DRAFT

Literature Review: 6PPD-Quinone Technical Information Relevant to Caltrans Stormwater Management Program

CTSW-OT-24-425.XX.XX

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http://www.dot.ca.gov/hg/env/stormwater/index.htm

CALTRANS TECHNICAL REPORT DOCUMENTATION PAGE

1.	Report No.:	CTSW-TM-23-421.XX.XX
2.	Type of Report:	Technical Memorandum
3.	Report Phase and Edition:	Draft
4.	Title and Subtitle:	Literature Review: 6PPD-Quinone Technical Information Relevant to Caltrans Stormwater Management Program
5.	Report Date:	September 2024
6.	Copyright Owner(s):	California Department of Transportation
7.	Caltrans Project Coordinator(s):	Charlow Arzadon
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9.	Contract No.:	43A0425
10.	Task Order No.:	20
11.	Sponsoring Agency Name and Address:	California Department of Transportation Division of Environmental Analysis Stormwater Program MS-27 1120 N Street Sacramento, CA 95814 www.dot.ca.gov/hq/env/stormwater/index.htm
12.	Caltrans Functional Reviewer(s):	Sean Penders, Bhaskar Joshi, Charlow Arzadon, Alison Pommerenck
13.	Supplementary Notes:	
14.	External Reviewer(s):	N/A
15.	Key Words:	6PPD-Q, 6PPD-Quininone, stormwater, toxicity
16.	Distribution Statement:	No restrictions. This document is available to the public upon request.
17.	No. of pages:	25
18.	Abstract:	This technical memorandum provides an overview of scientific literature of 6PPD-Q sources, transport, treatability, toxicity, and related information needed to guide monitoring design and evaluate stormwater treatment design guidance updates.

ACKNOWLEDGEMENTS

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Executive Summary

This literature review summarizes recent scientific publications on 6PPD-Quinone (6PPD-Q), an ozone breakdown product of the tire additive 6PPD that is lethal to aquatic life in low concentrations. The review presents findings from peer reviewed sciences relevant to 6PPD-Q risks to aquatic life and supports evaluation of potential treatment Best Management Practice (TBMP) strategies to minimize those risks. The review findings, organized by topic, lead to a shorter list of articles that will be referenced in a white paper on 6PPD-Q. The white paper is intended to assist Caltrans managers and partner agencies navigate stormwater management decisions in response to emerging concerns about 6PPD-Q.

Over 200 citations provided by Google Scholar were reviewed, findings summarized, and uncertainties and data gaps identified by topic. An initial review culled the list of 200 citations down to 129 citations that are summarized by topic below. Parentheses after each topic below indicates the number of citations reviewed.

Runoff and watershed assessment (12)

Data from seven of the twelve citations in this topic provide important context for Caltrans to interpret monitoring data. Expected concentrations of 6PPD-Q in Caltrans stormwater discharges, stormwater discharges from other sources, and in the receiving waterbodies guides monitoring for ecosystem vulnerability assessment.

For the white paper, two tables prepared from the seven citations will summarize reliable sources for 6PPD-Q concentrations in roadway runoff, other types of urban runoff, and receiving waters. A summary of errors in and corrections to the original findings of 6PPD-Q lethality and environmental concentrations helps understand the reliability of available data.

Airborne, dust, soils, and sediments (14)

Seven of the fourteen citations inform a table of 6PPD-Q concentrations in dusts, soils, and sediments. This helps Caltrans interpret monitoring data involving suspended and bedded sediments. All fourteen of the peer-reviewed citations represent urban and industrialized areas in China, and so may not reflect conditions in California. The unique geographic context of 6PPD-Q data in dusts, soils and sediments source will be noted when using the information in the white paper.

Leaching and adsorption (5)

These five citations help understand factors affecting partitioning of 6PPD-Q between solid and dissolved phases. These findings are useful to developing a conceptual model for 6PPD-Q fate, transport, and treatability to reduce 6PPD-Q risks to aquatic life. A key uncertainty is the toxicity of particle-associated 6PPD-Q vs dissolved 6PPD-Q.

Decomposition (4)

Unlike “forever” chemicals such as polychlorinated biphenyls (PCBs) and per- and poly-fluoralkyl substances (PFAS), 6PPD-Q rapidly degrades (in hours to weeks), especially in sunlight. Degradation of 6PPD-Q is important to treatability evaluation and risk assessment.

Aquatic life toxicity (26)

The low effect levels of 6PPD-Q on aquatic life, particularly on coho salmon, in comparison to environmental concentrations drive interest in 6PPD-Q monitoring and treatment effectiveness evaluations. Findings from the toxicity studies reviewed will be compiled into a single table for the white paper to provide context for interpreting monitoring results.

Chemical / physical evaluations (6)

Chemical / physical evaluation studies provide additional information on toxic effects of tire and road wear particles (TRWP) resulting from elevated oxidative potential and generation of environmentally persistent free radicals (EPFR); this is potentially significant to Caltrans. Even after product substitution potentially solves the problem of unusually potent and specific 6PPD-Q toxicity, concerns may persist over TRWP sources of EPFR.

Human / mammalian toxicity (12)

Information on human health effects of 6PPD-Q helps Caltrans prepare to address public concerns that may follow discovery of new pollutants of concern. The citations on this topic are outside the scope of the white paper, but retained in the Zotero database in case future interests make them topical.

Worm / microbial toxicity (8)

Citations in this topic support ecological risk assessments, which can guide development of water quality standards and other planning processes. The citations on this topic are outside the scope of the white paper, but retained in the Zotero database in case future interests make them topical.

Product Screening and Substitution (2)

Experience with leaded gasoline and copper brakes shows that product substitution is an effective long-term solution to vehicle-related pollutants. In March 2024, a global tire industry consortium released an alternatives analysis report for compounds that can replace 6PPD. The website and report provide an overview of the assessment process, regulatory framework, timeline, and approach. The time needed to develop and implement substitutes and replacements in the United States and global tire inventory is uncertain, but likely years.

Treatment (8)

Given the anticipated timeframe for product substitution, questions will likely persist regarding the progress of stormwater treatment in high-risk areas for coho salmon mortality. The full range of Caltrans-approved treatment best management practices (TBMPs) needs to be evaluated. Lessons learned from the literature review include:

- One of the citations shows potential benefits of permeable pavement. Full depth permeable pavement has limited application in California highways (e.g., shoulders but not lanes).
- Two of three citations describing bioretention and natural treatment rely on lab studies, while the third relies on a small-scale system.
- Detention basins pose a risk of increasing 6PPD-Q due to leaching from TRWP detained and conversion of 6PPD to 6PPD-Q within the basin.

Effectiveness of Caltrans TBMPs and meaningful data from other state transportation departments would address important information gaps as more information becomes available.

Chemical analysis (17)

To properly quantify 6PPD-Q in stormwater, Caltrans requires a USEPA-approved analytical test method. In December 2023, USEPA released Method 1634, a draft method for 6PPD-Q analysis, potentially addressing Caltrans' interest. The remainder of the citations underscore that 6PPD-Q analysis is new, and analytical procedures have been evolving over the past four years.

Reviews (13)

This literature review targets Caltrans' interests for managing 6PPD-Q in stormwater. The thirteen reviews available in journals are more broadly focused. They will be referenced in the white paper for completeness, and to acknowledge the efforts of other reviewers, but they are not anticipated to be relevant to Caltrans.

Government programs (2)

Activities by other state agencies help Caltrans and partner agencies set expectations for a collaborative approach to address 6PPD-Q. A report by Washington Department of Ecology documents their response to their State Legislature's direction:

- An assessment strategies workgroup provided initial guidance that vulnerable ecological areas include waterbodies with sensitive species that are potentially impacted by roadway stormwater discharges.
- A management strategies workgroup developed BMP selection criteria.

The report included a literature review, documentation of the partnering process and outcomes, and technical summaries of green stormwater infrastructure, comparable to the proposed scope of the Caltrans white paper.

Washington Department of Transportation (WSDOT) provided a memorandum documenting $500 million for enhanced stormwater treatment retrofits. The memorandum showed a map and table showing how funds could potentially be applied to vulnerable areas identified by the assessment workgroup.

Summary

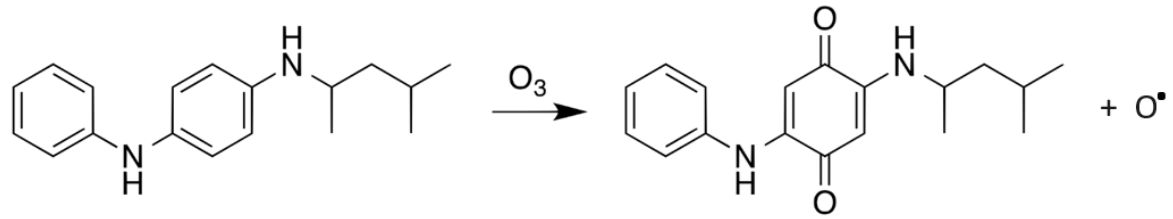
This literature review distilled more than 200 citations down to 127 and organized the top 80 relevant citations to a white paper which will evaluate risks of and management strategies for 6PPD-Q in stormwater. About thirty of those citations will be discussed at a high level as topics (i.e., Chemical Analysis and Reviews) in the white paper. The remaining 50 citations will support tables and conceptual figures to help evaluate and explain how Caltrans' program of stormwater monitoring and TBMP implementation can be refined to address 6PPD-Q. License copies will be procured for any of the 50 core citations used in the white paper that are not public access (many are). Many of the 50 core citations to be used in the white paper are publicly accessible, Caltrans will hold the licensed copy for those that are not.

Background

The California Department of Transportation (Caltrans) implements a Stormwater Management Plan (SWMP) to prevent pollution of stormwater discharges from Caltrans facilities. The SWMP directs an ongoing water quality monitoring program to characterize pollutants in Caltrans stormwater discharges and evaluates the effectiveness of treatment Best Management Practices (TBMPs) at reducing stormwater pollutants. The SWMP is updated from time to time as requirements from the State Water Resources Control Board change, new pollutant concerns arise and new TBMPs are developed.

In 2022, academic researchers revealed a new pollutant of concern was originating from tire and road wear particles (TRWP). The compound known as 6PPD, an ozone scavenger that is sacrificially oxidized to protect tire rubber from ozone oxidation, converts to 6PPD-quinone (6PPD-Q) by reaction with ozone (Figure 1). Leaching of 6PPD-Q from TRWP into stormwater appears to be a root cause of stormwater toxicity to aquatic life, particularly coho salmon, in small urban streams of the Pacific Northwest[1] (superscript refers to citation number in bibliography at end of this memorandum)

Caltrans responded to this information by including 6PPD-Q in ongoing characterization and effectiveness monitoring programs beginning January 2023. Caltrans also commissioned a literature review (this document) and white paper on 6PPD-Q to compile and synthesize existing scientific knowledge of 6PPD-Q sources, fate, transport, toxic effects, and treatability. This literature review summarizes recent scientific publications on 6PPD-Q, presents findings relevant to 6PPD-Q risks to aquatic life, and supports evaluation of potential TBMP strategies to minimize those risks. The findings are organized by topic and lead to a shorter list of articles that will be referenced in the white paper.



N-*(1,3-dimethylbutyl)*-N'-*phenyl*-p-*phenylenediamine (6PPD)*

N-*(1,3-dimethylbutyl)*-N'-*phenyl*-p-*phenylenediamine quinone (6PPD-Q) plus radical oxygen (O•)*

Figure 1. Conversion of 6PPD to 6PPD-Q by Ozone (O_3)*

* The environmental significance of ozonolysis generating radical oxygen - oxygen with an unpaired electron (symbolized as O•) - is discussed below in the "Chemical / physical evaluations" review topic.

Approach

A search of Google Scholar using the term “6PPD” yielded more than 5,000 results, whereas using terms “6PPD-Q” or “6PPD-Quinone” each yielded about 200 results. Narrowing the search window to specific years shows the rapid development of knowledge about 6PPD-Q (Table 1). Prior to 2021, articles on 6PPD focused solely on the materials science aspects, e.g., its antioxidant properties, or its capacity to migrate from the interior of rubber tires to exterior surfaces. In January 2021, researchers from the University of Washington published findings that they had identified 6PPD-Q as the cause of recurrent coho salmon mortality in West Coast urban streams[1]. Since then, the number of published articles, reviews, dissertations, and presentations related to 6PPD-Q has grown to over 200.

Table 1. Number of 6PPD-Q citation results from Google Scholar

Year(s)	# Citations
1960 - 2020	0
2021	4
2021-2022	36
2021-2023	120
2021-2024	205

An initial review culled the list of more than 200 citations down to 143 that are relevant to Caltrans and partnering agencies in the evaluation of 6PPD-Q risks and potential stormwater management strategies. Of those, 119 are peer-reviewed articles published in established scientific journals (Table 2). The remaining citations are government reports and articles undergoing peer review available through pre-print outlets such as ResearchGate, BioRxiv (pronounced “bio-archive”), ChemRxiv, SSRN, and Google Books. About two-thirds of the peer-reviewed articles appear in the top 5 journals ranked in Table 2 by number of 6PPD-Q articles published.

Citations were archived in the open-source bibliography database application Zotero (Zotero.org). Article PDF files were excluded from the database in respect of copyright protection. Most of the database citations include digital object identifier (DOI) links, making the abstract and publication sources readily available. Many of the articles are open access, meaning that purchase of a license is not needed to read the full article. Articles of interest that are not open access will be purchased on behalf of and provided to Caltrans for fair use as a government agency.

Table 2. Number of 6PPD-Q citations by journal

Journal	# Citations
Environmental Science and Technology	23
Science of The Total Environment	20
Journal of Hazardous Materials	13
Environmental Pollution	11
Environmental Science and Technology Letters	11
Chemosphere	6
Environment international	3
Environmental Research	2
ES&T Water	2
Sustainability	2
Trends in Analytical Chemistry	2
ACS Environmental Au	1
Analytical Chemistry	1
Analytical Science Advances	1
Archives of Environmental Contamination and Toxicology	1
Bulletin of Environmental Contamination and Toxicology	1
Comparative Biochemistry and Physiology Part C: Toxicology & Pharmacology	1
Ecocycles	1
Environment & Health	1
Environmental Health Perspectives	1
Environmental Monitoring and Assessment	1
Environmental Science and Pollution Research	1
Environmental Toxicology and Chemistry	1
Frontiers in Environmental Science	1
Frontiers in Nutrition	1
International Journal of Environmental Research and Public Health	1
Journal of Cleaner Production	1
Marine Pollution Bulletin	1
Process Safety and Environmental Protection	1
Progress in Rubber and Plastics Technology	1
Respiratory Medicine	1
Science	1
Sustainable Chemistry and Engineering	1
Talanta	1
Water Research	1
Total	119

Findings

Citations covered thirteen topics. Parentheses after each topic title below indicates the number of citations reviewed:

- Runoff and watershed assessment (12)
- Airborne, dust, soils, and sediments (14)
- Leaching and adsorption (5)
- Decomposition (4)
- Aquatic life toxicity (26)
- Chemical / physical evaluations (6)
- Human / mammalian toxicity (12)
- Worm / microbial toxicity (8)
- Product Screening and Substitution (2)
- Treatment (8)
- Chemical analysis (17)
- Reviews (13)
- Government programs (2)

For reader convenience, the bibliography of this literature review is organized by topic. The reviews by topic below focus on information relevant to Caltrans and partner agencies in the determination of 6PPD-Q risks from stormwater, and appropriate management strategies. Each topic briefly describes Caltrans' interest in relation to managing stormwater to minimize 6PPD-Q risks to aquatic life and identifies the citations relevant to be referenced in the white paper. Key findings are described, along with uncertainties and data gaps. Where citations were reviewed but are not anticipated to be used in the white paper, a brief rationale is provided.

Runoff and watershed assessment (13 citations)

Caltrans' interest

Data from eight of the thirteen citations in this topic help answer a critical question for Caltrans: what are expected concentrations of 6PPD-Q in roadway runoff (before treatment), other types of urban runoff, and streams impacted by roadway and urban runoff?

Key Findings

University of Washington Researchers first published their findings about 6PPD-Q as a cause of coho salmon mortality in *Science*[1] in 2021. A year later they published an *errata* to that article and a follow-up revised assessment of toxicity and environmental concentrations[2]. The *errata*

stated that the original publication overestimated environmental concentrations by 15-fold because original assessments used in-house 6PPD-Q calibration standards prepared by ozonolysis of 6PPD (Figure 1). Noting that response factors were 15-fold higher using commercial standards, data from the original publication were divided by 15-fold to provide a corrected estimate of environmental concentrations. Re-running the samples was not an option, as the extracts were already archived, and no more extract remained available for follow-up testing. Toxicity tests were re-run using commercial standards, resulting in the toxic thresholds being reduced by 8-fold.

In the errata to the original publication[1], the authors maintain that although absolute concentrations and thresholds shift lower when using the updated calibration, the relative relationship between 6PPD-Q environmental concentration and lethal thresholds are unchanged and the conclusions and implications of the 2021 *Science* paper[1] are otherwise not affected. The literature review presented herein supports the overall conclusion that 6PPD-Q environmental concentrations can exceed lethal thresholds as described by Tian et al (2022)[2].

However, the differential reductions in 6PPD-Q toxic thresholds versus 6PPD-Q found in stormwater and surface water concentrations does shift the perception of impairment based on a small data set, at least in the case of San Francisco Bay Area samples included in the 2021 and 2022 publications. In the 2021 publication[1], five out of nine Bay Area creeks sampled were non-detect, and the remaining four exceeded the toxic thresholds proposed at the time. In the 2022 revised assessment[2], only two of the four detected concentrations exceeded lethal thresholds, with the five non-detect creeks still non-detect.

The *errata* impacts the reliability of early publications on 6PPD-Q concentrations in stormwater and surface waters. Of the twelve citations screened for this subtopic, seven are considered reliable because the studies were performed after the 2022 revised assessment and relied on commercial standards[2–8]. Tables summarizing 6PPD-Q concentrations in stormwater and surface waters will be prepared for the white paper based on those seven reliable literature sources. This provides Caltrans managers and technical staff with context for interpretating stormwater and surface water monitoring results.

A reconnaissance survey of U.S. surface waters performed by the United States Geological Survey (USGS) showed surface water concentrations well below toxic effect levels in nine states surveyed[9]. Median 6PPD-Q concentrations in surface waters from Colorado, Georgia, Kansas, Michigan, Minnesota, North Carolina, Oregon and Texas were less than 20 ng/L, well below 95 ng/L lethal effect levels for coho salmon.

Uncertainties and Data Gaps

The white paper will provide a more detailed assessment of the creeks and stormwater sites monitored in California by Tian et al[1,2], including watershed settings and relevant roadways. Stormwater samples from the literature over-represent a limited geography and roadway type, i.e., heavily traveled multi-lane roadways in Hong Kong, Seattle, and Los Angeles. Monitoring 6PPD-Q in stormwater discharges from rural roads would address an important data gap for Caltrans because of the abundance of smaller two-lane state highways in California coho salmon watersheds.

None of the roadway runoff data presented in the literature are accompanied by suspended

sediment concentration (SSC) data in the publications or supplemental information. Evaluating whether 6PPD-Q is correlated with SSC in roadway runoff is helpful to predict whether TBMPs that remove particles will be effective. Stream monitoring data in the Helm citation[8] are paired with total suspended solids (TSS) measurements. Correlation between 6PPD-Q and TSS is poor in the overall data set (R^2 = 0.06), which is not surprising for streams, where roadway runoff is not the sole contributor of suspended particles. Correlations may be higher when 6PPD-Q measurements from roadway runoff are paired with concurrent SSC measurements.

Citations reviewed but not directly used in the white paper

Studies by Johannessen relied on in-house 6PPD-Q standards and are therefore not reliable for 6PPD-Q concentration data[10,11]. A study by Kang provided a correlation analysis based on detections for non-target screening but does not provide quantitative 6PPD-q data[12]. A citation by Peter[13] is included because the study provided the archival samples for the Tian study that revealed the lethality of 6PPD-Q to coho salmon, but the paper itself has no 6PPD-Q data. The original *Science* publication by Tian[1] will be included for completeness and is considered reliable because of the *errata*, but for referencing the revised assessment will be used to avoid confusion. The Johannessen[10,11], Tian[1] and Peter[13] studies will be briefly mentioned in the white paper for context, as described herein. Documenting data that are unreliable and have or have not been corrected is an intended outcome of this review.

Airborne, dust, soils, and sediments (14 citations)

Caltrans' interest

Tire and road-wear particles represent the primary source of 6PPD-Q in roadway runoff. Roadside dusts and soils represent a storage compartment and a transport pathway of TRWP. Data on 6PPD-Q concentrations in dusts from parking areas and tunnels, roadside dusts, and roadside soils help characterize this compartment.

Key findings

Seven citations provide data on the particulate concentrations of 6PPD-6 found in soils, estuarine sediments, roadside dust, and indoor dust[14–20]. Data from these citations will be used to develop a summary table for the white paper. A unique study of 6PPD-Q concentrations in fresh atmospheric snow provides insights into concentrations that 6PPD-Q can reach before rain even reaches paved surfaces[21].

Uncertainties and data gaps

The studies of 6PPD-Q in estuarine sediments, soils, roadside and indoor dust were all in developed, industrialized areas of China. Some of the studies targeted indoor and outdoor areas near e-waste recycling facilities. As such, the solids concentrations may not represent conditions near California roadways.

Citations reviewed but not used

The remaining citations in this topic were more focused on inhalation exposure risks to humans[22–26], and consequently only provided data on airborne 6PPD-Q concentrations (i.e., in units of pg/m^3), or lacked 6PPD-Q data[27]. Findings from those citations may be relevant to Caltrans in terms of worker and / or public exposure risk from TRWP but are outside the scope of this white paper.

Table 4. 6PPD-Q Concentrations in dust and soils

Location	Soil or Dust	% Urbanized	6PPD-Q (ng/g)* (median) **MAX** *single*	Citations
Tokyo, Japan	Road dust	High	(809) **1238**	Hiki et al. (2022) [28]
Dalian, China	Urban tunnel road	High	(433) **1906**	Zhang et al. (2024) [18]
Hong Kong, China	Roadside dust	High	(234) **936**	Cao et al. (2022) [4]
Dalian, China	Urban trunk road	High	(158) **874**	Zhang et al. (2024)[18]
Guangdong, China	Indoor parking lot dust	High	(154) **2369**	Deng et al. (2022)[15]
Guangdong, China	Road dust	High	(122) **509**	Deng et al. (2022) [15]
China	Vehicle dust	NA	(81) **146**	Huang et al. 2021[14]
55 Major cities of China	Urban truck road dust	Varies	(49) **349**	Zhang et al. (2024) [18]
China	Parking lot dust	NA	(42) **277**	Huang et al. (2021) [14]
China	Road dust	NA	(32) **88**	Huang et al. 2021[14]
Guiyu Town, Shantou City, Guangdong Province China	Traditional e-waste recycling zones	NA	46.4	Zhang et al. (2024) [19]
Guiyu Town, Shantou City, Guangdong Province China	Emerging e-waste recycling park	NA	15.4	Zhang et al. (2024) [19]
South China Sea	Sediment from Urban River in the Pearl River Delta	NA	(9.0) **18**	Zeng et al. 2023 [16]
South China Sea	Sediment from the Pearl River Estuary	NA	(2.0) **4.9**	Zeng et al. 2023 [16]
South China Sea	Sediment from coast of the South China Sea	NA	(1.3) **3.0**	Zeng et al. 2023 [16]
South China Sea	Sediment from deep-sea regions of the South China Sea	NA	(2.7) **3.0**	Zeng et al. 2023 [16]

Leaching and adsorption (5 citations)

Caltrans' interest

Understanding factors affecting partitioning of 6PPD-Q between solid and dissolved phases is critical to evaluating treatment efficacy, enhancing treatment approaches, and implementing source control.

Key findings

A study of roadside dusts provided 6PPD-Q concentrations in dust and determined a 6PPD-Q water-octanol partition coefficient (KOW)[28]. A leaching study showed the effects of ageing, including effects of temperature, sunlight, and mechanical abrasion on the leachability of numerous tire additives and transformation products, including 6PPD-Q[29]. A study of 6PPD-Q in rubberized asphalt reported that concentrations of 6PPD-Q leached from loose and compacted rubberized asphalt were below the concentration lethal to 50 percent of organisms (LC-50) for

coho salmon[30].

Leaching of tire additives has shown to increase with decreasing particle size[31]. A study that separated effects of leaching and biotransformation noted that 6PPD-Q particles steadily increased when tire wear particles were present, but decreased in leachate that had been separated from tire wear particles[32]. These findings are useful to developing a conceptual model for 6PPD-Q fate, transport, and treatability to reduce 6PPD-Q risks to aquatic life.

Uncertainties and data gaps

Understanding factors that increase and decrease 6PPD-Q concentrations is an evolving area of study. A key uncertainty is the toxicity of particle-associated 6PPD-Q vs dissolved 6PPD-Q. Analogous to trace metals, it is possible that dissolved 6PPD-Q is more toxic than particle-associated forms. Thus, detailed understanding of dissolved vs particulate 6PPD-Q in the environment and factors that promote leaching 6PPD-Q requires more information from studies in this topic area.

The study of rubberized asphalt[30] was a bench-scale laboratory study funded by Granite Construction Company (who has a potential interest in continued use of rubberized asphalt). Real-world investigations on operating roadways will help verify or refine the study findings.

Decomposition (4 citations)

Caltrans' interest

Unlike "forever" chemicals such as polychlorinated biphenyls (PCBs) and per- and poly-fluoralkyl substances (PFAS), 6PPD-Q rapidly degrades (in hours to weeks), especially in sunlight. Degradation of 6PPD-Q is important to treatability evaluation and risk assessment.

Key findings

A study of aging under natural and artificial light conditions determined the half-time of 6PPD-Q degradation was within a month under study conditions[33]. Another study found a photochemical degradation half-time of 17.5 hours, and probed into more detailed mechanisms of direct versus indirect photodegradation[34]. Direct photodegradation means the light itself degrades 6PPD-Q, whereas indirect means light creates reactive compounds that degrade 6PPD-Q. Intense UV irradiation with an artificial lamp removed 94% of 6PPD-Q within 40 minutes[35]. Sunlight can also transform the parent compound 6PPD into 6PPD-Q[36].

Uncertainties and data gaps

These four articles provide limited but important insights into 6PPD-Q decomposition pathways. The range of decomposition rates, i.e., a 17.5-hour vs. a one-month half-time, shows that more detailed understanding of factors affecting 6PPD-Q decomposition would be beneficial.

Aquatic life toxicity (28 citations)

Caltrans' interest

The effects of 6PPD-Q on aquatic life at low concentrations relative to environmental concentrations drives interest in 6PPD-Q monitoring and treatment effectiveness evaluations.

Key findings

The Tian re-assessment of 6PPD-Q toxicity using commercial standards established an LC-50

of 95 ng/L for coho salmon[2]. A different study by Lo[37] using methods similar to Tian[2] found an LC-50 of 41 ng/L for coho salmon. The lower LC-50 was attributed to lower subject fish body masses, lower water temperatures, and other study condition differences. The Lo study is also an example of erroneous comparison of the very low LC-50 to environmental 6PPD-Q concentrations determined by Johannessen[10,11] that are likely biased high due to use of in-house 6PPD-Q standards, as discussed above under the Runoff and Watershed Assessment topic.

Additional acute toxicity data are available for fathead minnow[38], larval zebrafish[39], and adult zebrafish[40]. 6PPD-Q toxicity was lethally toxic to white spotted char but not to two other salmonid species[41]. Findings from the above toxicity studies[2,37–39,41,42] are presented compiled in a single table for the white paper to provide context for interpreting monitoring results Table 3.

Toxicity studies inform water quality standards. Although the United States Environmental Protection Agency (USEPA) has not established enforceable numeric water quality criteria for 6PPD-Q at this time, the agency has released non-regulatory screening level guidance[43]. The screening value proposed by USEPA is 11 ng/L. The screening value was established using the studies cited in Table 3.

Table 3. Lethal concentrations of 6PPD-Q to aquatic organisms.

Fish Species	LC50 (ng/L)	Time (hr)	Citations
All – USEPA Screening Guideline = 11 ng/L[a]	Not applicable	1 hr	USEPA (2024) [43]
Oncorhynchus kisutch (juvenile coho salmon)	41	24	Lo et al (2023) [37]
Oncorhynchus kisutch (adult coho salmon)[a]	95	24	Tian et al (2022) [2]
Oncorhynchus kisutch (coho salmon)	80	24	Greer et al (2023) [51]
Salvelinus fontinalis (brook trout fry)	200	24	Philibert et al. (2024) [47]
Salvelinus fontinalis (brook trout fingerlings)	500	24	Philibert et al. (2024) [47]
Salvelinus namaycush (lake trout)	500	96	Roberts et al. (2024)[49]
Salvelinus leucomaenis Pluvius (white-spotted char)	510	24	Hiki and Yamamoto (2022) [41]
Salvelinus fontinalis (brook trout)	590	24	Brinkmann et al (2022) [52]
Oncorhynchus mykiss (rainbow trout)[a]	1,000	72	Brinkmann et al (2022) [52]
Oncorhynchus mykiss (rainbow trout)	2,260	96	Di et al. (2022) [44]
Pimephales promelas (adult fathead minnows)	>9400[b]	96	Anderson-Bain et al (2023) [38]
Salvelinus curilus (Southern Asian dolly varden)	>10,000[b]	24	Hiki and Yamamoto (2022) [41]
Oncorhynchus masou masou (masu salmon)	>10,000[b]	24	Hiki and Yamamoto (2022) [41]
Salvelinus alpinus (artic char)	>12,700[b]	24	Brinkmann et al (2022) [52]
Acipenser transmontanus (white sturgeon)	>12,700[b]	24	Brinkmann et al (2022) [52]
Pimephales promelas (fathead minnow embryo)	>39,970[b]	168	Anderson-Bain et al (2023) [38]
Oncorhynchus tshawytscha (chinook salmon)	>80,000[b]	24	Greer et al. (2023) [51]

a. Shaded cells indicate levels used for comparison to water concentrations in this report.

b. Value is greater than the highest concentration tested.

Uncertainties and data gaps

As noted above under leachability and adsorption, the relative toxicities of dissolved vs. particulate forms of 6PPD-Q are not well defined. Toxicity studies generally use pure product to set exposure concentrations, whereas environmental samples represent a mixture of dissolved 6PPD-Q leached from tire particles and 6PPD-Q present in tire particles.

Citations reviewed but not used

Twenty citations have been retained that may not be used in the white paper[51–66]. The retained studies are valuable in that they provide one or more of the following:

- Details of toxic effect mechanisms,
- Early evidence of tire particle leachate toxicity to coho salmon, and
- Information on uptake and biotransformation.

For example, one such paper provides interesting insights into how toxicity varies with structure across a variety of different PPD-quinones[66]. The study is not yet peer-reviewed and is more

relevant to product substitution efforts than the focus of the white paper on 6PPD-Q concentrations, effect levels, and management strategies. All the aquatic toxicity citations not used are retained in the Zotero database in case future interests make them topical.

Chemical / physical evaluations (3 citations)

Caltrans' interest

Chemical / physical evaluation studies provide additional information on toxic effects of TRWP rather than 6PPD-Q specifically. Although the potent toxic effects of 6PPD-Q are of primary concern, it is helpful for Caltrans to be aware of studies showing more general impacts of TRWP.

Key findings

Aging TRWP can increase the leaching concentration of 6PPD-Q[69]. The toxic effects of TRWP to aquatic biofilms is attributed to environmentally persistent free radicals (EPFR), which diminish in potency with aging[70]. Thus, aging TRWP can have both negative and positive effects on toxicity. PPD-quinones in general (i.e., quinones of 6PPD and other PPD forms) induce a strong oxidative potential in urban fine particulate matter (<2.5 microns) [71]. This finding is more directly related to human health than aquatic toxicity. All three of these citations will be included in the white paper as general background.

Ozonolysis, oxidation with ozone, likely explains generation of EPFR and increased oxidative potential in TRWP (Figure 1). Ozone, three oxygen atoms bonded in a triangular shaped molecule, tends to form free radicals in solution during ozonolysis[72–74]. Free radicals, atoms or molecules with an unpaired electron, are highly reactive because unpaired electrons are unstable, i.e., they "want" to form pairs to become stable. Radical reactions can damage cell tissue and fragment DNA.

Uncertainties and data gaps

This is an evolving area of study. The concept that TRWP can cause elevated oxidative potential and generate EPFR is significant to Caltrans. Even after product substitution solves the problem of unusually potent and specific 6PPD-Q toxicity, concerns may persist over TRWP sources of EPFR.

Human / mammalian toxicity (12 citations)

Caltrans' interest

Information on human health effects of 6PPD-Q helps Caltrans prepare to address public concerns that inevitably follow discovery of new pollutants of concern.

Key findings

The citations on this topic are outside the scope of the white paper, but retained in the Zotero database in case future interests make them topical[75–86] .

Worm / microbial toxicity (8 citations)

Caltrans' interest

Citations in this topic support ecological risk assessments, which can guide development of water quality standards and provide Caltrans with information that can guide input on proposed regulatory thresholds.

Key findings

The citations in this topic are outside the scope of the white paper, but retained in the Zotero database in case future interests make them topical[87–94].

Product Screening and Substitution (2 citations)

Caltrans' interest

Experience with leaded gasoline and copper brakes shows that product substitution and / or product improvement is an effective long-term solution to vehicle-related pollutants. Product substitution would mean alternative ozone-reactive compounds with non-toxic breakdown products. Product improvement would mean continued use of 6PPD in a manner that does not release 6PPD-Q at problematic levels.

Key findings

Three citations provide insights into the path forward via product substitution. In March 2024, a global tire industry consortium released an alternatives analysis report for compounds that can replace 6PPD-Q in tires while maintaining the protective properties needed for safe driving[95]. The website provides an overview of the assessment process, regulatory framework, timeline, and approach. Links to a fact sheet and the March 2024 Alternatives Analysis report is also provided on the website. The white paper will provide additional details from the study report.

The work of the global consortium is driven by the California Department of Toxics Substances Control's (DTSC) Safer Consumer Products program[96]. On October 1, 2023, DTSC finalized a regulation to list 6PPD as a new Priority Product. This action required manufacturers using 6PPD to submit an alternatives analysis by March 29, 2024. The above cited report by the global tire manufacturers consortium was submitted in response to that requirement.

A peer-reviewed journal article evaluated mechanisms of 6PPD-Q toxicity to coho salmon and design 129 derivatives of 6PPD, three of which showed promising antioxidant properties[97]. One of the three, designated 6PPD-106, showed lower toxicity to coho salmon compared to 6PPD-Q. The citation provides a specific example of peer-reviewed research in pursuit of product substitution alternatives.

Uncertainties and data gaps

The time to test, select, approve, and mass produce tires without 6PPD to replace the United States and global tire inventory is uncertain, but likely years.

Treatment (8 citations)

Caltrans' interest

Given the long timeframe for product substitution or improvement, Caltrans will be under pressure to show increased progress on stormwater treatment, particularly in high-risk areas for coho salmon mortality.

Key Findings

A study of 6PPD-Q in a Hong Kong municipal wastewater treatment plant showed reductions from the median influent concentration of 53 ng/L down to 3.4 ng/L [98]. The resulting biosolids had a median 6PPD-Q concentration of 6.4 ng/g.

Simulation studies in a bioretention cell suggest that bioretention can be effective in removing 6PPD-Q from stormwater[99]. A field study in a small system (22 m^2 treating an area of 694 m^2 with an underdrain) showed ten-fold reduction of 6PPD-Q from stormwater[100]. A pre-print of a study (not yet peer-reviewed) claims efficacy of floating treatment wetland based on lab studies using 12-gallon samples[101].

Treatment of tunnel wash water in a settling basin showed minimal 6PPD-Q removal[102]. The greatest removal (22%) occurred at seven days, after which removal diminished to near zero. The authors suggest that leaching from particles settled in the basin may explain poor removal effectiveness for 6PPD-Q. In addition to leaching, continued production of 6PPD-Q from 6PPD could explain the poor performance of the settling basin. Another study showed that flooded, anaerobic soils enhance production of 6PPD-Q in tire wear particles[103]. Chemically reducing, low oxygen conditions may be a risk factor for increasing, rather than decreasing 6PPD-Q in stormwater treatment systems and natural settings.

Permeable pavement was shown to be effective at removing tire wear particles, concurrently reducing 6PPD-Q in treated stormwater[104]. Another recent study showed that porous overlays and full-depth porous asphalt can reduce concentrations of 6PPD-Q and other pollutants for many years after installation[105].

Uncertainties and Data Gaps

The bioretention and natural treatment citations[99–101] rely on lab studies of very small scale systems. Treating large volumes of roadway runoff often requires more land than is available, and so the full range of Caltrans-approved TBMPs needs to be evaluated.

Chemical analysis (17 citations)

Caltrans' interest in the topic

To properly quantify 6PPD-Q in stormwater, Caltrans requires a USEPA approved analytical test method to pursue stable, reliable and commercially available test methods.

Key Findings

In December 2023, USEPA released Method 1634, a draft method for 6PPD-Q analysis[106]. The method documents the liquid chromatography / mass spectrometry approach to analysis and provides guidance for laboratory quality control.

The remaining citations on this topic show the development of analytical capabilities[107–122]. They will be cited in the white paper as a group for completeness but not described in great detail.

Uncertainties and Data Gaps

The EPA-approved method 1634 is marked draft, and therefore may change.

Reviews (13 citations)

Caltrans' interest

A literature review provides Caltrans with a ready reference to scientific knowledge on topics relevant to 6PPD-Q monitoring and management strategies.

Key Findings

This literature review is written targeting Caltrans' interests. The thirteen reviews available in

journals are more broadly focused[123–135]. They will be referenced in the white paper for completeness, and to acknowledge the efforts of other reviewers, but they are not anticipated to be directly relevant to Caltrans. The bibliographies from those other reviews can help verify that all relevant citations have been identified in this one.

Other State Government Programs (3 citations)

Caltrans' interest

Activities by other state agencies help Caltrans and partner agencies set expectations for a collaborative approach to address 6PPD-Q.

Key Findings

Michigan Surface Water Survey

A study by Michigan State Department of Ecology documented non-detects in most surface waters surveyed[136]. The study focused on rural Michigan roadways paved using crumb rubber asphalt. The study will be cited in the summary table of 6PPD-Q concentrations in water.

Washington Ecology Partnering Report

A report by Washington State Department of Ecology (Washington Ecology)[137] documents their response to the State Legislature's direction established through Senate Bill 5092:

> *$523,000 of the model toxics control operating account—state appropriation is provided solely for the department to work with the department of transportation, University of Washington-Tacoma, and Washington State University-Puyallup to identify priority areas affected by 6PPD or other related chemicals toxic to aquatic life from roads and transportation infrastructure and on best management practices for reducing toxicity. This includes developing a standard method for the laboratory measurement of 6PPD-q and related chemicals. The department will submit a report to the appropriate committees of the legislature by November 1, 2022.*

The report summarizes outcomes of two expert workgroups. The assessment strategies workgroup found that the need for and amount of stormwater management needed will vary by geography. The work group also provided an initial evaluation of vulnerable areas to guide planning. The evaluation found that waterways supporting sensitive species which are near transportation corridors are the most vulnerable to fish mortality caused by 6PPD-Q. This initial evaluation is the basis for a more expansive study using graphical information systems (GIS).

The mitigation and stormwater management workgroup developed a stormwater management strategies report with the assistance of a contracted consultant group. The report summarized key physical and chemical properties of 6PPD-Q that affect stormwater management. The anticipated fate and transport without management actions was described, followed by a description of a BMP evaluation process. The evaluation process included flow, treatment, and source control BMPs. The group reviewed stormwater design manuals from eight state transportation agencies, including Caltrans. The report did not specify BMPs, but rather provided evaluation criteria for BMP selection.

Caltrans would be interested in Appendix A of the report, which provides a memorandum from

the Washington State Department of Transportation (WSDOT). The memorandum describes WSDOT's partnering approach with Washington Ecology and other public and private groups. The memorandum notes a $500 million authorization for enhanced stormwater treatment retrofits, including a map and table linking potential retrofit priority areas to vulnerabilities identified by the assessment strategies workgroup.

Additional appendices to the Washington Ecology report include a literature review and a compendium of green stormwater infrastructure information. The report does not go into monitoring data details, although the vulnerabilities assessment is informed by monitoring. A link in the report leads to Washington Ecology's Stormwater Action monitoring program. The scope of monitoring and availability of data from Washington Ecology and WSDOT will be researched and documented in more detail for the white paper.

Case Study of 6PPD-Q Criteria Development in Washington State

In August 2024, Washington Ecology updated water quality criteria, including a new acute exposure (1 hour duration) criterion of 12 ng/L for 6PPD-Q[138]. Washington Ecology based the criterion on the screening level approach adopted by USEPA in its recent water quality criteria guidance[43].

The normal procedure to develop aquatic life criteria for toxic substances following USEPA guidelines[139] involves gathering data from peer-reviewed toxicology studies that represent at least eight different taxonomic families. Washington Ecology lacked complete data supporting the eight-family procedure and so relied on alternative procedures. The alternative pathways are enabled by coho salmon's extreme sensitivity to 6PPD-Q relative to other species, and its commercial and cultural importance. USEPA criteria development guidance[139] notes under Part IV.A that:

> "...if the Species Mean Acute Value of a commercially or recreationally important species is lower than the calculated Final Acute Value, then that Species Mean Acute Value replaces the calculated Final Acute Value in order to provide protection for that important species."

In essence, results from the eight-species approach can be trumped by a sensitive commercially or recreationally important species. The Washington Ecology approach was premised on findings that coho salmon are commercially and recreationally important, and that coho salmon represent the most sensitive commercially and recreationally important species. The Washington Ecology criteria evaluation was focused on establishing a criterion that was below threshold effect levels. After evaluating and rejecting two other alternative approaches, Washington Ecology concluded that USEPA's screening level approach provided the most appropriate to protect coho salmon. They removed one data source for a species of zebrafish not found in North America and included more recent data sources to arrive at the 12 ng/L acute criterion, comparable to the 11 ng/L screening level established by USEPA.

Regulatory Setting Documents

Caltrans' interest

Caltrans' mission is to "provide a safe, sustainable, integrated, and efficient transportation

system to enhance California's economy and livability." Caltrans' interest in the regulatory setting comes from the sustainability part of the mission statement. Regulations direct Caltrans and regulatory agencies to rely on scientific findings of fact to establish water quality standards and implement those standards in permits.

Key Findings

Relevant to 6PPD-Q, three primary regulatory / resource agencies (Figure 1) direct Caltrans activities to implement policies compliant with the Federal Clean Water Act (CWA)[140] and the Endangered Species Act (ESA)[141]:

- The United States Environmental Protection Agency (USEPA)
- The National Marine Fisheries Service (NMFS)
- The California State Water Resources Control Board (SWRCB) and affiliated Regional Water Quality Control Boards (RWQCBs).

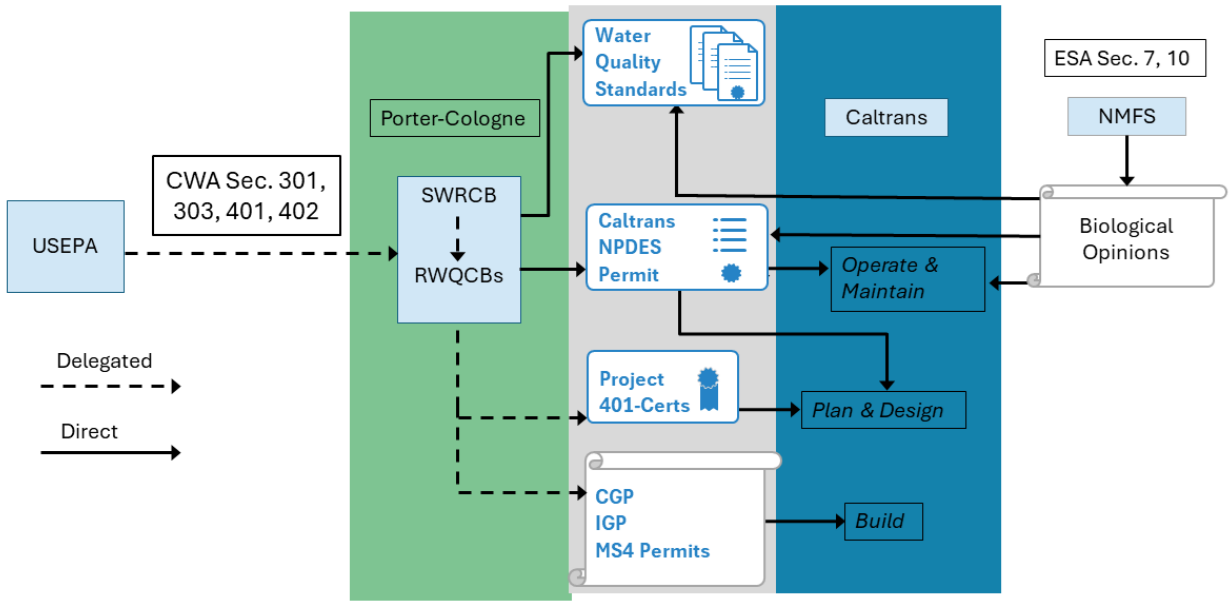


Figure 1. Regulatory Setting Requires Caltrans to Protect Water Quality and Endangered Species

Clean Water Act, USEPA, and the SWRCB

The USEPA delegates CWA authority to the SRWCB. Specific CWA programs are further delegated by SWRCB to the RWQCBs. Specific CWA sections germane to 6PPD-Q in Caltrans stormwater include:

- CWA Section 301 establishes the National Pollutant Discharge Elimination System (NPDES). This section prohibits the discharge of pollutants into navigable water except in compliance with the CWA.

- This section provides the SWRCB its delegated authority to issue Caltrans' NPDES permit, establish requirements in the permit, and take enforcement actions if requirements are not met.

- The SWRCB's CWA authorities are enhanced by water quality provisions in the State of California's Porter-Cologne Water Quality Control Act[142] (Porter-Cologne), for example:

    - Porter-Cologne section 13267 authorizes the SWRCB and RWQCBs to issue enforceable requests for monitoring information.

    - Porter-Cologne section 13383 serves a similar function as 13267 but is more commonly used when the recipient of the request for monitoring information is a public agency.

    - Porter-Cologne's jurisdiction for waters of the State extends beyond just navigable waterbodies, even though certain CWA authorities may be limited to navigable waters. For example, wetlands and groundwater are subject to Porter-Cologne in California.

- The SWRCB regulates all phases of Caltrans project activities, from planning, designing, and constructing new facilities to operating and maintaining existing ones.

- SWRCB directs Caltrans to manage water quality in stormwater discharges from Caltrans operations and maintenance through its NPDES permit.

    - SWRCB directs treatment in new or redeveloped Caltrans facilities, resulting in a rigorous program for including BMP in project designs.

    - During construction SWRCB regulates stormwater and other discharges from Caltrans facilities through the construction general permit (CGP), and limited use of the Industrial General Permit (IGP) and other general permits. Typically, Caltrans regulation during construction is delegated to the authority of the Regional Board with jurisdiction over the watershed containing the Caltrans construction project.

- Highway maintenance is considered an activity that causes less than significant impacts, according to Section 15300 of the California Environmental Quality Act (CEQA) guidelines[143].
    - Maintenance activities exempt under CEQA consist of the operation, repair, maintenance, permitting, leasing, licensing, or minor alteration of existing public or private structures, facilities, mechanical equipment, or topographical features, involving negligible or no expansion of use beyond that existing at the time of the lead agency's determination. The key consideration for exemption is whether the project involves negligible or no expansion of an existing use.
    - Examples include but are not limited to existing highways and streets, sidewalks, gutters, bicycle and pedestrian trails, and similar facilities. This includes road grading for the purpose of public safety.

- CWA Section 401 requires applicants for federal permits or licenses governing projects that may discharge pollutants into US waters obtain a state or interstate water quality certification. The "401-certification" describes measures and conditions to provide reasonable assurance that the project will comply with all applicable water quality standards.
    - Caltrans project 401-certifications are typically issued by the Regional Board with jurisdiction over the watershed containing the Caltrans construction project.
    - Regional Board 401-certifications may include additional requirements ranging from no additional action required, to monitoring requirements accompanied by trigger levels with associated response actions, to individual project Waste Discharge Requirements (WDRs).
- CWA Section 303 describes the federal program of water quality standards that is required to be implemented by states. Subsections relevant to 6PPD-Q include:
    - Section 303(c) requires states to adopt water quality standards, including water quality criteria.
        - Water quality standards under the CWA are comprised of designated uses of water and water quality criteria indicating attainment of designated uses.
        - Water quality criteria may be numeric or narrative.
    - Section 303(d) requires states to develop and maintain a list of impaired waterbodies and develop Total Maximum Daily Loads (TMDL) for impaired waterbodies that are not attaining water quality standards after all point sources in the watershed subject to NPDES permits have achieved existing permit effluent limits.
        - A TMDL is defined as the maximum pollutant load on a daily basis (or other time period) that a waterbody can assimilate and still achieve beneficial uses.
        - The TMDL is allocated among regulated point sources (i.e., point source discharges subject to NPDES permits) and non-point sources (e.g., forestry, agriculture). The allocations are translated into numeric limitations on pollutants discharged (effluent limits) and other requirements established in NPDES permits for point sources. In this context, Caltrans is considered a point source stormwater discharger subject to a NPDES Permit.
        - In the allocation of pollutant loads to meet water quality objectives the TMDL regulations require analysis of pollutant sources, fate, transport, degradation, dilution, and other relevant processes.

- CWA Section 304 defines the process to translate scientific data on pollutant toxicity into water quality objectives. Development of Water Quality Objectives requires considerable amounts of toxicity data, as described below[136]:
    - Acute toxicity data with at least one species of freshwater animal in at least eight different families.
    - Acute-chronic ratios (see Section VI) with species of aquatic animals in at least three different families provided that at least one is a fish, at least one is an invertebrate, and at least one is an acutely sensitive freshwater species.
    - Results of at least one acceptable test with a freshwater alga or vascular plant.
    - At least one acceptable bioconcentration factor determined with an appropriate freshwater species, if a maximum permissible tissue concentration is available.

In summary, data on 6PPD-Q toxicity to coho salmon alone are not sufficient to establish numeric water quality criteria following CWA Section 304 procedures. Establishment of water quality criteria by USEPA following normal CWA Section 304 guidance requires detailed, well-documented toxicity data from at least eight different species, acute-chronic ratios, and bioconcentration factors. However, as noted in the "Other State Government Programs" section above, states do have discretion to develop water quality criteria using toxicity data that do not completely fulfill the eight-families approach recommended by USEPA. Washington Ecology adopted an acute water quality criterion for 6PPD-Q (12 ng/L) following the USEPA's approach to develop non-regulatory screening levels for 6PPD-Q. In addition to the technical requirements of CWA Section 304, Porter-Cologne Section 13241 establishes administrative procedures for the SWRCB to adopt numeric water quality objectives with force of law in California. Factors Porter-Cologne Section 13241 requires SWRCB to consider when adopting water quality objectives include:

- Past, present, and probable future beneficial uses of water.
- Environmental characteristics of the hydrographic unit under consideration, including the quality of water available thereto.
- Water quality conditions that could reasonably be achieved through the coordinated control of all factors which affect water quality in the area.
- Economic considerations.
- The need for developing housing within the region.
- The need to develop and use recycled water.

NMFS ESA Consultations

Section 7 of the ESA requires federal agencies to consult with NMFS when any action that the agency carries out, funds, or authorizes may affect either a species listed as threatened or endangered under the Act, or any critical habitat designated for it. As illustrated in the right-hand side of Figure 1, consultations provide NMFS standing to influence Caltrans requirements under the CWA. Biological opinions issued pursuant to adoption of water quality standards, TMDLs, or NPDES permits have a federal nexus due to USEPA's ultimate approval authority over SWRCB actions.

Caltrans also consults directly with NMFS on projects and plans that may affect threatened or endangered species. For example, Caltrans is currently in negotiations with NMFS with the goal of proposing conservation measures to address the potential for direct impacts on listed endangered coho salmon resulting from 6PPD-Q in stormwater. In this negotiation, Caltrans is in the role of a federal lead agency acting as a delegate to the Federal Highway Administration. Section 7(a)(1) of the ESA charges federal agencies (and delegates) to aid in the conservation of listed species, and section 7(a)(2) requires the agencies to ensure their activities are not likely to jeopardize the continued existence of federally listed species or destroy or adversely modify designated critical habitat.

The negotiations are regarding Caltrans' Regional Programmatic Biological Opinion for routine construction and maintenance activities (Regional PBO). If, because of consultations, NMFS determines that threatened or endangered species may be in jeopardy, the Programmatic Biological Opinion Permit issued by NMFS may include reasonable and prudent measures to protect the species. Those reasonable and prudent measures would only apply to activities seeking coverage under the Regional PBO in the geographic area defined by the Regional PBO.

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