U.S. Tire Manufacturers Association

On behalf of the U.S. Tire Manufacturers Association, please see the attached comments in response to Washington Ecology's proposed revisions to chapter 173-201A WAC, Water Quality Standards for Surface Waters of the State of Washington.

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Submitted electronically
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Re: Proposed updates to Aquatic Life Toxics Criteria, WAC 173-201A-240

The U.S. Tire Manufacturers Association (USTMA) appreciates the opportunity to provide comments on Washington Ecology's Proposed updates to its Aquatic Life Toxics Criteria, WAC 173-201A-240 (hereinafter referred to as "the Criterion"). USTMA is the national trade association of tire manufacturers that produce tires in the United States. Domestic tire manufacturing is responsible for more than 291,000 jobs and has an annual economic footprint of $170.6 billion in the United States. The tires from our member companies make mobility possible and keep the U.S. economy moving. USTMA advances a sustainable tire manufacturing industry through a commitment to science-based public policy advocacy.

While USTMA supports Washington Ecology's efforts to better understand the impacts of N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine (6PPD) and 6PPD-quinone (6PPDQ) in the environment, we have multiple concerns with the proposal as written that should be addressed before the agency moves forward. The association recognizes the cultural importance of the coho salmon to Tribes in the Pacific Northwest. Since 6PPDQ was first identified in Tian et al., 2021[1], USTMA has engaged with Washington state, federal, and Tribal agencies, researchers, and other stakeholders to identify and support existing and future research related to 6PPDQ and to ensure research utilizes the most robust methodologies. USTMA continues to support the use of the best available, peer-reviewed science to inform regulatory actions. The association's members are committed to working with partners, including Washington Ecology, to fill knowledge gaps in existing research.

[1] Tian, Z; Zhao, H; Peter, KT; et al. 2021. A ubiquitous tire rubber-derived chemical induces acute mortality in coho salmon. Science 371(6525):185-189. doi: 10.1126/science.abd6951.

After reviewing the Technical Support Document for the Criterion, USTMA identified multiple concerns that should be addressed. Most pressing is that the referenced, applicable guidance, US EPA's 1985 Guidelines for Deriving Numerical National Water Quality Criteria for the Protection of Aquatic Organisms and Their Uses, Stephen et al., 1985[2] (hereinafter referred to as "the 1985 Guidelines"), was not followed consistently or appropriately. The agency does not provide sufficient justification for when it deviates from the 1985 Guidelines to warrant setting a criterion with a significant lack of data. Until this is addressed, USTMA believes that Washington Ecology should reevaluate setting a water quality criterion for 6PPDQ. In addition to this concern, USTMA has identified a number of technical inconsistencies and errors in the proposal that should be addressed. USTMA's comments outline these issues and concerns and provide several recommendations and requests to remedy these areas. USTMA requests that the agency makes the appropriate edits and repropose the Criterion for 6PPDQ for additional public review.

1. WASHINGTON ECOLOGY'S APPROACHES TO DERIVE AN ACUTE AQUATIC LIFE VALUE FOR 6PPDQ LACK TRANSPARENCY AND REPRODUCIBILITY

For 6PPDQ, several approaches were utilized to derive an acute aquatic life value. Given the limited availability of data for 6PPDQ toxicity in aquatic organisms included by Washington Ecology, multiple refinements were made. However, the document lacks transparency regarding the agency's reasoning and methodologies, which inhibit the replication of the agency's work.

The standard derivation method in the 1985 Guidelines that Washington Ecology used to derive aquatic life criteria requires toxicity data from at least eight taxonomic families. Washington Ecology was unable to derive a criterion value using this method due to insufficient data. To account for this lack of data, the agency utilized the Web-based Interspecies Correlation Estimation (WEB-ICE) model to estimate toxicity data for the missing families. This method does not follow the 1985 Guidelines, which states:

> Criteria should attempt to provide a reasonable and adequate amount of protection with only a small possibility of considerable overprotection or underproduction. It is not enough that a national criterion be the best

[2] Stephen, C. E.; Mount, D. I.; Hansen, D.J.; Gentile, J.R.; Chapman, G.A.; Brungs, W.A. 1985. Guidelines for Deriving Numerical National Water Quality Criteria for the Protection of Aquatic Organisms and their Uses. U.S. Environmental Protection Agency. Guidelines for Deriving Numerical National Water Quality Criteria for the Protection of Aquatic Organisms and Their Uses (epa.gov)

> estimate that can be obtained using available data; it is equally important that a criterion be derived only if adequate appropriate data are available to provide reasonable confidence that it is a good estimate…. If all the required data are not available, usually a criterion should not be derived [emphasis added].

The 1985 Guidelines clearly emphasize that a standard should not be set without the appropriate data, which Washington Ecology does not have. Without sufficient data to set a water quality standard for 6PPDQ, Washington Ecology should reconsider the Criterion for 6PPDQ.

Although Ecology reports the 6PPDQ WEB-ICE results in Appendix C, it is unclear which of these results (e.g., which LC50s) were incorporated into the "eight-family method" to fill in toxicity values for the three missing taxonomic families. Therefore, it is not possible to replicate this method, so we were unable to verify the resulting value of 46 ng/L as reported by Washington Ecology (p. 118).

Additionally, Washington Ecology's reference to "EPA's single species alternative method" is unclear. USTMA assumes that the agency is referring to using the geometric mean of the acute values for the most sensitive species (i.e., coho salmon), which is recommended in the 1985 Guidelines when this value is lower than the calculated Final Acute Value (based on the eight-family method).

Furthermore, for the species sensitivity distribution (SSD) using data available data for 6PPDQ provided in Table 55, and the methodology described in subsequent text for derivation of an aquatic life criterion for 6PPDQ (e.g., use of EPA's SSD Toolbox), USTMA was unable to replicate or verify a species sensitivity distribution 5th (LC5) percentile value of 0.008 µg/L.

USTMA requests that Washington Ecology provide additional information in the Technical Support Document that would enable verification of the Criterion derivation approaches, such as information on which specific EPA SSD tool was used, which SSD distribution was selected (e.g., normal, logistic), which fitting method was used (e.g., maximum likelihood, linearization), and the raw data used in the analysis to increase transparency and reproducibility.

2. WASHINGTON ECOLOGY DOES NOT PROVIDE A JUSTIFIED RATIONALE FOR DEVIATING FROM -THE 1985 GUIDELINES

The proposed acute aquatic life value is 0.008 µg/L (1-hour) for freshwater organisms. This means that freshwater aquatic organisms and their uses should not be affected unacceptably if the 1-hour average concentration of 6PPDQ does not exceed 0.008 µg/L. However, there is a lack of rationale for the decisions for refinements that Washington Ecology used to derive the Criterion. The agency utilized an inappropriate interpretation of the 1985 Guidelines which served as the basis for these derivations.

Within the Technical Support Document, Washington Ecology does not provide an adequate technical rationale for deviating from the 1985 Guidelines. The rationale for performing a "species sensitivity distribution, rather than a genus sensitivity distribution" is not consistent with the 1985 Guidelines for deriving water quality criteria. From the 1985 guidelines:

> The Final Acute Value is now defined in terms of Genus Mean Acute Values rather than Species Mean Acute Values. A Genus Mean Acute Value is the geometric mean of all the Species Mean Acute Values available for species in the genus. On the average, species within a genus are toxicologically much more similar than species in different genera, and so the use of Genus Mean Acute Values will prevent data sets from being biased by an overabundance of species in one or a few genera.

Ecology's decision to use species instead of genus results in a heavily biased distribution by omitting effect values of more resistant species, which would normally be accounted for in a Final Acute Value derived according to the 1985 Guidelines. Indeed, the basis for inclusion of only certain species in Washington Ecology's distribution is not fully explained.

Washington Ecology states that the 8 ng/L value derived using the SSD approach is sufficiently protective because it is below the currently reported LC5 values for coho. However, the 1985 Guidelines do not make any recommendations to compare final criterion values to a single species LC5 value when determining sufficiency of protection.

USTMA requests that Washington Ecology provide a rationale and reference in the Technical Support Document to support the exclusion of 6PPDQ data for tolerant species and further justification to support a "species sensitivity distribution" rather than a "genus sensitivity

distribution". Additionally, USTMA recommends that Washington Ecology provide rationale and reference for comparing the criterion value to a LC5 to support reasonable protection.

The agency also calculated the species mean acute value using only toxicity data from coho salmon studies in accordance with the 1985 Guidelines (i.e., single species method) to obtain the Criterion value of 34 ng/L. However, Washington Ecology concluded that this value was not protective of coho because the toxicity tests were conducted over 24 hours, instead of the standard 96 hours. Further, the agency cites the findings of Brinkmann et al., 2022[3] to indicate a 2-fold increase in toxicity to 6PPDQ from 24 to 96 hours in rainbow trout. No technical justification is provided for this generalization across species and dose-responses to assume that there will be a two-fold increase in toxicity from 24h to 96h for coho salmon.

USTMA recommends that the 1985 Guidelines be followed when deriving a criterion value for a commercially or recreationally important species (i.e., coho salmon) that shows sensitivity to the chemical of interest. The 1985 Guidelines *state that*

> *if for a commercially or recreationally important species the geometric mean of the acute values from the flow-through tests in which the concentrations of test material were measured is lower than the calculated Final Acute Value, then that geometric mean should be used as the Final Acute Value instead of the calculated Final Acute Value (Stephen et al., 1985).*

If the genus mean acute value is correctly calculated (see comments below re: inclusion of "greater than" values and non-North American species), it will likely result in a criterion that is not protective of coho salmon, due to their sensitivity to 6PPDQ. Thus, a more appropriate derivation for a Criterion Maximum Concentration (i.e., acute criterion) is the geometric mean of coho salmon effect concentrations and a safety factor of two, in accordance with the 1985 Guidelines. The resulting value would be 34 ng/L. This value is ~15% less than the lowest reported LC50 for coho salmon (41 ng/L; Lo et al., 2023).

Although standard Organisation for Economic Co-operation and Development (OECD) acute fish toxicity tests are 96h, the 24h tests presented in Table 55 of the Technical Support Document

[3] Brinkmann, M., Montgomery, D., Selinger, S., Miller, J. G. P., Stock, E., Alcaraz, A. J., Challis, J. K., Weber, L., Janz, D., Hecker, M., & Wiseman, S. 2022. Acute Toxicity of the Tire Rubber-Derived Chemical 6PPD-quinone to Four Fishes of Commercial, Cultural, and Ecological Importance. *Environmental Science & Technology Letters*, *9*(4), 333-338. https://doi.org/10.1021/acs.estlett.2c00050

are still substantially longer than the 1-hour averaging period, which is used in a criteria statement because "high concentrations of some materials can cause death in one to three hours" (Stephen et al., 1985). Therefore, 24h test results should be considered as reported, with no extrapolations. Furthermore, a 96h study using coho salmon fry indicated an LC50 of 70 ng/L (Lang et al., unpublished[4]), which is similar to the range of 24h values from Tian et al., 2022, Lo et al., 2023[5], and Greer et al., 2023[6]. USTMA recognizes that this (Lang et al.) data has not yet been peer-reviewed (SETAC 2023 poster). However, the lake trout toxicity data, which was used in the Criterion derivation, is also not yet peer reviewed. Further, USTMA notes that while the lake trout data is used to derive the Criterion, it is not cited in the Technical Support Document. USTMA recommends that the agency reevaluate its rationale for the extrapolation from 24h to 96h LC50 values by consulting the 1985 Guidelines and further review of the available evidence base.

3. THERE ARE ERRORS IN THE REPORTING AND APPLICATION OF THE STUDY ACCEPTABILITY REQUIREMENTS

The requirements for study acceptability are described on page 37-38 of Washington Ecology's Technical Support Document, which states that the requirements are based upon the 1985 Guidelines. Notably, two of these requirements specifically impact Ecology's evaluation of 6PPDQ. First, the Technical Support Document notes that acute values reported as "greater than" should not be used when they represent one of the four lowest genus mean acute values.

This requirement contradicts the 1985 Guidelines and directly influences the aquatic hazard data included or excluded in the evaluation. The 1985 Guidelines state, "if the tests were conducted properly, acute values reported as "greater than" values and those which are above the solubility of the test material should be used, because rejection of such acute values would unnecessarily lower the Final Acute Value by eliminating acute values for resistant species [emphasis add]." As indicated in Table 55 of the Ecology's Technical Support Document, the

[4] Lang, J., Cominassi, L., Chandler, A., Wong, L., Fangue, N., Mauduit, F., Connon, R., Segarra, A. 2023. When Rubber Meets the River: 6PPD-Quinone Acute and Sublethal Toxicity to San Francisco Bay-Delta Species of Conservation Concern. Poster presentation at SETAC North America, Louisville, KY.
[5] Lo, B. P., Marlatt, V. L., Liao, X., Reger, S., Gallilee, C., Ross, A. R. S., & Brown, T. M. 2023. Acute Toxicity of 6PPD-Quinone to Early Life Stage Juvenile Chinook (Oncorhynchus tshawytscha) and Coho (Oncorhynchus kisutch) Salmon. Environ Toxicol Chem, 42(4), 815-822. https://doi.org/10.1002/etc.5568
[6] Greer, JB; Dalsky, EM; Lane, RF; Hansen, JD. 2023. Establishing an in vitro model to assess the toxicity of 6PPDQ and other tire wear transformation products. Environ. Sci. Technol. Lett. 10(6):533-537. doi: 10.1021/acs.estlett.3c00196.

majority of the currently available evidence on toxicity of 6PPDQ in aquatic organisms was excluded on this basis. The inclusion of any of these values, in accordance with the 1985 Guidelines, would result in a higher final acute value.

USTMA requests that "greater than" i.e., non-definitive toxicity values (page 118; Table 55 of the Technical Support Document) be considered in the Criterion derivation for 6PPDQ in accordance with the 1985 Guidelines.

Second, Washington Ecology's Technical Support Document requires test species to be non-invasive North American species. This requirement is consistent with the 1985 Guidelines but was incorrectly applied by Washington Ecology. The 1985 Guidelines state, "data obtained with non-resident species in North America [emphasis added], or previously exposed organisms may be used to provide auxiliary information but should not be used in the derivation of criteria [emphasis added]." Table 55 and Figure 7 of Ecology's Technical Support Document indicate that two non-North American species were included in the derivation of the Criterion. White spotted char (*Salvelinus leucomaenis pluvius*) is not a North American species. Additionally, zebrafish (*Danio rerio*) are specifically listed as "nonresident" in Appendix 1 of the 1985 Guidelines and is not a North American species.

USTMA requests that Washington Ecology exclude S. leucomaenis pluvius and D. rerio from the Criterion derivation calculations in accordance with the study acceptability protocol provided in the Technical Support Document and recommended by the 1985 Guidelines.

4. ERRORS IN THE INTERPRETATION OF RESULTS FROM CITED STUDIES

USTMA identified several errors in the interpretation of the results of some cited studies that informed the derivation of the Criterion. These errors are outlined below.

a. In Ecology's Technical Support Document, page 118 states that "the only available data with definitive toxicity values included five fish species", and these five are listed in Table 55. However, it appears that the distribution (Figure 7) was generated using a sixth species (lake trout), which is not listed in Table 55.

   USTMA requests that Washington Ecology provide a reference for the lake trout data or remove the inclusion of this species and update Table 55 and Figure 7.

b. The LC50 value reported in Table 55 for *Oncorhynchus mykiss* (rainbow trout) by Di et al., 2022[7] is 1.66 µg/L; however, this value is for the S-enantiomer of 6PPDQ only. Di et al. report an LC50 value of 2.26 µg/L for 6PPDQ racemate. Since 6PPDQ is unlikely to ever occur as a discrete enantiomer in surface water, the LC50 value of 2.26 µg/L is more appropriate for derivation of a criterion value. Additionally, it is not appropriate to include the S-enantiomer only in Table 55, as all other toxicity tests in the table would have been conducted with racemate and, as Di et al. noted in the article, 6PPDQ effects may indeed be enantioselective.

   USTMA recommends that the value for the 6PPDQ *racemate be used rather than the enantiomer, as it is more environmentally relevant and consistent with the other* 6PPDQ *toxicity studies reported in Table 55.*

c. The LC50 value in Table 55 for *Oncorhynchus tshawytscha* (Chinook salmon) by Greer et al., 2023 is incorrect (LC50 = >80 µg/L). A definitive value of 82.1 µg/L is presented in Table S1 of Greer et al., 2023.

   USTMA requests that this definitive value be incorporated in the dataset and the 6PPDQ criterion be recalculated using an LC50 = 82.1 µg/L for Chinook salmon.

d. The LC50 for zebrafish (*Danio rerio*; Varshney et al., 2022[8]) is incorrectly reported as 139 µg/L in Table 55. The correct value is 132.92 µg/L. Additionally, zebrafish are a non-North American species, which is not noted in Table 55. It is, however, noted for other species in Table 55 as a basis for exclusion.

   Zebrafish are a non-native North American species. If Washington Ecology chooses to include zebrafish in the final criterion derivation, USTMA requests that the agency provide justification for including a non-North American species, and the correct LC50 value should be used.

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[7] Di, S., Liu, Z., Zhao, H., Li, Y., Qi, P., Wang, Z., Xu, H., Jin, Y., & Wang, X. 2022. Chiral perspective evaluations: Enantioselective hydrolysis of 6PPD and 6PPD-quinone in water and enantioselective toxicity to Gobiocypris rarus and Oncorhynchus mykiss. Environ Int, 166, 107374. https://doi.org/10.1016/j.envint.2022.107374

[8] Varshney, S., Gora, A. H., Siriyappagouder, P., Kiron, V., & Olsvik, P. A. 2022. "Toxicological effects of 6PPD and 6PPD quinone in zebrafish larvae." J Hazard Mater, 424(Pt C), 127623. https://doi.org/10.1016/j.jhazmat.2021.127623

e. The LC50 values in Table 55 for *Acipensar transmontanus* (white sturgeon, Brinkmann et al., 2022) and *Oryzias latipes* (medaka, Hiki et al., 2021[9]) are incorrect.

Although these two species and LC50 values were not used in criterion derivation, the LC50 for Acipensar transmontanus is >12.7 µg/L as reported in Brinkmann et al., 2022. The LC50 for Oryzias latipes is >34 µg/L as reported in Hiki et al., 2021. USTMA requests that Washington Ecology revise these values in Table 55 for technical accuracy.

5. CONCLUSION

USTMA's members are committed to collaborating with Washington Ecology and others to better understand 6PPD and 6PPDQ and fill knowledge gaps in existing research. USTMA supports the use of sound science and peer-reviewed data to inform regulatory actions. In summary, USTMA recommends that Washington Ecology follow the 1985 Guidelines for developing the Criterion or provide sufficient justification for when the agency deviates from those guidelines. If Washington Ecology is unable to satisfy these criteria, USTMA recommends that the agency reevaluate the proposed water quality criterion for 6PPDQ. Regardless of the agency's decision, the lack of data, rationale, and transparency made the proposed Criterion difficult to evaluate and reproduce. Once this additional information is available and corrections outlined in this letter have been made, USTMA requests that the agency repropose the 6PPDQ Criterion for additional public review. We are happy to answer any questions that Washington Ecology may have and look forward to working with the agency as this proposal moves forward.

Respectfully submitted,

Tracey Norberg
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Stephanie Schlea
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[9] Hiki, K., Asahina, K., Kato, K., Yamagishi, T., Omagari, R., Iwasaki, Y., Watanabe, H., & Yamamoto, H. 2021. "Acute Toxicity of a Tire Rubber-Derived Chemical, 6PPD Quinone, to Freshwater Fish and Crustacean Species." Environmental Science & Technology Letters, 8(9), 779-784. https://doi.org/10.1021/acs.estlett.1c00453