations, the ultimate effect of which will be to further delay cleanups.

Response. WET testing is included as an alternative in the MTCA Cleanup Rule, WAC 173-340-730(3)(b)(ii). It may be used on a site-specific basis and as an alternative to the predicted "no-observed adverse effect concentration" established in Implementation Memorandum No. 23.

Comment No. 11. The accepted practical quantitation limit (PQL) for diesel range organics in water with standard laboratory techniques is 250 micrograms per liter (µg/L). Both the fresh and marine water protective values listed in IM 23 are less than the PQL. Ecology should acknowledge this fact and indicate that adjustment upward to the PQL (250 µg/L at the time of publication of the memo) is acceptable.

Response. Discussion of PQLs and their role in establishing cleanup levels is outside the scope of Implementation Memorandum No. 23. However, Ecology agrees and acknowledges the point in Comment No. 11: an adjustment upward to an appropriate PQL is acceptable under the MTCA Cleanup Rule, WAC 173-340-730(5)(c).

Comment No. 12.

[Part 1]. Ecology has recently taken a (legally questionable) position at many sites that if surface water is a potential pathway at a cleanup site, groundwater cleanup levels must be equal to the surface water cleanup levels. Under this policy position, the surface water cleanup levels for TPH will become de facto groundwater cleanup levels. Ecology will be asking PLPs to clean up groundwater to non-detect concentrations of petroleum hydrocarbons (when accounting for the PQL).

[Part 2]. Ecology also does not currently allow the use of silica gel cleanup with the NWTPH methods, so measurements include naturally occurring organic compounds. Ecology will conceivably be asking potentially liable parties (PLPs) to clean up naturally occurring organic compounds in groundwater and surface water to non-detect concentrations, a requirement that is neither reasonable nor feasible. Promulgation of the protective values as cleanup levels will add excessive time and cost (in the range of years to decades of time and tens of thousands to millions of dollars in cost) to remediate petroleum hydrocarbon contaminated sites, without a clear environmental benefit. The effect will be to slow down or stall the remediation of many petroleum hydrocarbon contaminated sites. In many cases the cleanup levels may be unachievable in groundwater within any reasonable restoration timeframe and/or lead to many

feasibility study/disproportionate cost analysis results concluding that any active cleanup of TPH sites would disproportionately costly (especially for diesel sites with non-detect cleanup levels).

Response. Part 1 of Comment No. 12 appears to be a comment that is beyond the scope of this study.

Part 2 of Comment No. 12 pertains to the use of silica gel. It is important to note that there were no effects of silica gel on the NOEC's for the freshly spiked diesel. In the follow-up study (with protective concentrations provided) to document the effects of weathered diesel on aquatic organisms in surface waters, silica gel did have an impact (please see Table D-4 in Appendix D – Water Chemistry Results - . Environmental effects-based concentrations for weathered diesel range organics: Toxicity in marine water and freshwater[7]) (Ecology 2020). However, due to the added uncertainty of the silica gel cleanup process (cleanup, age of diesel range organics, amount of released of petroleum metabolites etc.), the values provided were without the use of silica gel.

Comment No. 13. Ecology's desire to protect aquatic organisms is understandable. However, Ecology must first undertake a rigorous evaluation of available scientific research; fill any gaps in the scientific research (such as evaluating multiple fresh and weathered product formulations); summarize and present that research to the public; and consult with the science advisory board, the department of health, and the United States Environmental Protection Agency as required under MTCA [WAC 173-340-702 (15)] before establishing cleanup levels.

Response. Implementation Memo No. 23 is not an attempt at rulemaking and does not change the MTCA Cleanup Rule. The regulation applicable to the protection of the surface water environment remains the same: WAC 173-340-730(3)(b)(ii) and (4)(b)(ii). See response to Comment No. 3. Ecology updated the memo to clarify that you may use these concentrations as a cleanup level with no adverse effect (Method B) or no significant adverse effect (Method C) on the protection and propagation of wildlife, fish, and other aquatic life because state or federal laws for gasoline or diesel have not yet established environmental effect based concentrations. This guidance provides an additional off-ramp (screening tool) for some sites to avoid costly, environmental studies. You may always establish site-specific protective concentrations using the protocols in Chapter 173-205 WAC, Whole Effluent toxicity Testing and Limits.

In addition, Ecology acknowledges that the study was completed using "fresh spiked" total petroleum hydrocarbons (TPH), which was the purpose of Ecology's 2018 toxicity study. However, Ecology has revised Implementation Memorandum No. 23 to also include the follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

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[7] https://apps.ecology.wa.gov/publications/SummaryPages/2003008.html

Comment No. 14.

Comment #1

Single point estimates of chronic toxicity to four aquatic species (two freshwater and two marine) are not sufficient to form the basis of a screening level. As evident in the reported results there is inherent variability in:

a. Test organism response both within and between species.

b. Gasoline and diesel constituents (e.g. dominant carbon ranges, additives) based on the wide variation in total petroleum hydrocarbon (TPH) sources materials and refining methods.

c. How the Water-accommodated Fraction (WAF) concentrations are prepared and the degree to which these fractions can degrade quickly.

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MEMO

i. Actual test concentrations of gasoline were 59-85% of the nominal concentrations and actual concentrations of diesel were 3-30% of the nominal concentrations.

ii. This variability in WAF concentrations was demonstrated by comparing measured "fresh" as compared to "stale" concentrations. Since these screening levels are intended to be used to screen fresh TPH samples, what will be considered as "fresh"?

Given these uncertainties, additional testing should be conducted to confirm and verify the results prior to proposing protective values.

Comment #2

These proposed values are overall lower than other recently published screening levels as summarized in the Interstate Technology & Regulatory Council (ITRC) 2018 report *TPH Risk Evaluation at Petroleum-Contaminated Sites*, and values reported in the ESL Workbook dated February 2016 (rev 3) by the San Francisco (SF) Regional Water Quality Control Board (RWQCB). Comparative screening levels are summarized in the table below.

	Washington State		Cited in Table 7-1 ITRC 2018 (except BET values)				
	2019 Draft Protective Value		SF RWQCB (Feb 2016 rev 3) [1]		Hawaii (HIDOH 2017)		Canada Atlantic Partnership (2012)
Hazardous Substance	Fresh	Marine	Fresh	Marine	Fresh	Marine	Fresh and Marine
Gasoline Range Organics	1000	1700	**443**	3700	**500**	3700	1500
Diesel Range Organics	150	50	640	640	640	640	**100**
Benzene	10	23	46	700			
Toluene	53	102	130	5000			
Ethylbenzene	12	21	290				
Total Xylenes	57	106					

Notes:
Units are µg/L
Fresh = freshwater
BET = benzene ethylbenzene toluene
SF = San Francisco
RWQCB = Regional Water Quality Control Board
HIDOH = Hawaii Department of Health
[1] ESL Workbook Table IP-5 Aquatic Habitat Goals for TPH and Table IP-6 for BET
Lower than the proposed Washington State Draft Protective Value

The benzene, ethylbenzene, toluene and xylene (BTEX) screening levels are particularly uncertain since only the SF RWQCB has screening levels to compare against and none for xylene. The marine values are consistently higher than the freshwater values (except for where the marine value was adopted as the freshwater value) making the Washington State proposed marine value for Diesel Range Organics suspicious since it is lower than the freshwater value. This lack of consistency with other values provides further support for the conclusion that additional testing is necessary before protective values are proposed.

Figure 1: Screenshot of comments sent by one reviewer on May 10, 2019. Ecology received the comments as an Adobe PDF memo and summarized them in Comment No. 14.

Response to Comment No. 14's comment #1.[8]

Comment #1 addresses the concern that there is too much variability in the tests to pin a single number to.

Ecology believes it would an unrealistic scope of work to continue testing all possible factors that can influence the variability of hydrocarbon toxicity. The variability in the daily test solutions is incorporated into the calculations of the effects thresholds, because the effects thresholds represent actual exposure concentrations.

The data we present on the stale solutions describe how hydrocarbons are lost during the test to organism uptake and volatilization. It is not feasible to maintain a constant exposure concentration on the organism without loss.

The variability in the water accommodated fraction (WAF) concentration versus the nominal concentration is due to the solubility of the mixtures; however, all test results are reported against the measured exposure concentrations.

Response to Comment No. 14's comment #2.

Ecology respectfully disagrees. The proposed values in Implementation Memo No. 23 are consistent with literature cited by the commenter, as well as other literature researched prior to and during the development of these values.

Points of clarification:

Although Comment No. 14 appears to cite three sources of literature, there were actually only two. Hawaii's Department of Health (HIDOH) values were derived from two California EPA (CAEPA) studies in 1998 and 1999.[9] See the HIDOH study footnotes that cite "CAEPA (1998, 1999)."

The 2019 user's guide by the San Francisco Bay Regional Water Quality Control Board (SFB RWQCB)[10] also appears to have carried forward CAEPA's values from 1998 and 1999. In the SFB RWQCB test, one species was tested for gasoline (Ceriodaphnia dubia), and one species

[8] Response prepared using correspondence from:
Curtis Eickhoff, Ph.D., Nautilus Environmental Company Inc., www.nautilusenvironmental.ca; and
Will Hobbs, Ph.D., Washington State Department of Ecology, www.ecy.wa.gov

[9] HIDOH. (2018). Collection and use of total petroleum hydrocarbon data for the risk-based evaluation of petroleum releases. Honolulu, HI: Hawai'i Department of Health, Hazard Evaluation and Emergency Response Office. Retrieved from: http://eha-web.doh.hawaii.gov/eha-cma/documents/e4c45bf6-f052-43a6-8372-b1f2df2758d0

[10] SFB RWQCB. (2019). User's guide: Derivation and application of environmental screening levels (ESLs). Oakland, CA: San Francisco Bay Regional Water Quality Control Board. Retrieved in August 2019 from: https://www.waterboards.ca.gov/sanfranciscobay/water_issues/programs/esl.html and https://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=2&ved=2ahUKEwiIu4HP5_PjAhWK_p8KHf_RDkUQFjABegQIBRAC&url=https%3A%2F%2Fwww.waterboards.ca.gov%2Fsanfranciscobay%2Fwater_issues%2Fprograms%2FESL%2FESL_Users%2520Guide_19.pdf&usg=AOvVaw10LAYHr2kwZzq4i7Adn_zH

(Americamysis bahia) was tested for diesel (based on petroleum jet fuel saltwater value as a surrogate) (SFB RWQCB 2019).

Values from Canada Atlantic Partnership (2012) do appear consistent with Ecology proposed values.

Comment No. 15. The toxicity test program that was used to develop the TPH effects-based concentrations has several issues that make the proposed "protective values" uncertain, particularly for diesel-range organics.

The concentration and composition of dissolved hydrocarbons generally determines the aquatic toxicity of physically and chemically dispersed oils (Redman and Parkerton 2015). While use of fresh gasoline and diesel to evaluate concentrations protective of surface waters may be applicable to cleanup of fresh spills, it is not appropriate for application at legacy MTCA cleanup sites with weathered petroleum constituents. WET testing results on weathered diesel constituents suggests protective concentrations on the order of 700 µg/L, more than four times the value proposed for freshwater in the implementation memo. The gasoline and diesel test materials used to develop these new values were supplied in carriers of methanol and acetone, respectively.

Redman, A.D., and T.F. Parkerton, 2015. "Guidance for Improving Comparability and Relevance of Oil Toxicity Tests." Marine Pollution Bulletin, 98: 156-170.

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 16. The variability between nominal and measured concentrations was higher for the diesel test than for the gasoline tests. This difference may be indicative of entrained droplets of oil. Droplet interaction would be expected to be most substantial with organisms having a high surface area to volume ratio.

Response.[11] For both gas and diesel, the test waters were prepared by injecting very small amounts of hydrocarbon spikes in solvent carriers into the stock water. The procedure that was conducted by staff at Nautilus Environmental Company Inc.:

> **Diesel (saltwater testing):** To achieve 32 mg/L in a 5.5L aspirator bottle, we added 3.52 mL of the 50,000 mg/L diesel stock solution.
>
> **Diesel (freshwater testing):** To achieve 60 mg/L in a 5.5L aspirator bottle, we added 6.6 mL of the 50,000 mg/L diesel stock solution.

[11] Response prepared using correspondence from: Curtis Eickhoff, Ph.D., Nautilus Environmental Company Inc., www.nautilusenvironmental.ca; and Will Hobbs, Ph.D., Washington State Department of Ecology, www.ecy.wa.gov

The spiked water was mixed for 24h and allowed to settle for at least one hour. The water was then drained via a port at the bottom of the aspirator bottle. Moreover, the Ceriodaphnia dubia tests were conducted using glass test tubes. Analysts can see the test solutions clearly through the test tubes when counting test organism neonates. Staff did not note any visible droplets in the test solutions. There was often a visible sheen on the surface of the test waters. It is possible that micro-droplets may have been there that we could not see. Because we used such small volumes of gasoline and diesel, we think the amount of droplet production would have been very small. This can be a problem when large volumes of test solution are used in water, however in our case, we do not believe it was a factor. The mixing process is specifically designed to produce the water accommodated fraction of the hydrocarbon (i.e. the dissolved fraction) and to prevent the influence of oil droplets on the test organisms. It's likely the variability in Dx concentrations is a function of the difficulty of creating a WAF [water accommodated fraction] with a low solubility hydrocarbon mixture for a daily renewal toxicity test.

Comment No. 17. Of the four species that were tested, the two species that would be most susceptible to physical fouling by droplets would be echinoderm eggs and Ceriodaphnia dubia. Interestingly, these two species were the most sensitive to diesel. At the least, the echinoderm and Ceriodaphnia tests should be rerun and concentrations validated using passive sampling to quantify the dissolved fraction and additional analysis to characterize saturated hydrocarbons that are diagnostic of droplets (Redman and Parkerton 2015). Also, the fact that the toxicity values from the gasoline exposures are similar for the fish and invertebrates suggests that the dissolved fractions were driving the toxicity for the gasoline tests, but that this was not necessarily the case in the diesel test.

Response.[5] Because the tests rely on the water accommodated fraction, and droplet exposure would be minimal given the stock water preparation techniques, additional testing on the Ceriodaphnia and Echinoderm eggs would not seem necessary. Ecology did not believe it was necessary to invoke the influence of droplets when the toxicity to gas and diesel for the biological endpoints differs. Reasons: the uptake and metabolism of various polycyclic aromatic hydrocarbons contained in the diesel fuel would vary between organisms. The observation that aquatic organisms are more sensitive to diesel fuel than gas has been documented in other publications.

Comment No. 18. The memorandum states: "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." We are concerned that Ecology is proposing new cleanup standards in advance of already planned MTCA rule revisions. The study on which these levels are based is one source of information that can be used in that planned rule-making process, but it is not definitive and needs to be considered along with other information at that time.

Response. Implementation Memo No. 23 is not an attempt at rulemaking and does not change the MTCA Cleanup Rule. The regulation applicable to the protection of the surface water

environment remains the same: WAC 173-340-730(3)(b)(ii) and (4)(b)(ii). See response to Comment No. 3.

In addition, Ecology acknowledges that the 2018 toxicity study was completed using “fresh spiked” total petroleum hydrocarbons (TPH), which was the purpose of the study. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 19.

General Comment – We commend Ecology's efforts for developing additional screening tools to help navigate the MTCA process. As a screening step intended to avoid false negatives, these values could serve a purpose (i.e., identify the potential for impacts, but not necessarily the occurrence of impacts) and trigger additional investigation activities that provide lines of evidence to assess whether actual impacts are occurring. However, because these values were generated using fresh product, their use as preliminary cleanup criteria could greatly overestimate the potential for adverse risk.

Petroleum Composition – Ecology states the purpose of Draft Implementation Memorandum No. 23 is to provide fresh gasoline and diesel range organic concentrations that are predicted to be protective of aquatic receptors in marine and fresh surface waters at any Model Toxics Control Act (MTCA) cleanup site." Does Ecology intend for these environmental effects values to be applied to release sites where the petroleum body is comprised of weathered product?

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 20. In addition, how would these values be used at sites where there may be a mixture of contaminants?

Response. Ecology updated the memo with a reference to values or methods for establishing values for other contaminants.

Comment No. 21. The values were developed using fresh hydrocarbon mixtures and don't consider the effects of weathering on mixture composition. As such, these values could be overly conservative for historical releases/legacy site issues. Will Ecology provide additional guidance or criteria for sites where the petroleum product is weathered?

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 22. Has Ecology considered how it will regulate proposed cleanup values for sites where the compositional structure of the petroleum hydrocarbon body in soil or groundwater is significantly different from fresh product (e.g. weathered diesel)?

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 23. It appears that the diesel solution used for the experiment was prepared using acetone to maximize its solubility in toxicity test water. What are the toxicological impacts on aquatic organisms from mixing acetone in with the diesel mixture?

Response.[6] This issue was one of concern during the study and was addressed through the following procedure:

We had Restek manufacture the hydrocarbon spikes using the minimum amount of solvent necessary. During the toxicity tests we also ran a negative lab control (blank water) and a positive lab control with solvent. Each set of tests had a separate acetone control based on the amount of acetone used in the top concentration of the test. For Topsmelt and Echinoderms tests, a 0.036% acetone solution was used. For fathead minnow and C. dubia, the percentage of acetone used in the solvent control solution was 0.067 and 0.012% acetone, respectively. The acetone control was prepared in separate containers on the day that the tests were set up and for all test solution renewals.

Calculations of effects concentrations presented by Ecology represent the comparison of toxicity test dilution series to the negative control which contained solvent in order to make them comparable. The survival in the negative solvent control and lab control were both 100%. The reproduction (mean ± SD) of the lab control was 20.2 ± 2.1 and the reproduction of the negative solvent control was 16.8 ± 4.4.

In addition, we calculate that there would have been 640 uL acetone/L in the top concentration of the saltwater solutions, and 1200 uL acetone/L in the top concentration of the freshwater solutions for the diesel samples. According to LeBlanc and Surprenant (1983)[7] the 48-h LC50 for acetone in D. magna was 39,000 (31,000 - 53,000) uL acetone/L. The concentrations of acetone that we were using are many orders of magnitude below this LC50 value. Therefore, we feel that the effects of acetone on the test organisms would have been negligible.

Regarding the chronic toxicity of acetone: LeBlanc and Surprenant exposed D. magna for 28 days in chronic tests and found that:

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[6] Response prepared using correspondence from:
Curtis Eickhoff, Ph.D., Nautilus Environmental Company Inc., www.nautilusenvironmental.ca; and
Will Hobbs, Ph.D., Washington State Department of Ecology, www.ecy.wa.gov

[7] LeBlanc, G.A., and Surprenant, D.C. (1983). The acute and chronic toxicity of acetone, dimethyl formamide, and triethylene glycol to Daphnia magna (Straus). Archives of Environmental Contamination and Toxicology. 12:3 305–310. Retrieved from: https://link.springer.com/article/10.1007%2FBF01059407

> The number of offspring produced by daphnids exposed to 690 and 1,400 uL acetone/L was comparable to the number of offspring produced by control daphnids for the duration of the exposure. Based on the reduced survival of daphnids exposed to 2800 ul acetone/L, the MATC was between 1,400 and 2,800 uL acetone/L.

The concentrations of acetone used in our study was at or below concentrations that showed no effect on reproduction of D. magna over a period of 28d by LeBlanc and Surprenant, 1983. Therefore, we may say that the acetone is likely to not have caused effects in our shorter term sublethal experiments.

Comment No. 24. Based on Implementation Memorandum No. 23, we understand that site-specific testing activities could be performed that focuses on individual constituents rather than a broad non-specific hydrocarbon mixture. Ecology's own publications indicate that ecological toxicity information is lacking for mixtures and assessments should be site-specific. Specifically, Ecology states:

The underlying study indicates that "the recommended surface water concentration of diesel that is protective of freshwater aquatic receptors is 150 ug/l". The 150 ug/l value equates to the lowest no-observed-effect concentration (NOEC) derived for a freshwater invertebrate, Ceridaphnia dubia. Ecology (2018) reports that they were "unable to find data on the impacts of a similar petroleum mixture on freshwater invertebrates". Does Ecology have plans to conduct additional testing to supplement the available NOEC data for invertebrates exposed to low concentrations of diesel?

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters. This weathered diesel (used in the study) provided a broad hydrocarbon mixture. Please note that the protective concentrations for “weathered diesel” were not as conservative as the “unweathered” or fresh-spiked diesel.

Comment No. 25. Ecology (2018) indicates: "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". Will toxicity tests using weathered diesel product be conducted prior to implementing the use of the proposed values?

Reference: Ecology. 2018. Environmental Effects-Based Concentrations for Total Petroleum Hydrocarbons (TPH), Toxicity in Marine Water and Freshwater. Publication No. 18-03-002.

Response. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 26. Section 1.0, 2nd paragraph, 1st sentence. Recommended the following change (added language in bold): “Cleanup project managers and consultants should use these surface water protective values (called environmental effects) at MTCA Cleanup sites **involving recent releases of fresh petroleum products**…”. The study cited by the Draft Memorandum evaluated only the effects of fresh gasoline and diesel on aquatic life, whereas the majority of cleanup sites involve historical releases of petroleum products that have undergone significant weathering and degradation. Available research (e.g., Zemo 2013; Mohler 2013) indicate that weathered/degraded petroleum mixtures likely have lower ecological toxicity than fresh petroleum, indicating that these values would be overly conservative for most cleanup sites. This fact should be noted in the memo.

Response.
Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. Ecology has revised Implementation Memorandum No. 23 to include a statement that these values could be applicable for releases of unweathered gasoline and diesel. In addition, Ecology has provided a follow-up study (with protective concentrations) to document the effects of weathered diesel on aquatic organisms in surface waters.

Comment No. 27. Section 2.0, second paragraph. The second sentence “These estimates represent the highest observed no-adverse effects concentration (NOEC) for surface water towards ecological receptors.” Should be revised to refer to the “most stringent” NOEC, rather than “highest observed”.

Response. Ecology has determined that the statement is correct as it stands: we would be unable to measure anything lower than the highest observed no-adverse effects concentration, because it would need to be placed in relation to the lowest observed adverse effects concentration.

Comment No. 28. The Protective Values for Diesel Range Organics in freshwater and marine water are lower than the PQL of 250 ug/L as identified in Table 2 of the study (Ecology 2018: Publication No. 18-03-002). These values should be adjusted upward to the PQL in accordance with MTCA.

Response. The reviewer is correct in that a final cleanup level could be adjusted upwards from a protective value to either the practical quantitation limit or natural background, whichever is greater. However, determination of final cleanup levels is beyond the scope of Implementation Memorandum No. 23.

Comment No. 29. Review of the collective test results for DRO in Table 26 of the toxicity study indicates that the <50 ug/L protective value (NOEC) for DRO is an outlier. Considering the statewide regulatory implications of applying such a low value (10 percent of the groundwater cleanup level that is applied on almost every site), that value should be removed as an outlier, or at least subject to further confirmation testing.

Response. An outlier test was conducted on the collective test results for DRO using EPA's ProUCL 5.1. Please note that 0.05 mg/L was not calculated as a statistical outlier at the 10%, 5%, or 1% significance level (Dixon's Outer Test) when tested against the collective results. Test statistic was 0.080, the 10% critical value = 0.434, 5% critical value = 0.507, 1% critical value = 0.637. Additionally, Statistical Guidance for Ecology Site Managers (Ecology 1992) states: "There are no provisions in MTCA for excluding "outliers" that cannot be demonstrated to be in error." [8]

Comment No. 30. The Protective Values identified for BTEX compounds are not appropriate based on the study that is the source of these values (Ecology 2018: Publication No. 18-03-002), and these values should be removed from the document. The study indicates the following:

"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)."

"Comparison of our findings to other studies found in EPA's EcoTox database is difficult because we used an unweathered gasoline standard and studies in EcoTox generally use either weathered hydrocarbons or specific BTEX compounds…In freshwater, concentrations for NOEC of BTEX on fathead minnows range from 5,400 to 10,200 ug/L (Marchini et.al., 1992).

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[8] Ecology. (1992). Statistical guidance for Ecology site managers (includes the S-6 supplement). (Ecology Publication No. 92-54.) Olympia: WA: Washington State Department of Ecology, Toxics Cleanup Program. Retrieved from https://fortress.wa.gov/ecy/publications/SummaryPages/9254.html

These results are significantly higher than the range of BTEX compounds found in the NWTPH-Gx NOEC for our study."

In other words, the study did not directly evaluate the effects of individual BTEX compounds, and the BTEX values identified were far lower than published literature values that specifically evaluated BTEX compounds. Therefore, there is no basis for using these values as indicative of BTEX toxicity.

Response. The purpose of the 2018 toxicity study was to find no observed effects levels for TPH contamination in unweathered product, and the BTEX compounds that were tested were indicative of those found in unweathered TPH contaminated sites. Ecology updated the memo to clarify that you may use these concentrations as a cleanup level with no adverse effect (Method B) or no significant adverse effect (Method C) on the protection and propagation of wildlife, fish, and other aquatic life because state or federal laws for gasoline or diesel have not yet established environmental effect based concentrations. This guidance provides an additional off-ramp (screening tool) for some sites to avoid costly, environmental studies. You may always establish site-specific protective concentrations using the protocols in Chapter 173-205 WAC, Whole Effluent toxicity Testing and Limits.