Exhibits

3M Chemical Operations LLC's Comments to
December 18, 2024 Draft
NPDES/SDS Permit No. MN0001449 for
3M Cottage Grove Facility
Cottage Grove, Washington County, Minnesota
February 3, 2025

Table of Contents

Exhibits to 3M Chemical Operations LLC's Comments to December 18, 2024 Draft NPDES/SDS Permit No. MN0001449

As stated in 3M's February 3, 2025 Comments to the Revised Draft Permit, 3M incorporates by reference the following materials:

3M Chemical Operations LLC's Comments to Draft NPDES/SDS Permit No. MN0001449 for 3M Cottage Grove Facility submitted August 30, 2024 (August 2024 Comments); and

3M Chemical Operations LLC's Exhibits A through L, and Appendix 1 and Appendix 2 to its August 2024 Comment's to MPCA Draft NPDES/SDS Permit MN0001449.¹

EXHIBIT NO. EXHIBIT DESCRIPTION

- 1. Comments of Robyn Prueitt, Ph.D. DABT, and Tim Verslycke, Ph.D., Related to Reissuance of the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit MN0001449 for the 3M Cottage Grove Center Facility in Cottage Grove, Minnesota (Jan. 31, 2025) ("Suppl. Gradient Report"). Attachment 1 is the Gradient Expert Report which was previously provided as "Exhibit G" to 3M's August 2024 Comments and is incorporated herein.
- 2. Supplemental Technical Review, 3M Cottage Grove Advanced Wastewater Treatment System PFAS Treatment System to Arcadis Expert Report, "Treatability Review Memorandum" prepared by Joseph Quinnan, PE, PG, SVP/Director of Emerging Contaminants, and Keith Foster, PG (Feb. 3, 2025) ("Suppl. Arcadis Report). Exhibit A is the Arcadis Expert Report which was previously provided as "Exhibit D" to 3M's August 2024 Comments and is incorporated herein.
- **3.** Declaration of Michael J. Parent, Ph.D.
- **4.** Low Flow Determination Notes prepared by Bruce Henningsgaard (July 17, 2023).
- 5. StreamStats Report-Unnamed Creek

¹ Due to electronic file size limitations, addition to the electronic files submitted online 3M filed a hard copy of its August 2024 Comments, Exhibits and Appendices with MPCA on August 30, 2024. *See attached herewith* August 2024 Comments Exhibit and Appendix Table of Contents. For complete copies of each exhibit, please reference the hard copies provided.

TABLE OF CONTENTS OF EXHIBITS AND APPENDIX TO 3M Chemical Operations LLC's August 2024 Comment's to MPCA Draft NPDES/SDS Permit MN0001449

Exhibits A through L, and Appendix 1 and Appendix 2

Table of Contents EXHIBITS & APPENDIX TO 3M COMMENTS TO MPCA DRAFT NPDES/SDS PERMIT MN0001449 Submitted August 30, 2024

EXHIBIT NO.	EXHIBIT DESCRIPTION
Α	PFAS Treatability Studies (herein referenced collectively as the "Treatability Study"
A-1	Montrose Environmental Group and Barr Engineering, PFAS Treatability Study Alternatives Identification Plan, 3M Cottage Grove, MN Facility (May 2021)
A-2	Montrose Environmental Group and Barr Engineering, PFAS Treatability Study Alternatives Identification Plan (Updated), 3M Cottage Grove, MN Facility (July 2021)
В	Barr Engineering, PFAS Treatability Study (Dec. 22, 2021) ("Pilot Study")
С	3M Cottage Grove Wastewater Treatment Facility, Plan and Specification Approval, Building 150 and Building 151 Project, NPDES/SDS Permit Number MN0001449, (May 17, 2023). ("Approval Letter")
D	Arcadis, Treatability Review Memorandum, prepared by Corey Theriault, PE, Keith Foster, Lauren March, PE of Arcadis ("Arcadis Expert Report")
Е	Impact of Intervention Limits on Advanced Wastewater Treatment System Performance, (Aug. 28, 2024) ("Kaczynski Expert Report")
F	Written correspondence cited in Background section of Comments Letter
F-1	January 12, 2024 Letter from MPCA to 3M transmitting PPN Draft Permit
F-2	January 22, 2024 Letter from 3M to MPCA requesting response extension
F-3	January 25, 2024 MPCA grants 3M extension
F-4	February 5, 2024 3M's revised request for extension
F-5	February 15, 2024 3M's initial comments re PPN Draft Permit
F-6	March 18, 2024 MPCA response to 3M's 2/15 comments

EXHIBIT NO.	EXHIBIT DESCRIPTION
F-7	March 26, 2024 3M comments re Compliance Schedule
F-8	March 28, 2024 3M Letter to Commissioner Kessler
F-9	April 3, 2024 MPCA letter re Phase 3 wastewater treatment system
F-7	March 26, 2024 3M comments re Compliance Schedule
F-8	March 28, 2024 3M Letter to Commissioner Kessler
F-9	April 3, 2024 MPCA letter re Phase 3 wastewater treatment system
F-10	April 11, 2024 3M response to 4/3 letter
F-11	April 23, 2024 MPCA request for additional maps and diagrams
F-12	April 26, 2024 3M response to 4/23 request
F-13	April 30, 2024 3M response to MPCA re Proposal for Changes to Draft Permit
F-14	May 1, 2024 MPCA request to 3M to provide data/calculations re reporting limits
F-15	May 10, 2024 MPCA correspondence re Updated Limits Notifications
F-16	May 29, 2024 3M letter re Compliance Schedule and Intervention Limits
F-17	May 30, 2024 3M provided AWTS milestones to MPCA
F-18	June 13, 2024 3M submittal to MPCA re NTAs
G	Report "Related to Reissuance of the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit MN0001449 for the 3M Cottage Grove Center Facility in Cottage Grove, Minnesota", prepared by Robyn Prueitt, Ph.D., and Tim Verslycke, Ph.D. ("Gradient Expert Report")
Н	Memorandum from Rock Vitale, CEAC, Environmental Standards, Inc., Response to MPCA Proposed Intervention Limits for 3M's Cottage Grove, Minnesota facility, Calendar Average and Daily Maximum ("Vitale Expert Report")
I	PFAS Analyte Table

EXHIBIT NO. **EXHIBIT DESCRIPTION** J Weston Solutions Inc., 3M 2023 Instream PFAS Characterization Study Final Report-Mississippi River, Cottage Grove, Minnesota (June 29, 2023) ("2023 IPC Study")1 K Tables and Figures from the 3M 2023 Instream PFAS Characterization Study Final Report-Mississippi River, Cottage Grove, MN, Weston Solutions, Inc. issued June 29, 2023 ("IPC Study") Table 1. PFAS Detections in Surface Water from Reaches 02 and 03 Table 2. PFAS Detections in Fish Fillet from Reaches 02 and 03 (7 fish species) Figure 2. PFOS Decrease in Pool 2 fish fillet (2005-2021) Table 3. DT50 and DT90 for PFOS in the Mississippi River Pools 2 and 3 (2005-2021) Figure 3. PFOS levels in Bde Maka Ska (formerly Calhoun) and Lake Harriet; MPCA Data Table 4. Comparison of 2021 IPCS to recent instream PFAS studies in scientific literature

L Settlement Agreement and Compliance Order between MPCA and 3M dated May 2027 ("SACO")

APPENDIX NO. APPENDIX DESCRIPTION

- 3M Comments on Draft Permit and Fact Sheet Additional Draft Permit Comments
- 2. 3M Comments on Draft Permit and Fact Sheet Additional Draft Permit Comments Compliance Dates

¹ Note: 3M hereby incorporates the final version of the 2023 IPC Study by reference due to size limitations. The study was provided to MPCA in draft on April 28, 2023 and in final on June 29, 2023.

EXHIBIT 1

Supplemental Gradient Report

Comments by Robyn Prueitt, Ph.D., DABT, and Tim Verslycke, Ph.D., Related to Reissuance of the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit MN0001449 for the 3M Cottage Grove Center Facility in Cottage Grove, Minnesota

Prepared by

Robyn Prueitt, Ph.D., DABT

Romm Prueitt

Tim Verslycke, Ph.D.

January 31, 2025



One Beacon Street, 17th Floor Boston, MA 02108 617-395-5000

Table of Contents

		<u>Page</u>
1	Introdu	oction1
2	Summa	rry of Opinions2
	2.1	MPCA's methodology for calculating fish bioaccumulation factors (BAFs) was corrected to be consistent with US EPA guidance
	2.2	MPCA's use of toxicological values is still inconsistent with Minnesota's water quality rules and previous approaches used by MPCA
	2.3	MPCA continues to rely on an interim fish consumption rate (FCR) that overestimates fish consumption, is not representative of site-specific conditions, and is higher than what is used by other states and US EPA
	2.4	MPCA continues to not provide the necessary underlying information to allow for an independent evaluation and verification of its analyses
	2.5	MPCA's approach to developing fish-tissue-based criteria for perfluorooctanoic acid (PFOA) and perfluorohexane sulfonic acid (PFHxS) remains inconsistent with its own guidance and best available science
	2.6	MPCA's continued and consistent reliance on unsupported toxicological values and exposure parameters, when considered in combination, results in site-specific criteria (SSCs) that are not site-specific, and are inconsistent with similar values developed by other regulatory entities and with MPCA's own regulatory processes to protect the designated uses of the Mississippi River Miles 820 to 8129
Referer	nces	11
Attachr	ment 1	Expert Report of Robyn Prueitt, Ph.D., DABT, and Tim Verslycke, Ph.D., Related to Reissuance of the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit MN0001449 for the 3M Cottage Grove Center Facility in Cottage Grove, Minnesota

Abbreviations

3M 3M Company

BAF Bioaccumulation Factor

BCC Bioaccumulative Chemical of Concern

CC_{FR} Fish Consumption and Recreation Use Class Chronic Criterion

CC_{FT} Fish-Tissue-Based Chronic Criterion

CSF Cancer Slope Factor

½ DL One-Half of the Detection Limit ECHA European Chemicals Agency FCR Fish Consumption Rate

FISH Fish Are Important for Superior Health

ITRC Interstate Technology and Regulatory Council

MDH Minnesota Department of Health MPCA Minnesota Pollution Control Agency

NPDES National Pollutant Discharge Elimination System

ORD Office of Research and Development
PFAS Per- and Polyfluorinated Substances

PFBA Perfluorobutanoic Acid

PFBS Perfluorobutane Sulfonic Acid

PFHxA Perfluorohexanoic Acid

PFHxS Perfluorohexane Sulfonic Acid

PFOA Perfluorooctanoic Acid

PFOS Perfluorooctane Sulfonic Acid

RfD Reference Dose

ROS Regression on Order Statistics

SDS State Disposal System
SSC Site-Specific Criterion
SSCs Site-Specific Criteria

US EPA United States Environmental Protection Agency

WCBA Women of Child-Bearing Age
WQC Water Quality Criterion
WQCs Water Quality Criteria

1 Introduction

Drs. Robyn Prueitt and Tim Verslycke were retained by 3M Chemical Operations LLC (3M) to provide technical expert services related to the reissuance of its National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit for the 3M Cottage Grove Center facility located in Cottage Grove, Minnesota, for which they generated an expert report on August 27, 2024 (Prueitt and Verslycke, 2024, included as Attachment 1). In their August 2024 report, Dr. Prueitt provided expert toxicology comments and Dr. Verslycke provided expert ecotoxicology comments related to the proposed effluent limits for per- and polyfluorinated substances (PFAS) in a draft permit published by the Minnesota Pollution Control Agency (MPCA) on July 1, 2024, for public notice (MPCA, 2024a,b). The draft permit was updated by MPCA due to comments received during public notice and a public notice for a revised draft permit was released on December 18, 2024 (MPCA, 2024c,d,e). Section 2 of this report presents an evaluation by Drs. Robyn Prueitt and Tim Verslycke of the revised permit and associated Human Health Protective Water Quality Criteria for PFAS in Mississippi River, Miles 820-812 (December 2024 sitespecific criteria [SSCs]; MPCA, 2024f) in light of their comments on the July version of the draft permit (Attachment 1).

The qualifications of Drs. Robyn Prueitt and Tim Verslycke are described in Section 1.2 of Attachment 1. Gradient is compensated at the rate of \$495/hour for the expert services of Drs. Robyn Prueitt and Tim Verslycke.

2 Summary of Opinions

Our opinions are based on the information sources we reviewed, in addition to our education, training, research, and professional experience in toxicology, ecotoxicology and risk assessment. Date and information sources that were relied upon in preparing this expert report are provided in the References section. We reserve the right to supplement or amend our opinions should new facts or information be made known to us.

The subsections below discuss how the revised permit addressed each of our prior opinions (as presented in Section 3 of our prior report, included here as Attachment 1). To the extent the revisions made to the permit do not impact the opinions in our prior report, we do not re-state our opinions in this supplemental report but rather incorporate our prior report by reference.

2.1 MPCA's methodology for calculating fish bioaccumulation factors (BAFs) was corrected to be consistent with US EPA guidance.

As described in Section 3.5 of our prior report (Attachment 1), MPCA originally calculated PFAS fish tissue and surface water geometric means for use in bioaccumulation factor (BAF) derivations using five different approaches to address non-detected data. The Regression on Order Statistics (ROS) method was ultimately chosen by MPCA (2024g) to calculate geometric means, and one-half of the detection limit (½ DL) was used as a substitution for values reported as non-detected when the data did not meet the ROS criteria. We noted that the ROS method, as chosen and implemented by MPCA (2024g) in the prior draft permit, was inconsistent with United States Environmental Protection Agency's (US EPA) Office of Research and Development's (ORD) guidance (US EPA, 2006), which requires that both the number of detected observations be large enough to obtain accurate and reliable results and that the data follow a well-known parametric distribution.

In the revised draft permit, MPCA (2024f) no longer uses ROS-based BAFs, noting that "With the current dataset, the fish tissue geometric means calculated from ROS were over-inflated when compared to other substitution methods," and stating that "This is likely due to the amount of non-detections in the dataset." Instead, MPCA (2024f) uses the ½ DL-based BAFs, which resulted in criteria that are now higher than those presented in the previous draft permit (Table 2.1).

MPCA's correction to its methodology for handling non-detected values in its derivation of BAFs is responsive to our prior comments and considered an appropriate methodology that is consistent with applicable US EPA guidance, albeit creating other problems by arbitrarily and artificially inflating detection frequencies to justify MPCA's decision to develop SSCs based upon fish consumption. However, we note that MPCA has not corrected other issues that we identified in our original report, as discussed below. We also identify an additional issue that was not described in our original report and relates to MPCA's inaccurate assignment of concentrations to a substantial number of non-detected fish tissue values, as described further in Section 2.4.

2.2 MPCA's use of toxicological values is still inconsistent with Minnesota's water quality rules and previous approaches used by MPCA.

In our prior report (Section 3.1, Attachment 1), we evaluated the toxicological values relied upon by MPCA as inputs in the derivation of SSCs for six PFAS for the Mississippi River Miles 820 to 812, as described in MPCA's May 2024 criteria development report (MPCA, 2024g). We found that MPCA used toxicological values developed by US EPA that are not consistent with Minnesota's water quality rules and that differ from the toxicological values that MPCA has previously used for developing water quality criteria for these same PFAS.

MPCA's December 2024 SSCs (MPCA, 2024f) continue to use US EPA values, which is inconsistent with Minnesota regulations. MPCA states in the December 2024 SSCs that the toxicological values used to derive the SSCs for the six PFAS were taken from US EPA's most recent evaluations of these PFAS and that the values meet the Minnesota water quality rules because they were developed using standard US EPA methodology, from which the MPCA's and Minnesota Department of Health's (MDH) methodologies for deriving toxicological values are based. As noted in our prior report (Section 3.1, Attachment 1), Minnesota's water quality rules indicate that toxicological values from US EPA can be used for deriving water quality criteria, but only after those values have been evaluated and any needed modifications by MDH have been completed (MPCA, 2017). There is no evidence that such an evaluation happened here or that any MDH evaluation has been subjected to public scrutiny and comment.

Further, MPCA's argument that MDH's methodologies are "based on" US EPA's methodology is not correct when it comes to PFAS. It is important to emphasize that even if MDH's general methodology for developing toxicological values is based on the US EPA standard methodology, MDH's methodology has differed from that of US EPA where PFAS are concerned. As noted in our prior report, MDH has used toxicokinetic model parameters to convert serum levels of PFAS to human equivalent doses that are different from those used by US EPA, which results in different toxicological values. For example, as described in Sections 3.1.1 and 3.1.2 of our prior report (Attachment 1), even when based on the same underlying health effects from the same studies, the reference dose (RfD) for perfluorooctane sulfonic acid (PFOS) developed by MDH (2024a) is an order of magnitude higher than the RfD developed by US EPA (2024a), and the cancer slope factor (CSF) for perfluorooctanoic acid (PFOA) developed by MDH (2024b) is less than half the value of the CSF developed by US EPA (2024b). Thus, the methodologies for deriving toxicological values for PFAS differ between the agencies, resulting in different values, and the use of the US EPA values without evaluation and modification by MDH (particularly in terms of toxicokinetic model parameters) is not consistent with Minnesota water quality rules. I

2.3 MPCA continues to rely on an interim fish consumption rate (FCR) that overestimates fish consumption, is not representative of site-specific conditions, and is higher than what is used by other states and US EPA.

As described in Section 3.2 of our prior report (Attachment 1), MPCA's interim fish consumption rate (FCR) for women of child-bearing age (WCBA) of 66 g/day has not been demonstrated to be reflective of fish consumption patterns for the Mississippi River Miles 820 to 812, is not consistent with US EPA guidance on the development of water quality criteria (WQCs), is greater than two-fold higher than

¹ We also noted in our prior report that MPCA inappropriately used a draft RfD value from US EPA for the perfluorohexane sulfonic acid (PFHxS) SSC in its May 2024 report (MPCA, 2024f), but this RfD has since been finalized by US EPA; however, it is still not consistent with Minnesota water quality rules to use US EPA toxicological values without evaluation and modification by MDH.

Minnesota's default FCR, is substantially higher than FCRs developed by other states and US EPA, and hence is not based on the best available and reliable data.

MPCA continues to rely on an interim FCR for WCBA of 66 g/day based on the MDH "Fish are Important for Superior Health (FISH)" survey of people residing on Minnesota's North Shore (MDH and UIC, 2017). We previously commented that MPCA (2024g) did not discuss how the fish consumption patterns and local conditions in the 2017 survey of people residing in the Lake Superior north shore area (MDH and UIC, 2017) reflect those of people located in the vicinity of the Mississippi River Miles 820 to 812, or how the surveyed population in the 2017 survey compares to the demographics of the target population that may consume fish caught in the Mississippi River near the Cottage Grove facility. In the revised permit, MPCA (2024f) provides additional discussion of its selection of an FCR of 66 g/day by stating that:

Women of Childbearing Age can be considered the most vulnerable population, due to the developmental effects on the unborn child, and this population is often advised to eat fish to support developing pregnancies. The recommended amount given by the FDA is 8 to 12 oz per week, which translates to 32 to 49 g/day, which is in excess of the FCR in rule (30 g/d) in Minnesota. WCBA are encouraged to consume more than what is in rule, making the nonspecific FCR in Rule inappropriate for this vulnerable population.

Additionally, the North Shore FISH survey did include indigenous women as part of the cohort, but the percentage of that population is not known, due to privacy given to the study participants. Indigenous populations have cultural practices that lead to consumption of larger quantities of harvested fish, which is accounted for in EPA's use of a subsistence fish consumption rate of 142.4 g/d. Prairie Island Indian Community is downstream of this discharge, and their WCBA may be at greater risk of excess exposure due to the additional fish consumption. Tribal members fish throughout Pools 2 and 3 and they consume whole fish in multiple ways that may increase their exposure to PFAS. It is critically important that MPCA consider this consumption, so that residents of the Prairie Island Community can safely enjoy eating fish taken from the river and fulfill their traditional practices. Using MPCA's interim FCR of 66 g/d is not unreasonable to ensure better protection of WCBA that may be consuming more fish to ensure a healthy pregnancy, based on current federal advice and/or cultural practices.

The data collected on the North Shore was the most robust dataset in Minnesota that evaluated WCBA, who are the most vulnerable population within the Cottage Grove site-specific area. The FCR is chemical specific, due to the developmental toxicity of the PFAS evaluated for this SSC. This makes the rate site specific because the chemicals being released at this site are developmental toxicants. Using the interim FCR_{WCBA} is more appropriate to ensure women's health is protected, rather than using the base FCR in rule, which was not calculated with the intent to protect vulnerable populations. Using a FCR that was calculated with consumption patterns of indigenous populations and WCBA helps protect the Prairie Island Indian Community that live and recreate in the area.

None of this information justifies MPCA's use of an FCR of 66 g/day, which continues to be a hypothetical fish consumption rate that is unsupported by site-specific fish survey information for the Mississippi River Miles 820 to 812. As described in our prior comments (Attachment 1), US EPA (2014) reported FCRs for WCBA (all races) of 15.8 g/day at the 90th percentile, 23.5 g/day at the 95th percentile, and 46.6 g/day at the 99th percentile. The interim FCR selected by MPCA is substantially higher than the 99th percentile value for WCBA derived by US EPA (2014). So, MPCA's support for use of 66 g/day as needed to protect WBCA is not consistent with information from national surveys as reported by US EPA (2014). Overall,

MPCA's interim FCR continues to overestimate fish consumption in Mississippi River Miles 820 to 812 and further exacerbates the errors in MPCA's approach.

Finally, MPCA refers to the FCR as "site-specific" because it considered the developmental toxicity of PFAS, and states that "Minnesota Rule allows for consideration of other scientifically defensible algorithms on a chemical-specific basis for evaluating developmental susceptibility to toxic pollutants in fish tissue (Minn. R. 7050.0219, subp. 2A)." That provision of the Minnesota Rules is inapplicable to MPCA's analysis here, for two reasons. First, whether or not a chemical is a developmental toxicant has no bearing on whether the chosen FCR is site-specific. Second, for the reasons noted above, MPCA's use of an FCR of 66 g/day is not a "scientifically defensible algorithm" and MPCA provides no evidence of prior use of an FCR of 66 g/day because a chemical is a developmental toxicant.

2.4 MPCA continues to not provide the necessary underlying information to allow for an independent evaluation and verification of its analyses.

As described in Section 3.3 of our prior report (Attachment 1), MPCA (2024f) provided no detail on how fish tissue and surface water data in its derivation of BAFs were adjusted or eliminated and for what reason. We also previously commented that MPCA did not follow US EPA Region III (1991) guidance by not clearly identifying what analytical quantitation limits it used to support its non-detect substitution calculations. No additional detail was provided in response to our comments in the revised draft permit. As a result, our prior comments continue to hold.

In our prior comments, we also pointed out errors based on an independent verification of MPCA's calculation, where possible, based on the available information. Some specific errors pointed out in our prior comments were corrected by MPCA (2024f). For example, the PFOS fish tissue geometric mean for trophic level 4 derived using the zero method (Table A-6) was corrected to 13.4 ng/g.

As described in Section 2.1, in the revised draft permit, MPCA (2024f) corrected its inappropriate use of ROS-based BAFs and implemented ½ DL-based BAFs. However, MPCA has continued to rely on an underlying concentration fish tissue dataset for PFOA, perfluorohexanoic acid (PFHxA), perfluorobutanoic acid (PFBA), and perfluorobutane sulfonic acid (PFBS) that is flawed. For 148 fish fillet concentrations that are qualified as non-detect (below the lower limit of quantitation and flagged with a "U,X" qualifier), MPCA used the full detection limit for its calculations instead of the ½ DL (3M Co., 2025). This resulted in inaccurate geomeans and artificially higher detection frequencies as reported by MPCA (37.1% for PFOA, 41.4% for PFHxA, 49.6% for PFBA, and 69.8% for PFBS. When properly accounting for these non-detected results, detection frequencies in fish fillets are much lower, 23.6% for PFOA, 5.7% for PFHxA, 34.5% for PFBA, and 28.1% for PFBS (3M Co., 2025). MPCA relied, in part, on these inaccurate and artificially inflated detection frequencies, to inappropriately support its decision to deriving fish tissue criteria, as described further in Section 2.5.

GRADIENT 5

-

² Using the electronic data deliverable (EDD), 3M verified that the "U" qualifier indicated a value below the method quantitation limit. The "X" qualifier denotes a quality control-related comment in the analytical report. Therefore, results flagged as "U,X" should be treated as non-detects.

2.5 MPCA's approach to developing fish-tissue-based criteria for perfluorooctanoic acid (PFOA) and perfluorohexane sulfonic acid (PFHxS) remains inconsistent with its own guidance and best available science.

As described in Section 3.4 of our prior report (Attachment 1), MPCA's approach for developing fish-tissue-based criteria for PFOA and PFHxS was not supported by the current state of the science and was inconsistent with its own prior interpretation of the bioaccumulation potential of PFOA and PFHxS (MPCA, 2024g). The December 2024 SSCs (MPCA, 2024f) do not make changes to the list of PFAS for which they derived fish tissue-based SSCs, but it provides additional discussion in support of deriving fish-tissue based criteria for PFOA and PFHxS:

While the mean BAFs for PFHxS and PFOA were less than 1,000 L/kg at this site, one black crappie fish tissue sample (measured at 15.2 ng/g) in the dataset used to determine the current SSC (Weston 2023) exceeded the BAF of 1,000 for PFHxS. Additionally, both PFOA and PFHxS have demonstrated BAFs > 1,000 L/kg in fish in other field studies (ITRC 2018). Both PFHxS and PFOA are known to be highly bioaccumulative in humans with long half lives (5.3 years and 2.7 years, respectively) (Li et al., 2018), and exhibit potential toxic effects at exceptionally low concentrations. In addition, PFHxS and PFOA are present in at least 40% and 37% of fish fillets at this site, respectively, presenting a route of exposure for people consuming fish collected in this area.

Both PFOA and PFHxS have the potential to cause adverse effects in humans (as further discussed in Sections 5 and 6 of this document), but have physiochemical properties that are unique from other bioaccumulative chemicals. Both PFOA and PFHxS are water soluble, and rather than partitioning to lipid tissue in aquatic organisms (as is typical of most bioaccumulative compounds), they bind to proteins in blood and liver and are expected to be excreted through the fish's gills during respiration. Aquatic organisms are less likely to accumulate PFOA and PFHxS, when compared to terrestrial animals (ECHA 2017). The Rule language in 7052.0010, subp. 4 allows for the consideration of metabolism and other physiochemical properties of the chemicals. The unique properties of these chemicals, which create an inhibition of bioaccumulation due to metabolism and excretion of these chemicals in aquatic organisms, should be considered alongside the knowledge about the accumulation in humans, because the purpose of the fish tissue criteria is to protect human health. If the narrow definition of having a BAF of > 1,000 in aquatic organisms is only considered, this can underestimate the exposure to humans via the route of fish consumption.

Bioaccumulation factors are not generally calculated for terrestrial organisms, but determination of chemical half-lives is often used instead, as an indication of how long a chemical will remain in the body. Both PFOA and PFHxS have very long half-lives in humans (5.3 and 2.7 years, respectively; Li et al. 2018), and both have demonstrated long half-lives in other terrestrial organisms as well. Perfluorohexane sulfonic acid has a half-life of nearly 2 years in pigs (which exceeds the half-life of PFOS in pigs; Numata et al., 2014), and nearly 5 months in monkeys (ECHA 2017). Perfluorooctanoic acid has a half-life in pigs of nearly 8 months (Numata et al. 2014). Due to the long half-lives of these chemicals, they will accumulate in human blood, because each new exposure (via water, fish, etc.) will add to what is already in the body. The tissue criteria values are important to ensure protection of human consumers because many fish at this site already contain PFOA and/or PFHxS at concentrations that exceed the calculated fish tissue values.

Perfluorooctane sulfonic acid, PFOA and PFHxS are entering the fish and accumulating to levels that can cause adverse effects to vulnerable human populations (such as women of childbearing age, subsistence fishing populations, etc.).

Many fish at the site had detectable levels of PFOA and PFHxS (37% and 40% of filets, respectively). The detection and reporting limits for the two chemicals are higher than their calculated CC_{FT} values. This means that potentially, additional fish could contain the chemicals at concentrations higher than the CC_{FT}. Because the chemicals elicit adverse effects at very low concentrations, and take years to be eliminated from the body, consumption of fish could be a significant exposure route, even with lower BAFs. The intent of fish tissue criteria is to protect human health from significant exposures to toxic chemicals, when exposure via fish may be an important route of exposure (MPCA 2014). Even though PFOA and PFHxS do not consistently result in BAFs in fish that exceed the > 1000 threshold, their properties still cause a significant exposure route for humans, given their low toxicological values and very long half-lives in humans.

The additional support provided by MPCA (2024f) for the appropriateness of developing fish-tissue-based criteria for PFOA and PFHxS does not address our previous comments, and MPCA's decision to develop fish-tissue-based criteria for PFOA and PFHxS remains unsupported by the current state of the science and inconsistent with its prior interpretation of the bioaccumulation potential of PFHxS and PFOA, for the following reasons:

- MPCA's statement that "Even though PFOA and PFHxS do not consistently result in BAFs in fish that exceed the >1,000 L/kg threshold" is misleading and not supported by the data. In fact, the existing dataset clearly supports the opposite, PFOA and PFHxS consistently result in site-specific BAFs significantly below the 1,000 L/kg threshold by more than an order of magnitude. MPCA (2024f) points to a single PFHxS black crappie fish tissue sample (15.2 ng/g) with a calculated BAF exceeding 1,000 L/kg. Based on the underlying data (Weston Solutions, 2023), this sample is one of 20 black crappie PFHxS filet samples collected from Reach 2/Pool 3/Section 1 and Reach 2/Pool 2/Section 4, with a geometric mean concentration of 0.292 ng/g). This one PFHxS fish tissue sample of 15.2 ng/g is clearly a statistical outlier, as shown in Figure 2.1. Therefore, the BAF of this one unrepresentative, outlier sample is not convincing and consistent evidence. Further, MPCA's pointing to a BAF from a single fish tissue sample as support for categorizing PFHxS as a bioaccumulative chemical of concern (BCC) is inappropriate and inconsistent with the Minnesota Rules for developing BAFs (Minn. R. 7050.0219, subp. 10-11 [MPCA, 2020] and Minn. R. 7052.0110; [MPCA, 2024h]), which state that BAFs should be derived on the basis of a geometric mean, calculated using acceptable individual BAFs (i.e., the determination of whether a substance is a BCC should be based on the geomean BAF and not individual BAFs). MPCA (2024f) provides no discussion about the representativeness of this single tissue sample as compared to other fish tissue samples for PFHxS for the same, as well as other, fish species.
- The citing of BAFs from different studies compiled by Interstate Technology and Regulatory Council (ITRC) is irrelevant and contrary to the value of using site-specific information to derive site-specific BAFs and criteria. Further, as previously described in our prior report (Attachment 1), the field studies in the ITRC database that MPCA refers to carry substantial uncertainty since they collected whole tissue instead of fillets and collected fish tissue and water at different times. As a result, the findings of these studies are not appropriate for evaluating bioaccumulation in edible fish tissue.

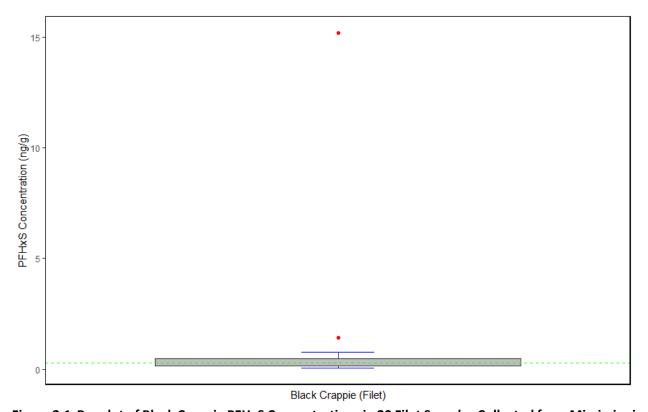


Figure 2.1 Boxplot of Black Crappie PFHxS Concentrations in 20 Filet Samples Collected from Mississippi River Miles 820 to 812. Two outliers are represented by red points (1.41 ng/g and 15.2 ng/g); the gray box represents the range between the lower quartile [Q1], 0.138 ng/g, and the upper quartile [Q3], 0.480 ng/g, known as the interquartile range [IQR]; the geometric mean (0.292 ng/g) is represented by the dashed green line; the whiskers are in blue and represent the lower bound ([Q1 - 1.5 x IQR]; -0.375 ng/g) and the upper bound ([Q3 + 1.5 x IQR]; 0.993 ng/g). Values that fall below the lower bound or exceed the upper bound are considered outliers in the IQR Method.

- As discussed in Section 2.4, for 148 fish samples with concentrations below the lower limit of quantitation, MPCA inappropriately applied the full detection limit instead of ½ DL. As a result, MPCA's reported detection frequencies are inaccurate and artificially high for some PFAS. For example, MPCA states that "PFHxS and PFOA are present in at least 40% and 37% of fish fillets at this site" when in fact the detection frequency for PFOA is 23.6% when properly assigning non-detects in the underlying dataset. MPCA supports its decision to derive fish tissue criteria, in part, by referencing inaccurate detection frequencies, which undermines the scientific validity of that decision.
- MPCA's discussion of physiochemical properties and metabolism in humans, as related to the Rule language in 7052.0010, subp. 4, is confusing. That Rule provides:

"Bioaccumulative chemical of concern' or 'BCC' means any chemical that has the potential to cause adverse effects which, upon entering the surface waters of the state, by itself or as its toxic transformation product, accumulates in aquatic organisms by a human health bioaccumulation factor (BAF) greater than 1,000, after considering metabolism and other physiochemical properties that might enhance or inhibit bioaccumulation, in accordance with the methodology in part 7052.0110, subpart 3. Chemicals with half-lives of less than eight weeks in the water column, sediment, and biota are not BCCs. The minimum BAF information needed to define an organic

chemical as a BCC is either a field-measured BAF or a BAF derived using the biota-sediment accumulation factor (BSAF) methodology. The minimum BAF information needed to define an inorganic chemical, including an organometal, as a BCC is either a field-measured BAF or a laboratory-measured bioconcentration factor. The BCCs are a subset of the GLI pollutants, and are listed in part 7052.0350. A chemical may not be treated as a BCC for purposes of this chapter unless and until it is added to the list in part 7052.0350."

It is not clear how this Rule language allows for a qualitative consideration of metabolism in terrestrial animals as opposed to fish, as well as a qualitative consideration of physiochemical properties in determining whether a chemical is a bioaccumulative chemical of concern (BCC). Contrary to the language in the Rule, which requires a finding that a chemical "accumulates in aquatic organisms by a human health bioaccumulation factor (BAF) greater than 1,000, after considering metabolism and other physiochemical properties,", MPCA's interpretation appears to support designating chemicals as BCCs almost independent of their BAFs in fish (*i.e.*, if a chemical is deemed by MPCA to meet some unknown threshold for metabolism and/or the chemical has physiochemical properties that are deemed to be problematic, then the fish BAF can be discounted, and the chemical can be considered a BCC). MPCA provides no evidence of a prior instance where a chemical has been designated as a BCC on the basis of these qualitative considerations when the BAF of that chemical was well below 1,000.³

In sum, while MPCA (2024f) has provided additional support for its designation of PFOA and PFHxS as BCCs, our prior comments hold, and MPCA's decision to develop fish-tissue-based criteria for PFOA and PFHxS is not supported by the current state of the science and inconsistent with its own prior interpretation of the bioaccumulation potential of these two PFAS. Further, in the additional support MPCA provided, it inappropriately points to a single outlier fish PFHxS BAF value as support for categorizing PFHxS as a BCC. Finally, MPCA has not corrected issues related to inaccurately assigning concentrations to non-detected values in the underlying fish tissue dataset, which has resulted in MPCA's reporting of artificially high detection frequencies for some PFAS, including PFHxS.

2.6 MPCA's continued and consistent reliance on unsupported toxicological values and exposure parameters, when considered in combination, results in site-specific criteria (SSCs) that are not site-specific, and are inconsistent with similar values developed by other regulatory entities and with MPCA's own regulatory processes to protect the designated uses of the Mississippi River Miles 820 to 812.

As described in Section 3.6 of our prior report (Attachment 1), MPCA did not include any discussion of the uncertainty associated with its SSC derivation and the importance of the various input parameters it selected. We stated that this is a significant omission and contrary to established state and federal guidance and policy on risk assessment in environmental decision-making. MPCA's revised draft permit continues to lack an uncertainty discussion, and there is no evaluation of how alternative toxicological values and exposure parameters impact the SSCs it developed.

MPCA (2024f) did not make any changes to the toxicological values (with the exception of correcting the US EPA RfD for PFHxS, which was a transcription error we identified in our prior comments) and exposure parameters in this revised draft permit. MPCA did change the methodology for calculating BAFs to be consistent with our comments. This resulted in higher fish consumption and recreation use class chronic

³ To the best of our knowledge, none of the BCCs listed in Minn. R. 7050.0350 have field-measured BAFs below 1,000.

criterion (CC_{FR}) values for PFOA, PFHxS, PFHxA, PFBS, and PFBA, as well as a higher fish-tissue-based chronic criterion (CC_{FT}) for PFHxS (see Table 2.1).

Table 2.1 SSCs Derived by MPCA (2024f,g)

ssc	PFAS	MPCA	
		May 2024	December 2024
CC _{FR}	PFOS	0.027	0.027
	PFOA	0.0092	0.033
	PFHxS	0.0023	0.0087
	PFHxA	4,400	11,000
	PFBS	3,000	5,500
	PFBA	25,000	53,000
CC _{FT}	PFOS	0.021	0.021
	PFOA	0.00036	0.00036
	PFHxS	0.000043	0.000085

Notes:

 CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; CC_{FT} = Fish-Tissue-Based Chronic Criterion; MPCA = Minnesota Pollution Control Agency; PFAS = Per- and Polyfluorinated Substance; PFBA = Perfluorobutanoic Acid; PFBS = Perfluorobutane Sulfonic Acid; PFHxA = Perfluorohexanoic Acid; PFHxS = Perfluorohexane Sulfonic Acid; PFOA = Perfluorooctanoic Acid; PFOS = Perfluorooctane Sulfonic Acid; SSC(s) = Site-Specific Criterion (Criteria).

Given that MPCA (2024f) continues to rely on unsupported toxicological values and exposure parameters, the sensitivity analysis that was included in our prior comments remains relevant. It remains clear from our prior sensitivity analysis that reliance on a more reasonable set of alternate parameters results in substantially different criteria (See "Alternate RfD or CSF" and "Alternate FCR" in Tables 3.2a and 3.2b in Attachment 1). Overall, MPCA's continued and consistent selection of overly conservative toxicological values and exposure parameters has a substantial compounding effect, resulting in overly conservative SSCs that are not site-specific and inconsistent with similar values developed by other regulatory entities.

References

3M Co. 2025. "3M Review of MPCA's Data, Calculations and Site-Specific Water Quality Criteria for PFAS, River Miles 812-820." 15p., January 27.

Minnesota Dept. of Health (MDH). 2024a. "Health Based Guidance for Water, Toxicological Summary for: Perfluorooctane sulfonate (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 16p., February.

Minnesota Dept. of Health (MDH). 2024b. "Health Based Guidance for Water, Toxicological Summary for: Perfluorooctanoate (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 16p., January.

Minnesota Dept. of Health (MDH); University of Illinois at Chicago (UIC). 2017. "Technical Report: Fish are Important for Superior Health (FISH) Project." Environmental Health Division, 34p., May.

Minnesota Pollution Control Agency (MPCA). 2017. "Human Health-based Water Quality Standards Technical Support Document: Water Quality Standard Amendments – Minn. R. chs. 7050 and 7052 (Final)." 181p., June.

Minnesota Pollution Control Agency (MPCA). 2020. "Human health-based criteria and standards." Minn. R. 7050.0219. 14p. Accessed on May 20, 2024 at https://www.revisor.mn.gov/rules/7050.0219/

Minnesota Pollution Control Agency (MPCA). 2024a. "National Pollutant Discharge Elimination System/State Disposal System Permit (MN0001449) for 3M Cottage Grove Center, Unnamed creek - Class 2Bg, 3, 4A, 4B, 5, 6 water, Cottage Grove, Washington County (Draft)." Industrial Division, Water and Mining Section. Submitted to 3M Chemical Operations LLC. 415p.

Minnesota Pollution Control Agency (MPCA). 2024b. "Public Notice of intent to reissue, Wastewater Permit and TMDL Wasteload Allocation: Wastewater Permit MN0001449, 3M Chemical Operations' Cottage Grove Facility, 10746 Innovation Rd, Cottage Grove, MN 55016-4600." Industrial Division, 7p.

Minnesota Pollution Control Agency (MPCA). 2024c. "Public Notice of intent to reissue, Wastewater Permit and TMDL Wasteload Allocation: Wastewater Permit MN0001449, 3M Chemical Operations' Cottage Grove Facility, 10746 Innovation Rd, Cottage Grove, MN 55016-4600." Industrial Division, 8p., December.

Minnesota Pollution Control Agency (MPCA). 2024d. "National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit Program Fact Sheet: Permit Reissuance (MN0001449) for 3M Cottage Grove Center, Unnamed creek to the Mississippi River - Class 2Bg, 3, 4A, 4B, 5, 6 waters, Cottage Grove, MN." Industrial Division, 156p., December.

Minnesota Pollution Control Agency (MPCA). 2024e. "National Pollutant Discharge Elimination System/State Disposal System Permit (MN0001449) for 3M Cottage Grove Center, Unnamed creek - Class 2Bg, 3, 4A, 4B, 5, 6 water, Cottage Grove, Washington County (Draft)." Industrial Division, Water and Mining Section. Submitted to 3M Chemical Operations LLC. 441p.

Minnesota Pollution Control Agency (MPCA). 2024f. "Human Health Protective Water Quality Criteria for Per- and Polyfluoroalkyl Substances (PFAS) in Mississippi River, Miles 820 to 812." wq-s6-69e, 50p., December.

Minnesota Pollution Control Agency (MPCA). 2024g. "Human Health Protective Water Quality Criteria for Per- and Polyfluoroalkyl Substances (PFAS) in Mississippi River, Miles 820 to 812." wq-s6-69a, 45p., May.

Minnesota Pollution Control Agency (MPCA). 2024h. "Lake Superior Basin Water Standards." Minn. R. 7052. 60p. Accessed on August 12, 2024 at https://www.revisor.mn.gov/rules/7052/

Prueitt, R; Verslycke, T. [Gradient]. 2024. "Expert Report of Robyn Prueitt, Ph.D., DABT, and Tim Verslycke, Ph.D., Related to Reissuance of the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit MN0001449 for the 3M Cottage Grove Center Facility in Cottage Grove, Minnesota." Report to 3M Chemical Operations LLC. 77p., August 27.

US EPA. 2006. "On the Computation of a 95% Upper Confidence Limit of the Unknown Population Mean Based Upon Data Sets with Below Detection Limit Observations." EPA/600/R-06/022, 141p., March.

US EPA. 2014. "Estimated Fish Consumption Rates for the U.S. Population and Selected Subpopulations (NHANES 2003-2010) (Final Report)." EPA-820-R-14-002, 110p., April.

US EPA. 2024a. "Human Health Toxicity Assessment for Perfluorooctane Sulfonic Acid (PFOS) and Related Salts (Final)." Health and Ecological Criteria Division, Office of Science and Technology, Office of Water, EPA Document No. 815R24007, 514p., April. Accessed on May 29, 2024 at https://www.epa.gov/system/files/documents/2024-04/main_final-toxicity-assessment-for-pfos_2024-04-09-refs-formatted_508c.pdf.

US EPA. 2024b. "Human Health Toxicity Assessment for Perfluorooctanoic Acid (PFOA) and Related Salts (Final)." Health and Ecological Criteria Division, Office of Science and Technology, Office of Water, EPA Document No. 815R24006. 556p., April. Accessed on May 29, 2024 at https://www.epa.gov/system/files/documents/2024-04/main_final-toxicity-assessment-for-pfoa_2024-04-09-refs-formatted.pdf.

US EPA Region III. 1991. "Regional Guidance on Handling Chemical Concentration Data Near the Detection Limit in Risk Assessments (Interim Final)." November 4. Accessed on May 22, 2019 at https://www.epa.gov/risk/regional-guidance-handling-chemical-concentration-data-near-detection-limit-risk-assessments

Weston Solutions. 2023. "Appendix A: Summary of PFAS and TOC Analytical Results." 168p., June 29.

Attachment 1

Expert Report of Robyn Prueitt, Ph.D., DABT, and Tim Verslycke, Ph.D., Related to Reissuance of the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit MN0001449 for the 3M Cottage Grove Center Facility in Cottage Grove, Minnesota

Expert Report of Robyn Prueitt, Ph.D., DABT, and Tim Verslycke, Ph.D., Related to Reissuance of the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit MN0001449 for the 3M Cottage Grove Center Facility in Cottage Grove, Minnesota

Prepared by

Robyn Prueitt, Ph.D., DABT

Tim Verslycke, Ph.D.

August 27, 2024



www.gradientcorp.com One Beacon Street, 17th Floor Boston, MA 02108 617-395-5000

Table of Contents

	<u>Page</u>
1 Introd 1.1 1.2	duction
1.3	1.2.2 Dr. Tim Verslycke
2 Sumr	mary of Opinions3
3 Basis 3.1	for Opinions
3.2	MPCA relies on an interim fish consumption rate (FCR) that overestimates fish consumption, is not representative of site-specific conditions, and is higher than what is used by other states and US EPA
3.3	MPCA does not provide the necessary underlying information to allow for ar independent evaluation and verification of its analyses. A number of calculation discrepancies and errors were identified where data verification was possible 11
3.4	MPCA's approach to developing fish-tissue-based criteria for perfluorooctanoic acid (PFOA) and perfluorohexane sulfonic acid (PFHxS) is inconsistent with its owr guidance and best available science
3.5	MPCA's methodology for calculating fish bioaccumulation factors (BAFs) is technically flawed and is inconsistent with US EPA guidance
3.6	MPCA's consistent reliance on unsupported toxicological values and exposure parameters, when considered in combination, results in site-specific criteria (SSCs that are not site-specific, and are inconsistent with similar values developed by other regulatory entities and with MPCA's own regulatory processes to protect the designated uses of the Mississippi River Miles 820 to 812
References	28
Attachment	• • • • • • • • • • • • • • • • • • • •

List of Tables

Table 3.1	Sensitivity Analysis of the SSC Calculation Using Alternate Input Parameters
Table 3.2a	Sensitivity Analysis of the Surface Water Chronic Criteria Calculation Using Alternate Input Parameters
Table 3.2b	Sensitivity Analysis of the Fish Tissue Chronic Criteria Calculation Using Alternate Input Parameters

List of Figures

Figure 3.1	Sensitivity Analysis of PFOS CC _{FR} Using Alternate Input Parameters
Figure 3.2	Sensitivity Analysis of PFOA CC _{FR} Using Alternate Input Parameters
Figure 3.3	Sensitivity Analysis of PFHxS CC _{FR} Using Alternate Input Parameters
Figure 3.4	Sensitivity Analysis of PFHxS CC _{FR} Using Alternate RfDs
Figure 3.5	Sensitivity Analysis of PFHxA CC _{FR} Using Alternate Input Parameters
Figure 3.6	Sensitivity Analysis of PFBS CC _{FR} Using Alternate Input Parameters
Figure 3.7	Sensitivity Analysis of PFBA CC _{FR} Using Alternate Input Parameters
Figure 3.8	Sensitivity Analysis of PFOS CC _{FT} Using Alternate Input Parameters
Figure 3.9	Sensitivity Analysis of PFOA CC _{FT} Using Alternate Input Parameters
Figure 3.10	Sensitivity Analysis of PFHxS CC _{FT} Using Alternate Input Parameters
Figure 3.11	Sensitivity Analysis of PFHxS CC _{FT} Using Alternate RfDs

Abbreviations

3M Company

AF_{lifetime} Lifetime Adjustment Factor

ATSDR Agency for Toxic Substances and Disease Registry

BAF Bioaccumulation Factor

BCC Bioaccumulative Chemical of Concern

CC_{FR} Fish Consumption and Recreation Use Class Chronic Criterion

CC_{FT} Fish-Tissue-Based Chronic Criterion

CSF Cancer Slope Factor
DL Detection Limit

EGLE Michigan Department of Environment, Great Lakes, and Energy

FCR Fish Consumption Rate

FISH Fish Are Important for Superior Health

FLDEP Florida Department of Environmental Protection

GLCFCA Great Lakes Consortium for Fish Consumption Advisories
IBERA International Board of Environmental Risk Assessors

IRIS Integrated Risk Information Systems

ITRC Interstate Technology and Regulatory Council

MCLG Maximum Contaminant Level Goal
MDH Minnesota Department of Health
MPCA Minnesota Pollution Control Agency

MRL Minimal Risk Level

NHANES National Health and Nutrition Examination Survey NPDES National Pollutant Discharge Elimination System

PFAS Per- and Polyfluorinated Substance

PFBA Perfluorobutanoic Acid

PFBS Perfluorobutane Sulfonic Acid

PFHxA Perfluorohexanoic Acid

PFHxS Perfluorohexane Sulfonic Acid

PFOA Perfluorooctanoic Acid

PFOS Perfluorooctane Sulfonic Acid

POD Point of Departure

POD_{HED} Point of Departure Human Equivalent Dose

RfD Reference Dose

ROS Regression on Order Statistics
RSC Relative Source Contribution
RSL Regional Screening Level
SDS State Disposal System

SETAC Society of Environmental Toxicology and Chemistry

SSC Site-Specific Criterion
SSCs Site-Specific Criteria
UF Uncertainty Factor

US EPA United States Environmental Protection Agency

WCBA Women of Child-Bearing Age

GRADIENT III

WDNR Wisconsin Department of Natural Resources WHOI Woods Hole Oceanographic Institution

WQC Water Quality Criterion WQCs Water Quality Criteria

GRADIENT iv

1 Introduction

1.1 Scope of Report

Drs. Robyn Prueitt and Tim Verslycke were retained by 3M Chemical Operations LLC (3M) to provide technical expert services related to the reissuance of its National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit for the 3M Cottage Grove Center facility located in Cottage Grove, Minnesota. Specifically, Dr. Prueitt was asked to provide expert toxicology support and Dr. Verslycke was asked to provide expert ecotoxicology support related to evaluating the proposed effluent limits for per- and polyfluorinated substances (PFAS) in a draft permit published by the Minnesota Pollution Control Agency (MPCA) on July 1, 2024 (MPCA, 2024a,b).

The qualifications of Drs. Robyn Prueitt and Tim Verslycke are presented in Section 1.2. The documents and sources relied upon are discussed in Section 1.3, with a full list provided in the References section at the end of this report. Gradient is compensated at the rate of \$475/hour for the expert services of Drs. Robyn Prueitt and Tim Verslycke.

1.2 Professional Qualifications

1.2.1 Dr. Robyn Prueitt

I am a board-certified toxicologist with expertise in toxicology, carcinogenesis, and human health risk assessment. I received a BS degree in biology from Pacific Lutheran University and a Ph.D. in cell and molecular biology/human genetics from the University of Texas Southwestern Medical Center at Dallas. I was a postdoctoral fellow at the National Cancer Institute, where I managed multiple projects related to breast and prostate carcinogenesis. I was also a staff scientist at Fred Hutchinson Cancer Research Center, where I studied prostate tumor biology and biomarkers.

I joined Gradient in 2007, and my work has focused on evaluating human, experimental animal, and *in vitro* toxicology studies for health risk assessments of cancer and non-cancer endpoints, with special emphasis on mechanistic and weight-of-evidence evaluations of health risk and causation for chemical exposures. I have conducted some of this work in the context of regulatory comment and/or testimony to various state, national, and international regulatory agencies. I have previously provided toxicology and human health risk assessment support to 3M in several litigation matters involving PFAS and have testified on behalf of 3M to an Illinois State regulatory agency at a public hearing on proposed groundwater standards for PFAS.

I have been active in the Society of Toxicology since 2008. I have published multiple articles on toxicology, carcinogenesis, and risk assessment in peer-reviewed journals, books, and meeting proceedings, and I have been a peer reviewer for multiple toxicology journals. My *curriculum vitae* is provided as Attachment 1.

1.2.2 Dr. Tim Verslycke

I am an ecotoxicologist with 20 years of combined consulting and academic research experience in ecological risk assessment. I received a B.A. and an M.S. in bioscience engineering and subsequently a

Ph.D. in applied biological sciences from Ghent University (Ghent, Belgium) in one of the world's premier laboratories for ecotoxicology and risk assessment. Thereafter, I was a postdoctoral scholar in a toxicology laboratory at the Woods Hole Oceanographic Institution (WHOI), under WHOI Ocean Life Institute and Belgian American Educational Foundation scholarships and competitively funded government grants. Until 2019, I was appointed as a visiting investigator in the Biology Department at WHOI.

I have worked at Gradient since 2007. Gradient is an environmental and risk science consulting firm specializing in contaminant fate and transport analyses, human health and ecological risk assessment, and environmental chemistry. I am a principal at Gradient and my consulting practice consists of ecological risk assessments of contaminated sites, environmental safety assessments of new and existing products, and regulatory ecotoxicity testing. I have served, in an advisory capacity, to a wide range of governmental and non-profit organizations on issues related to environmental toxicology and ecological risk assessment. I have been active in the Society of Environmental Toxicology and Chemistry (SETAC) for many years and served as president of the North Atlantic Chapter. I am a founding member and currently serve as president of the International Board of Environmental Risk Assessors (IBERA). IBERA established the first international certification program in ecological risk assessment. I also served on the United States Environmental Protection Agency's (US EPA) Board of Scientific Counselors Safe and Sustainable Water Resources Subcommittee, which provides advice and recommendations to US EPA's Office of Research and Development on technical and management issues of its research programs. I have previously provided expert opinions regarding the scientific state of knowledge of PFAS ecotoxicity and bioaccumulation in organisms on behalf of the 3M Company in a number of cases.

I have published over 40 articles on environmental toxicology and risk assessment in peer-reviewed journals, books, and meeting proceedings. I have been a peer reviewer for multiple journals in the environmental sciences field. My *curriculum vitae* is provided as Attachment 2.

1.3 Information Sources

Data and information sources used to develop this report include academic journal articles, regulatory documents, textbooks, technical reports, publicly accessible databases, government studies and reports, and materials provided to us by counsel. Data and information sources that we relied upon in preparing this report are provided in the References section.

The types of information relied upon in this report are customarily reviewed, considered, and relied upon by experts in our field. The information we reviewed for this matter, in addition to our education, training, and professional experience, have allowed us to provide the opinions herein with a reasonable degree of scientific certainty. Upon review of additional information that may become available to us, we reserve the right to modify or supplement our opinions accordingly.

2 Summary of Opinions

Our opinions are based on the information sources we reviewed, in addition to our education, training, research, and professional experience in toxicology, ecotoxicology and risk assessment. Section 3 of this report provides the basis for these opinions. Date and information sources that were relied upon in preparing this expert report are provided in the References section. We reserve the right to supplement or amend our opinions should new facts or information be made known to us.

Dr. Prueitt offers the following opinion with a reasonable degree of scientific certainty:

1. MPCA's use of toxicological values is inconsistent with Minnesota's water quality rules and previous approaches used by MPCA.

Dr. Verslycke offers the following opinions with a reasonable degree of scientific certainty:

- 2. MPCA relies on an interim fish consumption rate (FCR) that overestimates fish consumption, is not representative of site-specific conditions, and is higher than what is used by other states and US EPA.
- 3. MPCA does not provide the necessary underlying information to allow for an independent evaluation and verification of its analyses. A number of calculation discrepancies and errors were identified where data verification was possible.
- 4. MPCA's approach to developing fish-tissue-based criteria for perfluorooctanoic acid (PFOA) and perfluorohexane sulfonic acid (PFHxS) is inconsistent with its own guidance and best available science.
- 5. MPCA's methodology for calculating fish bioaccumulation factors (BAF) is technically flawed and is inconsistent with US EPA guidance.

Drs. Prueitt and Verslycke offer the following joint opinion with a reasonable degree of scientific certainty:

6. MPCA's consistent reliance on unsupported toxicological values and exposure parameters, when considered in combination, results in site-specific criteria (SSCs) that are not site-specific, and are inconsistent with similar values developed by other regulatory entities and with MPCA's own regulatory processes to protect the designated uses of the Mississippi River Miles 820 to 812.

3 Basis for Opinions

This section provides the basis for the opinions summarized in Section 2. We evaluated various inputs and assumptions that MPCA relied upon to derive SSCs using the algorithms described below, as described in its May 2024 criteria development report (MPCA, 2024c). Dr. Prueitt evaluated the toxicological values and health endpoints (Section 3.1). Dr. Verslycke evaluated the FCR (Section 3.2), the adequacy of the provided information to be able to verify MPCA's analyses (Section 3.3), the fish-tissue-based criteria (Section 3.4), and the fish BAFs (Section 3.5). Drs. Prueitt and Verslycke jointly evaluated the cumulative impact of MPCA's reliance on unsupported assumptions on the SSCs MPCA developed (Section 3.6). As described further in the sections below, MPCA's derivation of SSCs is based on analyses that cannot be fully verified, contain calculation and transcription errors where verification was possible, and inappropriately compound overly conservative assumptions. This results in SCCs that are inconsistent with the prescribed regulatory process that was designed to ensure adequate water quality to protect the designated uses of the Mississippi River Miles 820 to 812

MPCA derived site-specific human health protective water quality criteria (WQCs) for six PFAS in a report dated May 2024: perfluorobutanoic acid (PFBA), perfluorobutane sulfonic acid (PFBS), perfluorohexanoic acid (PFHxA), perfluorohexanoic acid (PFHxA), perfluorooctanoic acid (PFOA), and perfluorooctane sulfonic acid (PFOS) (see Table 1-1 in MPCA, 2024c). The "Site" is defined by MPCA as the Mississippi River main channel between river miles 820 and 812. This area is immediately adjacent to and downstream of 3M's Cottage Grove manufacturing facility (see Figure 1-1 in MPCA, 2024c).

As described in Section 3 of MPCA (2024c), MPCA states that the SSCs were derived for the Mississippi River Miles 820 to 812 to protect humans from potential adverse effects of eating fish and other edible aquatic organisms and incidental ingestion of water while recreating. The algorithms that MPCA used to derive chronic criteria for noncarcinogens (all six PFAS) and carcinogens (only PFOS and PFOA) were taken from Minn. R. 7050.0219 Subp.14 and Subp.15 (MPCA, 2020a), as presented below:

Surface water-based chronic criteria for noncarcinogenic chemicals:

$$CC_{FR} = \frac{RfD_{Chronic} (mg/kg-d) \times RSC (unitless) \times 1,000,000 \text{ ng/mg}}{\{IWR_{Chronic} (L/kg-d) + FCR_{Adult} (kg/kg-d)[(0.24 \times BAF_{TL3} (L/kg)) + (0.76 \times BAF_{TL4} (L/kg)]\}}$$

Surface water-based chronic criteria for linear carcinogenic chemicals with lifetime adjustment factors (AF_{lifetime}):

$$CC_{FR} = \frac{CR (1 \times 10^{-5})}{CSF (mg/kg-d)^{-1} \times AF_{Lifetime}} \times \frac{1,000,000 \text{ ng/mg}}{\{IWR_{Chronic} (L/kg-d) + FCR_{Adult} (kg/kg-d)[(0.24 \times BAF_{TL3} (L/kg)) + (0.76 \times BAF_{TL4} (L/kg))]\}}$$

Fish tissue-based chronic criteria for noncarcinogenic chemicals:

Fish tissue-based chronic criteria for linear carcinogenic chemicals with AF_{lifetime}:

$$CC_{FT} = \frac{CR (1 \times 10^{-5})}{CSF (mg/kg-d)^{-1} \times AF_{Lifetime}} \times \frac{1,000,000 \text{ ng/mg}}{FCR_{Adult} (kg/kg-d)}$$

where:

1,000,000 ng/mg = Conversion Factor

 $AF_{Lifetime} = Lifetime Adjustment factor (unitless)$

BAF_{TL3} = Final Bioaccumulation Factor (BAF) for Trophic Level 3 Fish in L/kg; Accounts for 24% of Fish Consumed

BAF_{TL4} = Final BAF for Trophic Level 4 Fish in L/kg; Accounts for 76% of Fish Consumed

 CC_{FR} = Fish Consumption and Recreation Chronic Criterion in Class 2B Waters (ng/L)

 CC_{FT} = Fish Consumption and Recreation Chronic Criterion Applied for Bioaccumulative Chemicals of Concern (BCC) in all Class 2 Waters (ng/g)

CR = Cancer Risk Level or an Additional Excess Cancer Risk Equal to 1×10^{-5}

 $CSF = Cancer Slope Factor in (mg/kg-d)^{-1}$

 $FCR_{Adult} = 0.00094 \text{ kg/kg-d}$; MPCA Interim Fish Consumption Rate for Women of Childbearing Age

IWR_{Chronic} = 0.0013 L/kg-d; Assumed Incidental Water Intake Rate Based on Minimum Chronic Duration

RfD_{Chronic} = Reference Dose for Chronic Duration (mg/kg-d)

RSC = Relative Source Contribution (unitless)

The unsupported assumptions used by MPCA as inputs to these various algorithms and the cumulative impact of those assumptions on the SSCs is the basis of our opinions, summarized in Section 2 and detailed below.

3.1 MPCA's use of toxicological values is inconsistent with Minnesota's water quality rules and previous approaches used by MPCA.

The SSC for six PFAS for the Mississippi River Miles 820 to 812 that were developed by MPCA (2024c) used toxicological values from US EPA that are not consistent with Minnesota's water quality rules (MPCA, 2017, 2020a) and that differ from the toxicological values that MPCA previously used for developing WQCs for these same PFAS (MPCA, 2020b, 2023a). These toxicological values are reference doses (RfDs) or cancer slope factors (CSFs). An RfD is defined by Minnesota rules as "an estimate of a dose for a given duration to the human population, including susceptible subgroups such as infants, that is likely to be without an appreciable risk of adverse effects during a lifetime" (MPCA, 2017). A CSF, or cancer potency factor, is "an upper bound value for the number of cases of cancer estimated from a lifetime of exposure to a chemical" (MPCA, 2017). The RfD and CSF are determinative factors in the algorithms specified by Minnesota's water quality rules for developing site-specific WQCs (see algorithms for SSC above in Section 3).

According to the Technical Support Document for amendments to methods regarding human health-based water quality standards in Minnesota's water quality rules (Minn. R. chs. 7050 and 7052) (MPCA, 2017), and consistent with Minn. R. 7050.0219, Subp.2 (MPCA, 2020a), SSCs are to be based on RfDs and CSFs from Minnesota Department of Health's (MDH's) health risk limits or health-based guidance values for drinking water. While the rules indicate that these toxicological values can be RfDs and CSFs from US

EPA, such values can only be used after evaluation and completion of any needed modifications by MDH (MPCA, 2017). MDH's methodology for developing toxicological values for PFAS has generally differed from that of US EPA, as MDH has had a different understanding of the toxicokinetics (*i.e.*, the absorption, distribution, metabolism, and excretion) of PFAS in the body and thus has used different toxicokinetic model parameters to convert serum levels of PFAS to human equivalent doses compared to US EPA.

MPCA based its 2020 WQC for PFOS (MPCA, 2020b) and its 2023 WQCs for PFOA, PFHxS, PFHxA, PFBS, and PFBA (MPCA, 2023a) on RfDs developed by MDH, which is consistent with Minnesota's water quality rules. By contrast, and without an explanation, for the SSC for Mississippi River Miles 820 to 812 MPCA (2024c) used RfDs and CSFs from US EPA human health toxicity assessments and Integrated Risk Information Systems (IRIS) toxicological reviews that differ from the most recently developed RfDs and CSFs for the six PFAS by MDH.

3.1.1 The RfD and CSF used by MPCA for the PFOS SSC are inconsistent with those developed by MDH and with Minnesota's water quality rules.

MPCA (2024c) used an RfD of 1×10^{-7} mg/kg-d and a CSF of 39.5 per mg/kg-d from the US EPA Final Human Health Toxicity Assessment for PFOS (US EPA, 2024a). While MDH (2024a) developed an RfD for PFOS based on the same underlying health effect from the same study relied upon by US EPA (2024a), the MDH RfD was derived by dividing the point of departure (POD) of 7.7 ng/mL in serum by an uncertainty factor (UF) of 3, whereas US EPA (2024a) first converted the 7.7 ng/mL serum concentration to a POD human equivalent dose (POD_{HED}) and divided the POD_{HED} by a UF of 10. MDH (2024a) did not calculate a POD_{HED} in its derivation of the PFOS RfD; instead, MDH (2024a) represented the RfD as a serum concentration, stating that serum concentrations are the most appropriate dose metric for PFOS given its "highly bioaccumulative nature" (MDH, 2024a). Even if MDH had calculated a POD_{HED} for PFOS, it would differ from US EPA's POD_{HED} because MDH uses a different toxicokinetic model than US EPA to calculate POD_{HED} values for PFOS. If MDH (2024a) had calculated a POD_{HED} value using its toxicokinetic model for PFOS, this value would be 3×10^{-6} mg/kg-d; dividing this value by a UF of 3 would yield a PFOS RfD of 1×10^{-6} mg/kg-d. Thus, the US EPA (2024a) RfD used for the PFOS SSC for Mississippi River Miles 820 to 812 is different from the RfD developed by MDH (2024a), based on the application of different toxicokinetic models for PFOS and different UF values.

MDH (2024a) used the same POD (19.8 mg/L in serum) as US EPA (2024a) to develop its CSF for PFOS, but the MDH CSF (13 per mg/kg-d) differs from the US EPA CSF (39.5 per mg/kg-d) because it was converted from a serum concentration to a dose in mg/kg-d using a different dosimetric adjustment factor for PFOS. Thus, the US EPA (2024a) CSF used for the PFOS SSC for Mississippi River Miles 820 to 812 is different from the CSF developed by MDH (2024a). However, the SSC for PFOS for Mississippi River Miles 820 to 812 is ultimately based on the use of the RfD as the toxicological value because MPCA stated that the non-carcinogenic SSC was lower than the carcinogenic SSC that was based on the use of the CSF for PFOS (MPCA, 2024c).

For its 2020 WQC for PFOS that is not specific to Mississippi River Miles 820 to 812, MPCA (2020b) used an RfD of 3.1×10^{-6} mg/kg-d, as developed by MDH (2019). This RfD is also different from the US EPA RfD MPCA used for the SSC for Mississippi River Miles 820 to 812.

The use of the RfD and CSF developed by MDH, rather than the values developed by US EPA, would result in a SSC for PFOS that is consistent with Minnesota's water quality rules and with other WQC for

GRADIENT 6

r082924z

¹ The POD_{HED} is calculated by multiplying the POD of 0.0077 mg/L by a dosimetric adjustment factor that is equivalent to the clearance rate of PFOS (MDH, 2024a). Clearance rate = Volume of distribution (L/kg) × (Ln2/half-life, days) = 0.56 L/kg × (0.693/996 days) = 0.00039 L/kg-d. POD_{HED} = 0.0077 mg/L × 0.000039 L/kg-d = 3 × 10⁻⁶ mg/kg-d.

PFOS developed by MPCA. MPCA (2024c) offered no explanation for not using the RfD and CSF developed by MDH (2024a) so we are unable at this time to comment further on MPCA's choice of these toxicological values.

3.1.2 The RfD and CSF used by MPCA for the PFOA SSC near Cottage Grove are inconsistent with those developed by MDH and with Minnesota's water quality rules.

MPCA (2024c) used an RfD of 3×10^{-8} mg/kg-d and a CSF of 29,300 per mg/kg-d derived from the US EPA Final Human Health Toxicity Assessment for PFOA (US EPA, 2024b). MDH (2024b) developed an RfD for PFOA of 2.8 ng/mL (serum concentration), which is based on a different underlying health effect and study than that used by US EPA for its RfD. The US EPA RfD is equivalent to a serum concentration RfD of 0.2 ng/mL. Thus, the US EPA (2024b) RfD used for the PFOA SSC for Mississippi River Miles 820 to 812 is different from the RfD developed by MDH (2024b).

MDH (2024b) used the US EPA (2024b) CSF as a basis to develop a CSF for PFOA of 12,600 per mg/kg-d, which differs from the US EPA CSF of 29,300 per mg/kg-d because it was converted from a serum concentration to a dose in mg/kg-d using a different dosimetric adjustment factor for PFOA. Thus, the US EPA (2024b) CSF used for the PFOA SSC for Mississippi River Miles 820 to 812 is different from the CSF developed by MDH (2024b). The SSC for PFOA for Mississippi River Miles 820 to 812 is based on the use of this CSF because MPCA stated that the carcinogenic SSC was lower than the non-carcinogenic SSC that was based on the use of the RfD for PFOA (MPCA, 2024c).

For its 2023 WQC for PFOA that is not specific to Mississippi River Miles 820 to 812, MPCA (2023a) used an RfD of 1.8×10^{-5} mg/kg-d (equivalent to a serum concentration RfD of 130 ng/mL) developed by MDH. This is also different from the US EPA RfD used for the SSC for Mississippi River Miles 820 to 812.

MPCA (2024c) offered no explanation for not using the RfD and CSF developed by MDH (2024b) so we are unable at this time to comment further on MPCA's choice of these toxicological values.

3.1.3 The RfD used by MPCA for the PFHxS SSC is inconsistent with the RfD developed by MDH and with Minnesota's water quality rules.

MPCA (2024c) used an RfD of 2×10^{-10} mg/kg-d from the External Review Draft of the IRIS Toxicological Review of PFHxS (US EPA, 2023a). The value for this RfD is incorrect and appears to be derived from an erroneous value listed in Table ES-1 in the Executive Summary of the US EPA draft document. The actual RfD value from US EPA (2023a) for PFHxS is 4×10^{-10} mg/kg-d. MPCA used the incorrect, lower RfD value of 2×10^{-10} mg/kg-d rather than the actual draft RfD value of 4×10^{-10} mg/kg-d. In addition, the RfD from US EPA (2023a) is a draft value that has not undergone external peer review and has not been finalized by US EPA; as such, it is not a reliable basis for use in developing WQCs. In fact, US EPA did not even use this draft RfD value as a basis for its most recent (May 2024) regional screening levels (RSLs) for PFHxS (US EPA, 2024c) or for its recent development of the maximum contaminant level goal (MCLG) for PFHxS in drinking water (US EPA, 2024d). Instead, US EPA used the minimal risk level (MRL) of 2×10^{-6} mg/kg-d for PFHxS derived by the Agency for Toxic Substances and Disease Registry (ATSDR, 2021) as the RfD for use in deriving its RSLs and the MCLG for PFHxS. Thus, the draft US EPA (2023a)

GRADIENT 7

r082924z

 $^{^2}$ The intermediate oral MRL for PFHxS developed by ATSDR (2021) is 2×10^{-5} mg/kg-d and is based on an underlying toxicity study with a subchronic, and not chronic, duration of exposure. While US EPA (2024c) used the 2×10^{-5} mg/kg-d MRL as a basis for its RSLs for PFHxS, US EPA (2024d) divided the MRL by an additional UF of 10 to account for the subchronic exposure duration of the underlying study when applying the MRL to the development of a MCLG for PFHxS, yielding an RfD of 2×10^{-6}

RfD that MPCA (2024c) used for the PFHxS SSC for Mississippi River Miles 820 to 812 is 10,000-fold lower than the PFHxS RfD used by US EPA to derive the MCLG for PFHxS in drinking water (US EPA, 2024d).

MPCA's use of the RfD of 2×10^{-10} mg/kg-d in calculating the SSC for PFHxS is also inconsistent with the RfD for PFHxS of 9.7×10^{-6} mg/kg-d that MDH developed for its most recent health-based guidance in drinking water (MDH, 2023a). Moreover, for its 2023 WQC for PFHxS that is not specific to Mississippi River Miles 820 to 812, MPCA (2023a) also used the MDH RfD of 9.7×10^{-6} mg/kg-d. Thus, the draft US EPA (2023a) RfD that MPCA (2024c) used for the PFHxS SSC for Mississippi River Miles 820 to 812 is nearly 10,000 fold lower than the PFHxS RfD developed by MDH (2023a).

The use of the RfD developed by MDH, rather than the draft value developed by US EPA, would result in a dramatically higher SSC for PFHxS that is consistent with Minnesota's water quality rules and with other WQC for PFHxS developed by MPCA. MPCA (2024c) offered no explanation as to how the use of US EPA's draft RfD for PFHxS is more appropriate than the RfD recently developed by MDH (2023a) or is consistent with Minnesota WQC regulations. As a result, we are unable at this time to comment further on MPCA's choice of RfD.

3.1.4 The RfD used by MPCA for the PFHxA SSC is inconsistent with the RfD developed by MDH and with Minnesota's water quality rules.

MPCA (2024c) used an RfD of 5×10^{-4} mg/kg-d from the US EPA IRIS Toxicological Review of PFHxA (US EPA, 2023b). MDH developed an RfD for PFHxA of 3.2×10^{-4} mg/kg-d that was used in its health-based guidance in drinking water (MDH, 2023b). For its 2023 WQC for PFHxA that is not specific to Mississippi River Miles 820 to 812, MPCA (2023a) used the 3.2×10^{-4} mg/kg-d RfD that was developed by MDH (2023b).

The RfD used by MPCA (2024c) for the PFHxA SSC for Mississippi River Miles 820 to 812 is not consistent with Minnesota's water quality rules or with other WQC for PFHxA developed by MPCA. MPCA (2024c) offered no explanation for not using the RfD developed by MDH (2023b) so we are unable at this time to comment further on MPCA's choice of RfD.

3.1.5 The RfD used by MPCA for the PFBS SSC is inconsistent with the RfD developed by MDH and with Minnesota's water quality rules.

MPCA (2024c) used an RfD of 3×10^{-4} mg/kg-d from the US EPA Human Health Toxicity Values for PFBS (US EPA, 2021). MDH developed an RfD for PFBS of 8.4×10^{-5} mg/kg-d that was used in its health-based guidance for drinking water (MDH, 2023c). For its 2023 WQC for PFBS that is not specific to Mississippi River Miles 820 to 812, MPCA (2023a) used the 8.4×10^{-5} mg/kg-d RfD developed by MDH (2023c).

The RfD used by MPCA (2024c) for the PFBS SSC for Mississippi River Miles 820 to 812 is not consistent with Minnesota's water quality rules or with other site-specific WQCs developed by MPCA. MPCA (2024c) offered no explanation for not using the RfD developed by MDH (2023c) so we are unable at this time to comment further on MPCA's choice of RfD.

GRADIENT 8

r082924z

mg/kg-d for use in calculating the PFHxS MCLG. It is appropriate to apply the additional UF for exposure duration in this case because MCLGs (as well as surface water SSCs developed according to Minnesota regulations) incorporate chronic RfDs, not subchronic RfDs, in their derivation (see algorithms for SSC above in Section 3).

3.1.6 The RfD used by MPCA for the PFBA SSC is inconsistent with the RfD developed by MDH and with Minnesota's water quality rules.

MPCA (2024c) used an RfD of 1×10^{-3} mg/kg-d from the US EPA IRIS Toxicological Review of PFBA (US EPA, 2022a). MDH developed an RfD for PFBA of 3.8×10^{-3} mg/kg-d that was used in its health-based guidance in drinking water (MDH, 2018). Thus, the US EPA (2022a) RfD used for the PFBA SSC for Mississippi River Miles 820 to 812 is different from the RfD developed by MDH (2018). For its 2023 WQC for PFBA that is not specific to Mississippi River Miles 820 to 812, MPCA (2023a) used the 3.8×10^{-3} mg/kg-d RfD developed by MDH (2018).

The use of the RfD developed by MDH, rather than the value developed by US EPA, would result in a SSC for PFBA that is consistent with Minnesota's water quality rules and with other WQC for PFBA developed by MPCA. MPCA (2024c) offered no explanation for not using the RfD developed by MDH (2018) so we are unable at this time to comment further on MPCA's choice of RfD.

3.2 MPCA relies on an interim fish consumption rate (FCR) that overestimates fish consumption, is not representative of site-specific conditions, and is higher than what is used by other states and US EPA.

MPCA (2024c) used an interim FCR for women of child-bearing age (WCBA) of 66 g/d based on the MDH Fish are Important for Superior Health (FISH) survey of North Shore Minnesotans (MDH and UIC, 2017). MPCA (2024c) references a 2022 MPCA document, called "Interim fish consumption rate for women of childbearing age" for further detail on the derivation of this interim FCR (MPCA, 2022). In its 2022 document, MPCA states that the default FCR for adults in the Minnesota Rule chapters 7050 and 7052 is not appropriate given that PFOA and PFOS (and possibly other PFAS) have developmental health endpoints (MPCA, 2022). Instead, MPCA developed an interim FCR for WCBA of 66 g/d using what it calls "best available and reliable data" to meet its and US EPA's objectives for setting human health-protective WQCs. For the reasons detailed below, MPCA's interim FCR is not reflective of fish consumption patterns for the Mississippi River Miles 820 to 812, is not consistent with US EPA guidance on the development of WQCs, is greater than two-fold higher than Minnesota's default FCR, is substantially higher than FCRs developed by other states and US EPA, and hence is not based on the best available and reliable data:

- As cited by MPCA (2022), US EPA (2014) recommends that states develop WQCs that reflect the fish consumption patterns of the target population rather than using default values. Specifically, US EPA (2014) recommends using the following hierarchy of data sources to develop FCRs: (1) use local data; (2) use data reflecting similar geographical or population groups; (3) use data from national surveys; and (4) use US EPA's default FCR. MPCA's (2022) Table 1 describes information on fish consumption patterns from a range of regional and national surveys. Yet, inconsistent with US EPA's guidance, MPCA derived its interim FCR solely on the results of a 2017 survey of WCBA (ages 16 to 50) residing on the North Shore³ (MDH and UIC, 2017) and provides no discussion of how the fish consumption patterns and local conditions in the 2017 survey of North Shore Minnesotans reflect those in the Mississippi River Miles 820 to 812:
 - The fish species included in the MDH survey of North Shore Minnesotans (MDH and UIC, 2017) are not representative of the fish species likely to be present and consumed in the Mississippi River near the Cottage Grove facility. The MDH survey of North Shore Minnesotans lists the following fish/shellfish species in descending order of mean number of

-

³ The North Shore refers to the northern shore of Lake Superior in Minnesota.

meals consumed in the past 3 months as: tuna, canned; shellfish; salmon; lake trout; walleye; lake herring; whitefish, menominee; fish sticks/sandwiches; tuna steak; cod; tilapia; stream trout; other fish; northern pike; perch; bass; panfish and halibut (MDH and UIC, 2017, Table 4). Only three species (walleye, northern pike, and bass) that were reported as being consumed in lower relative amounts by North Shore Minnesotans in the 2017 survey are present in Mississippi River Miles 820 to 812 (Minnesota DNR, 2024a). The MDH and UIC (2017) survey reports fish caught from Lake Superior, which is a different watershed basin than the Mississippi River Miles 820 to 812, which is in the Upper Mississippi River basin (Minnesota DNR, 2024b).

- The MDH and UIC (2017) survey included questions pertaining to the consumption of store-bought and caught fish. Meals of fish that were caught comprised only 35 percent of total fish meals consumed by participants in the survey. The inclusion of purchased fish may have resulted in an overestimation of the FCR of the surveyed population. Further, MPCA applies the FCR from this survey to Mississippi River Miles 820 to 812 and incorrectly assumes that all consumed fish would be from the Mississippi River Miles 820 to 812.
- The MDH-surveyed population on the North Shore of Minnesota is not representative of the population that is expected to fish Mississippi River Miles 820 to 812 (MDH and UIC, 2017). A Great Lakes WCBA diary survey (Connelly *et al.*, 2019) is described as a relevant and reliable survey by MPCA (2022) in its development of an interim FCR for WCBA. This survey found women participating (95% Caucasian) consumed less than 30 g/d (20.7 g/d at the 90th percentile) of total freshwater fish based on the reported portion size. In comparison, the higher amount of fish eaten in the MDH and UIC (2017) survey is consistent with the fact that study participants include subpopulations of WCBA who may eat more fish and shellfish for subsistence or cultural reasons. MPCA does not discuss how the surveyed population in the MDH and UIC (2017) survey compares to the demographics of the target population that may consume fish caught in the Mississippi River near the Cottage Grove facility.
- MPCA's (2024c) interim FCR of 66 g/d is substantially higher than Minnesota's default FCR and FCRs developed by other states and US EPA:
 - MPCA's (2024c) interim FCR of 66 g/d is greater than two-fold higher than the default FCR described in Minnesota Rules 7050.0219 Subp.13 (MPCA, 2020a) (30 g/d).
 - Wisconsin and Michigan rely on default FCRs of 20 and 15 g/d, respectively, for use in their state-specific human health water quality guidelines based on an average freshwater fish FCR for sport anglers (Ruffle *et al.*, 2024).
 - GLCFCA (2019) assumes a FCR of 32 g/d.
 - US EPA (2014) derived a default FCR of 22 g/d at the 90th percentile for the US adult population (21 years of age or older) based on data from the National Health and Nutrition Examination Survey (NHANES) from 2003-2010. US EPA reported FCRs for WCBA (all races) of 15.8 g/d at the 90th percentile, 23.5 g/d at the 95th percentile, and 46.6 g/d at the 99th percentile. The interim FCR selected by MPCA is substantially higher than the 99th percentile value for WCBA derived by US EPA.

Overall, MPCA's interim FCR overestimates fish consumption in Mississippi River Miles 820 to 812 and results in overly conservative criteria, as illustrated further in Section 3.6.

3.3 MPCA does not provide the necessary underlying information to allow for an independent evaluation and verification of its analyses. A number of calculation discrepancies and errors were identified where data verification was possible.

A bioaccumulation factor (BAF) is the ratio of a chemical's concentration in fish tissue to its concentration in ambient surface water at steady-state (in L/kg). It is used in the derivation of the fish consumption and recreation use class chronic criterion (CC_{FR}), which, when met, will also result in compliance with the fish-tissue-based criterion (CC_{FT}). MPCA (2024c) states that it derived BAFs using fish tissue and surface water datasets collected in 2021 by 3M's contractor, Weston Solutions, Inc., representing the most recent data available for Mississippi River Miles 820 to 812 (Weston Solutions, Inc., 2023).

MPCA (2024c) describes how it processed the PFAS surface water and fish data prior to deriving the BAFs. As described in Appendix A to MPCA (2024c), data processing was completed to account for unit conversions, remove quality control sample data, remove data obtained using specific analytical methods, and address duplicates. No detail is provided on which data were adjusted or eliminated and for what reason. As a result, it is not possible to independently verify or provide comment on the appropriateness of MPCA's processing of the data that it relied upon to derive BAFs.

A review of the raw surface water and fish tissue datasets MPCA relied upon (provided in MPCA [2024d,e]) revealed that the method detection limit and reporting detection limit data fields are identical, and a quantitation limit is not clearly identified. US EPA Region III (1991) states that both a reporting limit and a quantitation limit need to be reported for each datapoint. A review of the underlying laboratory analytical reports included in Weston Solutions, Inc. (2023) shows that MPCA used the analytical reporting limit for non-detect substitutions where data verification was possible. However, a number of analytical reports lacked sufficient detail to distinguish between the analytical detection and reporting limits and MPCA's selected value for non-detect substitution could not be verified in these instances. Therefore, MPCA did not follow US EPA Region III (1991) by not clearly identifying what analytical quantitation limits it used to support its non-detect substitution calculations.

The processed data presented in Appendix A were used to independently verify MPCA's calculation of BAFs. Our review of MPCA's calculation identified a number of calculation discrepancies and errors, as illustrated by the following examples:

- The PFOA fish tissue geometric means for trophic levels 3 and 4 derived using the Regression on Order Statistics (ROS) method (Table 6) paired with the PFOA surface water geometric mean derived using the ROS method (Table 2) do not equate to the BAFs presented in MPCA (2024c) Section 5.2. The PFOA BAFs reported in Section 5.2 are 0.68 L/kg and 1.28 L/kg greater than the derived values from the data presented in Appendix A for trophic levels 3 and 4, respectively. 4
- The PFOS fish tissue geometric mean for trophic level 4 derived using the zero method (Table 6) is presented as 10.6 ng/g. However, the geometric mean for PFOS trophic level 4 fish should be

GRADIENT 11

r082924z

⁴ The PFOA fish tissue geometric means for trophic levels 3 and 4 derived using the ROS method and presented in Appendix A Table 6 of MPCA (2024c) are 0.511 and 0.955 ng/g, respectively. The PFOA surface water geometric mean derived using the ROS method and presented in Appendix A Table 2 of MPCA (2024c) is 23 ng/L. The fish tissue geometric mean by trophic level is divided by the surface water geometric mean and this product is then multiplied by a conversion factor of 1,000 to calculate a BAF in units of L/kg per trophic level. Using the values presented in Appendix A Tables 2 and 6, the calculated PFOA BAFs for trophic levels 3 and 4 are 22.22 and 41.52 L/kg, respectively, which are 0.68 and 1.28 L/kg less than the PFOA BAFs for trophic levels 3 and 4 presented in MPCA (2024c) Section 5.2.

13.4 ng/g and not 10.6 ng/g. The 10.6 ng/g value presented by MPCA in Table 6 appears to be a transcription error and reflects the PFOS fish tissue geometric mean for trophic level 3.

Overall, information is lacking to independently verify or meaningfully comment on MPCA's data processing and analyses and data discrepancies and errors were identified in MPCA's analyses.

3.4 MPCA's approach to developing fish-tissue-based criteria for perfluorooctanoic acid (PFOA) and perfluorohexane sulfonic acid (PFHxS) is inconsistent with its own guidance and best available science.

MPCA (2024c) derived chronic fish tissue (CC_{FT}) to protect fish consumers in Mississippi River Miles 820 to 812 from bioaccumulative chemicals of concern (BCCs), specifically PFOS, PFOA and PFHxS. BCCs are defined by Minnesota rules as any chemical that accumulates **in aquatic organisms** [emphasis added] by a BAF greater than 1,000 L/kg, as described in Minn. R. 7052.0010 Subp.4 (MPCA, 2024f). The datasets used in MPCA (2024c) show that PFOS BAFs exceed 1,000 L/kg for *Pomoxis nigromaculatus* (black crappie), *Sander canadensis* (sauger), and *Morone chrysops* (white bass) fish tissue samples collected adjacent to Cottage Grove. The Interstate Technology and Regulatory Council (ITRC, 2021; Table 5-1) reviewed BAFs for PFOS from freshwater field studies and similarly found values that exceed 1,000 L/kg.

However, evidence supporting PFOA and PFHxS as BCCs is lacking. MPCA (2024c) justifies the bioaccumulative potential of PFOA and PFHxS in fish with evidence that these PFAS are known to be highly bioaccumulative in humans. This qualitative consideration of the bioaccumulation potential of a chemical in humans as opposed to aquatic organisms is not consistent with how BCCs are defined in the Minnesota rules (MPCA, 2024f). MPCA (2024c) further cites ITRC (2021) as evidence that both PFOA and PFHxS have demonstrated BAFs greater than 1,000 L/kg in other field studies. However, an independent review of the studies cited in ITRC does not support MPCA's conclusion:

- Two field studies with PFOA and PFHxS BAFs greater than 1,000 L/kg in the Great Lakes Region were reported in ITRC (2021, Table 5-1). As described below, these studies calculated BAFs using whole fish instead of fish fillet analyses. Moreover, the collection of fish samples and surface water samples occurred at different times. Therefore, the findings in these studies carry substantial uncertainty and are not appropriate for evaluating bioaccumulation into edible fish tissue. Further, despite these uncertainties, one of the studies (*i.e.*, De Silva *et al.*, 2011, as cited in ITRC, 2021, Table 5-1) describes PFOA field BAFs that are well below the BCC threshold of 1,000 L/kg.
 - Furdui *et al.* (2007, as cited in ITRC, 2021, Table 5-1) reported PFOA field BAFs from whole body *Salvelinus namaycush* (lake trout) collected from Lakes Superior, Huron, Erie, Ontario and Michigan in the range of 398-3,981 L/kg wet weight, and PFHxS field BAFs from whole body lake trout collected from Lakes Superior, Huron, Erie and Ontario in the range of 63-1,995 L/kg wet weight. Fish were collected in 2001 and surface water was collected in 2005 and 2006.
 - De Silva *et al.* (2011, as cited in ITRC, 2021, Table 5-1) reported PFOA field BAFs from whole body lake trout from Lakes Superior, Erie, and Ontario in the range of 10-203 L/kg wet weight, and from whole body *Sander vitreus* (walleye) from Lake Erie with a reported BAF of 91 L/kg wet weight. De Silva *et al.* (2011, as cited in ITRC, 2021, Table 5-1) reported PFHxS field BAFS derived from whole body lake trout from Lakes Huron, Erie and Ontario in the range of 745-2,183 L/kg wet weight. Fish were collected between 2006 and 2008 and surface water was collected between 2005, 2006, 2007 and 2010.

A review of the recent scientific literature on PFAS bioaccumulation and MPCA's own analyses further support the conclusion that PFOA and PFHxS are not BCCs:

- MPCA's own analysis presented in Figure 3 of Appendix A (MPCA, 2024c) clearly shows the difference in bioaccumulation of PFOS versus PFOA and PFHxS. While PFOS geomeans are greater in trophic level 4 fish than in trophic level 3 fish, providing evidence of biomagnification, geomeans between trophic levels 3 and 4 are nearly the same for PFOA and PFHxS, indicating that these two PFAS do not biomagnify and their relative tissue concentrations are well below those measured for PFOS.
- US EPA recently published a review of BCF and BAF values for PFOS, PFHxS and PFOA in aquatic organisms and reported median BAFs for fish muscle as 1,514, 20 and 8.5 L/kg wet weight for PFOS, PFHxS and PFOA, respectively (Burkhard, 2021, Table 4). Similarly, US EPA describes the current state of the science of PFOS and PFOA bioaccumulation in its draft aquatic life criteria documents for these two PFAS and reported geometric mean BAFs for fish muscle as 1,069 and 7.2 L/kg wet weight for PFOS and PFOA, respectively (US EPA, 2022b,c). These reviews by US EPA indicate that PFOA and PFHxS have BAFs that are much lower than those obtained for PFOS and would not meet the 1,000 L/kg BCC threshold.
- Lastly, MPCA came to the same conclusion that PFOA and PFHxS are not BCC in its 2023 generalized guidance for PFAS WQC to protect human health (MPCA, 2023a). MPCA states in that document that deriving CC_{FT} for PFOA and PFHxS is not applicable because BAFs derived from fish tissue-based field datasets indicate BAFs less than 1,000 L/kg, with geometric mean BAFs in a similar range of 32 to 60 L/kg.

Overall, MPCA's (2024c) approach to developing fish-tissue-based criteria for PFOA and PFHxS is not supported by the current state of the science and inconsistent with its own prior interpretation of the bioaccumulation potential of these two PFAS.

3.5 MPCA's methodology for calculating fish bioaccumulation factors (BAFs) is technically flawed and is inconsistent with US EPA guidance.

MPCA (2024c) calculated PFAS fish tissue and surface water geometric means for use in BAF derivations using five different approaches to address non-detected data (Appendix A in MPCA, 2024c). The ROS method was ultimately chosen by MPCA to calculate geometric means, and one half of the detection limit was used as a substitution for values reported as non-detected when the data did not meet the ROS criteria. MPCA (2024c, Section 3.2) cites US EPA Region III (1991) to support its use of ROS and one half of the detection limit as appropriate approaches for addressing non-detect data. Although US EPA Region III (1991) states that statistical estimates of concentrations below the detection limit (such as the ROS method) are technically superior to evaluating non-detects at one half of the detection limit, this approach is only effective for datasets with a high proportion of detected results, typically greater than 50%. However, an US EPA's Office of Research and Development's (ORD) National Exposure Research Laboratory publication (US EPA, 2006) that post-dates US EPA Region III's 31-year-old guidance, emphasizes the need to consider data distribution and data outliers in selecting the appropriate method to address non-detect values. To appropriately use the ROS method, US EPA's ORD states both that the number of detected observations must be large enough to obtain accurate and reliable results and that the data follow a well-

GRADIENT 13

r082924z

⁵ MPCA describes that it did not use the ROS method when (1) two or fewer values in a given dataset were detected or (2) two or fewer values in a given dataset were not detected (MPCA, 2024c, Appendix A, p. 30).

known parametric distribution (US EPA, 2006). MPCA's approach is inconsistent with US EPA's ORD guidance for several reasons:

- In 7 of the 10 instances where the ROS method was selected to calculate fish tissue geometric means across PFAS compounds and trophic levels, the frequency of non-detected results exceeded 50% (MPCA, 2024c, Appendix A). Due to the high percentage of non-detected results in the fish tissue dataset, ROS would not be an appropriate method for calculating geometric means used in BAF derivations.
- MPCA does not provide a rationale for using the ROS method in light of the distribution of the underlying dataset and the potential presence of data outliers. Multiple ROS methods are available to compute non-detected results based on different data distributions (normal, lognormal, gamma), although verifying the distribution of left-censored datasets is challenging when the frequency of non-detected results is large (US EPA, 2006). The distribution of the datasets used by MPCA is not adequately described to allow for independent ROS method verification. US EPA (2006) also summarizes the influence of outliers on various ROS methods and details how ROS approaches do not perform well when datasets contain outliers. MPCA does not describe whether statistical tests were used to identify outliers in these datasets or whether any outliers were identified, and does not discuss the potential impact of statistical outliers on its derivation of BAFs.
- The ROS method is known to potentially extrapolate non-detected results that are greater than detected values in the dataset, which can result in overestimates of a data population's geomean. When handling non-detects at the detection limit (DL), US EPA Region III (1991) states: "in this highly conservative approach, all non-detects are assigned the value of the DL, the largest concentration of analyte that could be present but not detected. This method always produces a mean concentration, which is biased high, and is not consistent with Region III's policy of using best science in risk assessments." However, MPCA's ROS-based geomeans are even higher than the detection limit-based geomeans that US EPA would consider inappropriately biased high. Specifically, all fish tissue geometric means calculated using the ROS method exceed the geometric means calculated using the detection limit method to evaluate non-detects, as presented in Appendix A Table 6 (MPCA, 2024c). In some instances, the ROS-based geomean that MPCA derived is greater than two times higher than the detection limit-based geomeans that US EPA would consider inappropriately biased high (e.g., PFHxA fish trophic level 3 and PFOA fish trophic level 4).

MPCA used R software for statistical computing, which relies upon specialized programming languages that is not technically accessible. MPCA's use of R results unnecessarily complicates independent verification of their analyses. Instead, MPCA could have relied on US EPA's ProUCL statistical software which has functions for imputing non-detects using ROS methods. ProUCL is publicly available, easy to use, and considered the default software package by risk assessment practitioners for environmental data calculations.

Overall, MPCA's approach to addressing data sets with below detection limit observations is not consistent with applicable US EPA guidance, technically flawed, unnecessarily complicated, and lacks transparency. MPCA's approach resulted in higher BAF values and lower criteria as described further in Section 3.6.

3.6 MPCA's consistent reliance on unsupported toxicological values and exposure parameters, when considered in combination, results in site-specific criteria (SSCs) that are not site-specific, and are inconsistent with similar values developed by other regulatory entities and with MPCA's own regulatory processes to protect the designated uses of the Mississippi River Miles 820 to 812.

MPCA did not include any discussion of the uncertainty associated with its SSC derivation and the importance of the various input parameters it selected. This is a significant omission and contrary to established state and federal guidance and policy on risk assessment in environmental decision-making. For example, it is long-standing US EPA policy that stakeholders in environmental issues be provided with sufficient information to allow them to independently assess environmental risks and the reasonableness of risk reduction actions (US EPA Region VI and US EPA Region V, 2008). To ensure that risk assessments exhibit these qualities, US EPA has specified requirements that must be met when characterizing risk: (1) addressing qualitative and quantitative features of the risk assessment and (2) identifying uncertainties as a measure of the confidence in the assessment. Quantifying uncertainty in risk assessment is typically performed by conducting a sensitivity analysis where exposure parameters are varied, and the changes in risk estimates are compared to characterize the uncertainty associated with the final risk estimates (US EPA, 1989). In its exposure factors handbook, US EPA further describes how accounting for variability and uncertainty is fundamental to exposure assessment and risk analysis (US EPA, 2011). While historically, risk assessors may have used qualitative descriptors (e.g., high-end, worst case, average), it is no longer considered best practice to rely on these types of descriptors when the data allow for quantification of the uncertainty as it relates to exposure estimates, risk estimates, environmental policy options, or – as in this case – WQCs. MPCA similarly has recognized the importance of uncertainty analysis in environmental decision making (e.g., MPCA, 2023b).⁶

In consideration of MPCA's consistent reliance on overly conservative toxicological values and exposure parameters, we sought to understand the sensitivity of these parameters when used in combination to derive the SSCs for Mississippi River Miles 820 to 812. It is clear from the sensitivity analysis presented here that reliance on a more reasonable set of alternate parameters results in substantially different criteria. MPCA's consistent selection of overly conservative toxicological values and exposure parameters has a substantial compounding effect, as illustrated by our analysis. Our analyses consider the impact of changing one parameter at a time on the SSCs in a stepwise fashion, as summarized in Table 3.1:

- 1. The toxicological values derived by MDH were used instead of the US EPA values to better align with Minnesota Rules.
- 2. The BAFs derived using one half of the detection limit for non-detects were used (as presented in MPCA 2024c, Appendix A). Given the number of non-detects in several of the PFAS datasets, this substitution method is considered more appropriate than the ROS method used by MPCA (2024c).
- 3. The FCR was updated to 0.00043 kg/kg-d based on the 30 g/d default FCR as outlined in Minnesota Rules 7050.2019 Subp.13 (MPCA, 2020a), compared to the interim FCR for WCBA of 0.00094 kg/kg-d chosen by MPCA (2024c).

GRADIENT 15

r082924z

⁶ In this example, MPCA identifies that risk assessments should include an uncertainty analysis with discussion of possible sources of uncertainty. The discussion should also indicate whether the uncertainty has a biased impact on the risk characterization results (*e.g.*, leading to an over- or under-estimation of risk) and, if possible, the magnitude of the effect.

Surface water and fish tissue-based chronic criteria were then calculated using these alternate parameters and the Minnesota Rules 7050.2019 Subp.14 and 15 (MPCA, 2020a) algorithms presented in Section 3 above. The stepwise increase in the calculated chronic criteria is shown in Figures 3.1 through 3.11. The multi-fold increase over MPCA's derived SSC is shown in these figures for each stepwise change in input parameter. The impact of individually changing each of the four parameters in Table 3.1 on the resulting SSCs is summarized in Table 3.2a and Table 3.2b.

Table 3.1 Sensitivity Analysis of the SSC Calculation Using Alternate Input Parameters

Parameter	MPCA (2024c)	Alternate Parameter	Reason for Alternate Parameter
RfD or CSF	Sourced from US EPA ^a	Sourced from MDH ^b	Aligns with Minnesota Rules
BAF	ROS Method ^c	½ Detection Limit Method ^c	More Appropriate Substitution Method
FCR	0.00094 kg/kg-d ^d	0.00043 kg/kg-d ^e	Aligns with Minnesota Rules

Notes:

BAF = Bioaccumulation Factor; CSF = Cancer Slope Factor; FCR = Fish Consumption Rate; MDH = Minnesota Department of Health; MPCA = Minnesota Pollution Control Agency; PFBA = Perfluorobutanoic Acid; PFBS = Perfluorobutane Sulfonic Acid; PFHXA = Perfluorobexanoic Acid; PFOA = Perfluoroctanoic Acid; PFOS = Perfluoroctane Sulfonic Acid; RfD = Reference Dose; ROS = Regression on Order Statistics; SSC = Site-Specific Criterion; US EPA = United States Environmental Protection Agency.

- (a) Toxicological values used by MPCA (2024c) can be found in the following MPCA (2024c) sections: PFOS Section 4.1; PFOA Section 5.1; PFHxS Section 6.1; PFHxA Section 7.1; PFBS Section 8.1; PFBA Section 9.1.
- (b) Toxicological values sourced from MDH can be found in the following sections of our report: PFOS Section 3.1.1; PFOA Section 3.1.2; PFHxS Section 3.1.3; PFHxA Section 3.1.4; PFBS Section 3.1.5; PFBA Section 3.1.6.
- (c) Data sourced from MPCA (2024c) Appendix A.
- (d) FCR used by MPCA (2024c) can be found in MPCA (2024c) Section 3.3.
- (e) Alternate parameter sourced from Minnesota Rules 7050.0219 (MPCA, 2020a).

Table 3.2a Sensitivity Analysis of the Surface Water Chronic Criteria Calculation Using Alternate Input Parameters

	CC _{FR} (ng/L)				
PFAS	MPCA (2024c)	MPCA (2024c) Alternate RfD Alternate BAF		Alternate FCR	
		or CSF			
PFOS	0.027	0.270	0.027	0.060	
PFOA	0.0092	0.021	0.033	0.019	
PFHxS	0.0023	110	0.0043	0.0046	
PFHxA	4,400	2,800	11,000	9,000	
PFBS	3,000	840	5,500	6,100	
PFBA	25,000	96,000	53,000	46,000	

Notes:

BAF = Bioaccumulation Factor; CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; CSF = Cancer Slope Factor; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFAS = Per- and Polyfluorinated Substance; PFBA = Perfluorobutanoic Acid; PFBS = Perfluorobutane Sulfonic Acid; PFHXA = Perfluorohexanoic Acid; PFHXS = Perfluorohexane Sulfonic Acid; PFOA = Perfluorooctanoic Acid; PFOS = Perfluorooctane Sulfonic Acid; RfD = Reference Dose.

This table shows the surface water chronic criterion when using an alternate input for the RfD/CSF, BAF, or FCR (see Table 3.1)

Table 3.2b Sensitivity Analysis of the Fish Tissue Chronic Criteria Calculation Using Alternate Input Parameters

		CC _{FT} (ng/g)	
PFAS	MPCA (2024c)	Alternate RfD or	Alternate FCR
		CSF	
PFOS	0.021	0.210	0.047
PFOA	0.00036	0.00084	0.00079
PFHxS	0.000043	2.10	0.000093

Notes:

CC_{FT} = Fish-Tissue-Based Chronic Criterion; CSF = Cancer Slope Factor; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFAS = Per- and Polyfluorinated Substance; PFHxS = Perfluorohexane Sulfonic Acid; PFOA = Perfluorooctanoic Acid; PFOS = Perfluorooctane Sulfonic Acid; RfD = Reference Dose.

This table shows the fish tissue chronic criterion when using an alternate input for the RfD/CSF or FCR (see Table 3.1)

Alternate PFOS surface water chronic criteria were derived using an RfD of 1×10^{-6} mg/kg-d (MDH, 2024a) and the alternate exposure parameters outlined in Table 3.1. Updating the PFOS RfD results in a 10-fold greater CC_{FR} . Additionally updating the FCR parameter results in a CC_{FR} value that is 21.8 times higher than the CC_{FR} derived by MPCA (Figure 3.1). Updating the BAF substitution method is not applicable to PFOS since this PFAS was detected in all fish tissue and surface water samples.

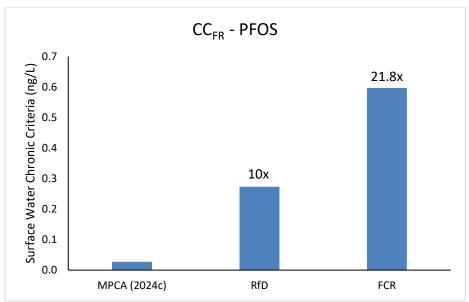


Figure 3.1 Sensitivity Analysis of PFOS CC_{FR} Using Alternate Input Parameters. CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFOS = Perfluorooctane Sulfonic Acid; RfD = Reference Dose. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

Alternate PFOA surface water chronic criteria were derived using a CSF of 12,600 mg/kg-d (MDH, 2024b) and the alternate exposure parameters outlined in Table 3.1. Updating the PFOA CSF results in a 2.3-fold greater CC_{FR} . Additionally updating the BAF and FCR parameters results in CC_{FR} values that are 8.3 and 15.9 times higher than the CC_{FR} derived by MPCA, respectively (Figure 3.2).

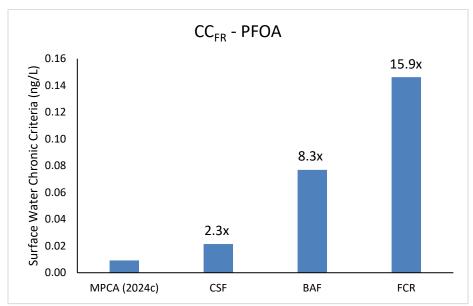


Figure 3.2 Sensitivity Analysis of PFOA CC_{FR} Using Alternate Input Parameters. BAF = Bioaccumulation Factor; CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFOA = Perfluorooctanoic Acid. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

Alternate PFHxS surface water criteria were derived using an RfD of 9.7×10^{-6} mg/kg-d (MDH, 2023a) and the alternate exposure parameters outlined in Table 3.1. Updating the PFHxS RfD results in a 48,500-fold greater CC_{FR} . Additionally updating the BAF and FCR parameters results in CC_{FR} values that are 92,533 and 173,334 times higher than the CC_{FR} derived by MPCA, respectively (Figure 3.3).

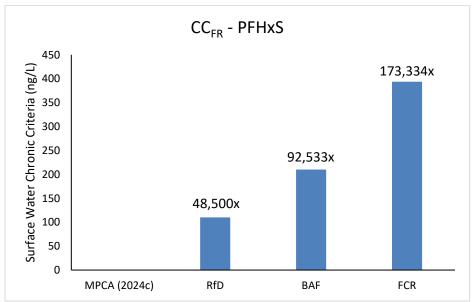


Figure 3.3 Sensitivity Analysis of PFHxS CC_{FR} Using Alternate Input Parameters. BAF = Bioaccumulation Factor; CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; MPCA = Minnesota Pollution Control Agency; PFHxS = Perfluorohexane Sulfonic Acid; RfD = Reference Dose. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

In addition, changing only the PFHxS RfD for the US EPA (2023a) draft IRIS RfD (4×10^{-10} mg/kg-d), the RfD used by US EPA (2024d) to derive the MCLG for PFHxS (2×10^{-6} mg/kg-d), or the RfD developed by MDH (2023a) (9.7×10^{-6} mg/kg-d) results in CC_{FR} values that are either 2, 10,000, or 48,500 times higher than the CC_{FR} derived by MPCA, respectively (Figure 3.4).

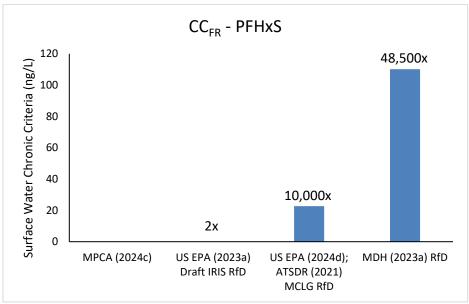


Figure 3.4 Sensitivity Analysis of PFHxS CC_{FR} **Using Alternate RfDs.** ATSDR = Agency for Toxic Substances and Disease Registry; CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; IRIS = Integrated Risk Information Systems; MCLG = Maximum Contaminant Level Goal; MDH = Minnesota Department of Health; MPCA = Minnesota Pollution Control Agency; PFHxS = Perfluorohexane Sulfonic Acid; RfD = Reference Dose; US EPA = United States Environmental Protection Agency.

Alternate PFHxA surface water chronic criteria were derived using an RfD of 3.2×10^{-4} mg/kg-d (MDH, 2023b) and the alternate exposure parameters outlined in Table 3.1. Updating the PFHxA RfD results in a lower CC_{FR}. Additionally updating the BAF and FCR parameters results in CC_{FR} values that are 1.6 and 3.0 times higher than the CC_{FR} derived by MPCA, respectively (Figure 3.5).

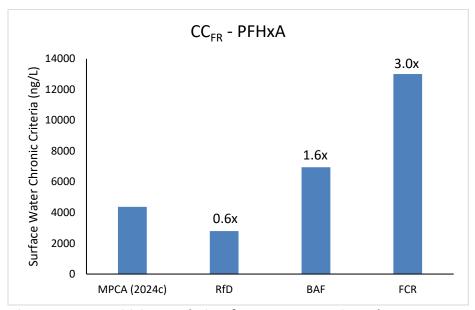


Figure 3.5 Sensitivity Analysis of PFHxA CC_{FR} Using Alternate Input Parameters. BAF = Bioaccumulation Factor; CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFHxA = Perfluorohexanoic Acid; RfD = Reference Dose. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

Alternate PFBS surface water chronic criteria were derived using an RfD of 8.4×10^{-5} mg/kg-d (MDH, 2023c) and the alternate exposure parameters outlined in Table 3.1. Updating the PFBS RfD results in lower CC_{FR}. Additionally updating the BAF and FCR parameters similarly results in a CC_{FR} that is lower than the CC_{FR} derived by MPCA (Figure 3.6).

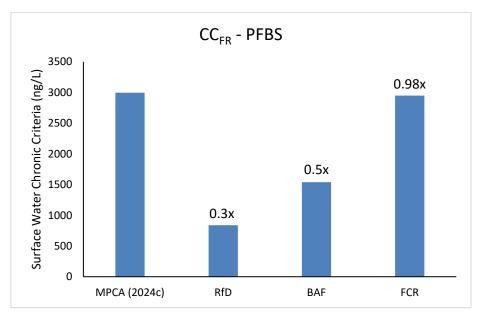


Figure 3.6 Sensitivity Analysis of PFBS CC_{FR} Using Alternate Input Parameters. BAF = Bioaccumulation Factor; CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFBS = Perfluorobutane Sulfonic Acid; RfD = Reference Dose. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

Alternate PFBA surface water chronic criteria were derived using an RfD of 3.8×10^{-3} mg/kg-d (MDH, 2018) and the alternate exposure parameters outlined in Table 3.1. Updating the PFBA RfD results in a 3.8-fold greater CC_{FR}. Additionally updating the BAF and FCR parameters results in CC_{FR} values that are 8.0 and 12.4 times higher than the CC_{FR} derived by MPCA, respectively (Figure 3.7).

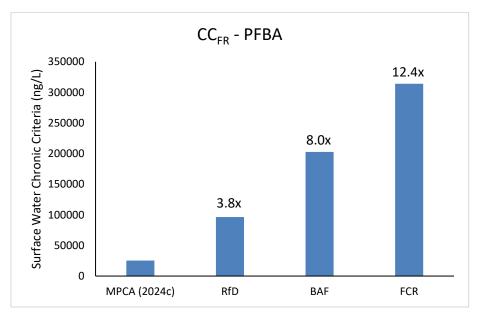


Figure 3.7 Sensitivity Analysis of PFBA CC_{FR} Using Alternate Input Parameters. BAF = Bioaccumulation Factor; CC_{FR} = Fish Consumption and Recreation Use Class Chronic Criterion; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFBA = Perfluorobutanoic Acid; RfD = Reference Dose. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

Alternate PFOS CC_{FT} were derived using an RfD of 1×10^{-6} mg/kg-d (MDH, 2024a) and the alternate exposure parameters outlined in Table 3.1. CC_{FT} is only derived for chemicals determined to be BCC and the BAF exposure parameter is not included in these algorithms. Updating the PFOS RfD results in a 10-fold increase in the CC_{FT} . Additionally updating the FCR parameter results in a CC_{FT} that is 21.9 times higher than the CC_{FT} derived by MPCA (Figure 3.8).

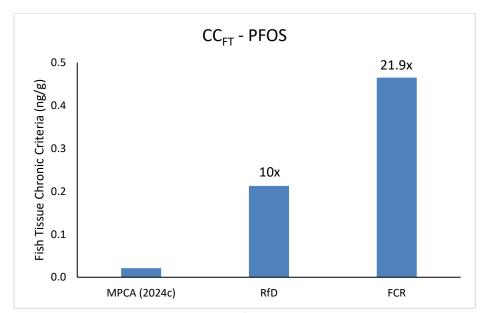


Figure 3.8 Sensitivity Analysis of PFOS CC_{FT} Using Alternate Input Parameters. CC_{FT} = Fish Tissue-Based Chronic Criterion; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFOS = Perfluorooctane Sulfonic Acid; RfD = Reference Dose. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

Alternate PFOA CC_{FT} were derived using a CSF of 12,600 mg/kg-d (MDH, 2024b) and the alternate FCR outlined in Table 3.1. CC_{FT} is only derived for chemicals determined to be BCC and the BAF exposure parameter is not included in these algorithms. Updating the PFOA CSF and FCR parameter results in CC_{FT} values that are 2.3 and 5.1 times higher than the CC_{FT} derived by MPCA, respectively (Figure 3.9).

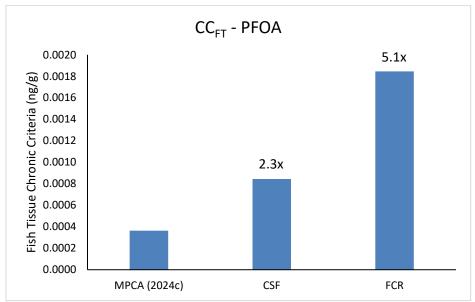


Figure 3.9 Sensitivity Analysis of PFOA CC_{FT} **Using Alternate Input Parameters.** CC_{FT} = Fish Tissue-Based Chronic Criterion; CSF = Cancer Slope Factor; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFOA = Perfluorooctanoic Acid. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

Alternate PFHxS CC_{FT} were derived using an RfD of 9.7×10^{-6} mg/kg-d (MDH, 2023a) and the alternate exposure parameters outlined in Table 3.1. CC_{FT} is only derived for chemicals determined to be BCC and the BAF exposure parameter is not included in these algorithms. Updating the PFHxS RfD and FCR results in CC_{FT} values that are 48,500 and 106,023 times higher than the CC_{FT} derived by MPCA, respectively (Figure 3.10).

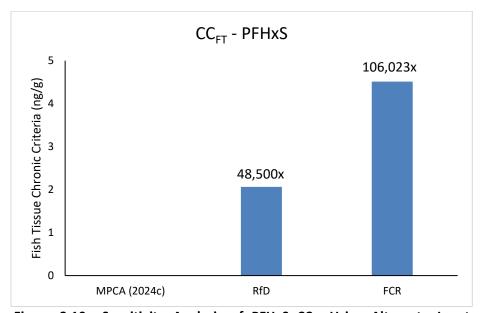


Figure 3.10 Sensitivity Analysis of PFHxS CC_{FT} Using Alternate Input Parameters. CC_{FT} = Fish Tissue-Based Chronic Criterion; FCR = Fish Consumption Rate; MPCA = Minnesota Pollution Control Agency; PFHxS = Perfluorohexane Sulfonic Acid; RfD = Reference Dose. Criteria were adjusted in a stepwise manner (as shown from left to right). The cumulative increase of the criteria is shown above each bar on the graph.

In addition, changing only the PFHxS RfD to either the correct value for the US EPA (2023a) draft IRIS RfD (4×10^{-10} mg/kg-d), the RfD used by US EPA (2024d) to derive the MCLG for PFHxS (2×10^{-6} mg/kg-d), or the RfD developed by MDH (2023a) (9.7×10^{-6} mg/kg-d) results in CC_{FT} values that are either 2, 10,000, or 48,500 times higher than the CC_{FT} derived by MPCA, respectively (Figure 3.11).

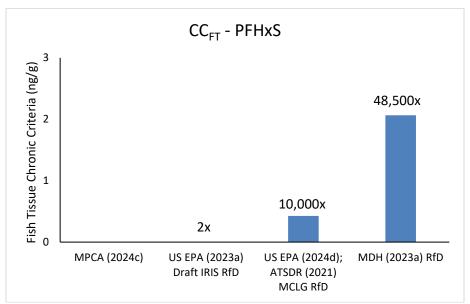


Figure 3.11 Sensitivity Analysis of PFHxS CC_{FT} **Using Alternate RfDs.** ATSDR = Agency for Toxic Substances and Disease Registry; CC_{FT} = Fish Tissue-Based Chronic Criterion; IRIS = Integrated Risk Information Systems; MCLG = Maximum Contaminant Level Goal; MDH = Minnesota Department of Health; MPCA = Minnesota Pollution Control Agency; PFHxS = Perfluorohexane Sulfonic Acid; RfD = Reference Dose; US EPA = United States Environmental Protection Agency.

The overall impact of MPCA's assumptions and parameter selection is a set of SSCs that is not supported by the best available science, is overly protective, and is inconsistent with similar criteria developed by other states. For example, the fish tissue action level for PFOS developed by WDNR (2022) is 50 ng/g as compared to 0.021 ng/g developed by MPCA (2024c). Similarly, human health WQCs for PFOS and PFOA, 12 and 170 ng/L, respectively, developed by Michigan Department of Environment, Great Lakes, and Energy (EGLE, 2024) are much higher than the SSC for PFOS and PFOA, 0.027 and 0.0092 ng/L, respectively, derived by MPCA (2024c).

References

Agency for Toxic Substances and Disease Registry (ATSDR). 2021. "Toxicological Profile for Perfluoroalkyls." 993p., May.

Burkhard, LP. 2021. "Evaluation of published bioconcentration factor (BCF) and bioaccumulation factor (BAF) data for per- and polyfluoroalkyl substances across aquatic species." *Environ. Toxicol. Chem.* 40(6):1530-1543. doi: 10.1002/etc.5010.

Connelly, NA; Lauber, TB; Niederdeppe, J; Knuth, BA. [Cornell University, Dept. of Natural Resources, Center for Conservation Social Sciences]. 2019. "Reducing Toxic Exposure from Fish Consumption in Women of Childbearing Age and Urban Anglers: Results of a Two-Year Diary Study (Final)." HDRU Publ. No. 16-3, 162p., August.

Great Lakes Consortium for Fish Consumption Advisories (GLCFCA). 2019. "Best Practice for Perfluorooctane Sulfonate (PFOS) Guidelines." 22p., November.

Interstate Technology and Regulatory Council (ITRC). 2021. "Partitioning of PFAS into Fish - BCF and BAF." Per- and Polyfluoroalkyl Substances (PFAS) Technical Team, October 7. Accessed http://pfas-dev.itrcweb.org/wp-content/uploads/2023/10/ITRC_PFAS_-BCF-BAF_compilation_Table5-1 Oct2021.xlsx.

Michigan Dept. of Environment, Great Lakes, and Energy (EGLE). 2024. "Rule 57 water quality values." Accessed at https://www.michigan.gov/egle/about/organization/water-resources/glwarm/rule-57-water-quality-values.

Minnesota Dept. of Health (MDH). 2018. "Health Based Guidance for Water, Toxicological Summary for: Perfluorobutanoate (CAS Nos. 45048-62-2 (anion); 375-22-4 (acid))." Environmental Health Division, Health Risk Assessment Unit, 9p., August.

Minnesota Dept. of Health (MDH). 2019. "Health Based Guidance for Water, Toxicological Summary for Perfluorooctane sulfonate (PFOS) (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 19p., April. Accessed at https://www.health.state.mn.us/communities/environment/risk/docs/guidance/gw/pfos.pdf.

Minnesota Dept. of Health (MDH). 2023a. "Health Based Guidance for Water, Toxicological Summary for: Perfluorohexane sulfonate (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 13p., November.

Minnesota Dept. of Health (MDH). 2023b. "Health Based Guidance for Water, Toxicological Summary for: Perfluorohexanoate (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 8p., November.

Minnesota Dept. of Health (MDH). 2023c. "Health Based Guidance for Water, Toxicological Summary for: Perfluorobutane sulfonate (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 9p., November.

Minnesota Dept. of Health (MDH). 2024a. "Health Based Guidance for Water, Toxicological Summary for: Perfluorooctane sulfonate (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 16p., February.

Minnesota Dept. of Health (MDH). 2024b. "Health Based Guidance for Water, Toxicological Summary for: Perfluorooctanoate (Various CAS Nos.)." Environmental Health Division, Health Risk Assessment Unit, 16p., January.

Minnesota Dept. of Health (MDH); University of Illinois at Chicago (UIC). 2017. "Technical Report: Fish are Important for Superior Health (FISH) Project." Environmental Health Division, 34p., May.

Minnesota Dept. of Natural Resources (Minnesota DNR). 2024a. "Mississippi River Pool 2." Accessed at https://www.dnr.state.mn.us/areas/fisheries/eastmetro/rivers/pool2.html.

Minnesota Dept. of Natural Resources (Minnesota DNR). 2024b. "Minnesota's watershed basins." Accessed at https://www.dnr.state.mn.us/watersheds/map.html

Minnesota Pollution Control Agency (MPCA). 2017. "Human Health-based Water Quality Standards Technical Support Document: Water Quality Standard Amendments – Minn. R. chs. 7050 and 7052 (Final)." 181p., June.

Minnesota Pollution Control Agency (MPCA). 2020a. "Human health-based criteria and standards." Minn. R. 7050.0219. 14p. Accessed at https://www.revisor.mn.gov/rules/7050.0219/.

Minnesota Pollution Control Agency (MPCA). 2020b. "Water Quality Standards Technical Support Document: Human Health Protective Water Quality Criteria for Perfluorooctane Sulfonate (PFOS)." 38p., December.

Minnesota Pollution Control Agency (MPCA). 2022. "Interim Fish Consumption Rate for Women of Childbearing Age (Water Quality Criteria – Minn. R. chs. 7050 and 7052)." wq-s6-60, 30p., November.

Minnesota Pollution Control Agency (MPCA). 2023a. "Water Quality Standards: Human Health Protective Water Quality Criteria for Per- and Polyfluoroalkyl Substances (PFAS)." wq-s6-63, 67p., January.

Minnesota Pollution Control Agency (MPCA). 2023b. "MPCA Remediation Division PFAS Guidance (Draft)." Remediation Division, c-rem3-28, 60p., August.

Minnesota Pollution Control Agency (MPCA). 2024a. "National Pollutant Discharge Elimination System/State Disposal System Permit (MN0001449) for 3M Cottage Grove Center, Unnamed creek - Class 2Bg, 3, 4A, 4B, 5, 6 water, Cottage Grove, Washington County (Draft)." Industrial Division, Water and Mining Section, Submitted to 3M Chemical Operations LLC. 415p.

Minnesota Pollution Control Agency (MPCA). 2024b. "Public Notice of intent to reissue, Wastewater Permit and TMDL Wasteload Allocation: Wastewater Permit MN0001449, 3M Chemical Operations' Cottage Grove Facility, 10746 Innovation Rd, Cottage Grove, MN 55016-4600." Industrial Division, 7p.

Minnesota Pollution Control Agency (MPCA). 2024c. "Human Health Protective Water Quality Criteria for Per- and Polyfluoroalkyl Substances (PFAS) in Mississippi River, Miles 820 to 812." wq-s6-69a, 45p., May.

Minnesota Pollution Control Agency (MPCA). 2024d. "Human Health Protective Water Quality Criteria for Per- and Polyfluoroalkyl Substances (PFAS) in Mississippi River, Miles 820 to 812: Water data for miles 820 to 812." wq-s6-69d, May. Accessed at https://www.pca.state.mn.us/sites/default/files/wq-s6-69d.xlsx.

Minnesota Pollution Control Agency (MPCA). 2024e. "Human Health Protective Water Quality Criteria for Per- and Polyfluoroalkyl Substances (PFAS) in Mississippi River, Miles 820 to 812: Fish tissue data for miles 820 to 812." wq-s6-69c, May. Accessed at https://www.pca.state.mn.us/sites/default/files/wq-s6-69c.xlsx.

Minnesota Pollution Control Agency (MPCA). 2024f. "Lake Superior Basin Water Standards." Minn. R. 7052. 60p. Accessed on August 12, 2024 at https://www.revisor.mn.gov/rules/7052/

Ruffle, B; Archer, C; Vosnakis, K; Butler, JD; Davis, CW; Goldsworthy, B; Parkman, R; Key, TA. 2024. "US and international per- and polyfluoroalkyl substances surface water quality criteria: A review of the status, challenges, and implications for use in chemical management and risk assessment." *Integr. Environ. Assess. Manag.* 20(1):36-58. doi: 10.1002/ieam.4776.

US EPA. 1989. "Risk Assessment Guidance for Superfund (RAGS). Volume I: Human Health Evaluation Manual (Part A) (Interim final)." Office of Emergency and Remedial Response, Washington, DC, NTIS PB90-155581, EPA-540/1-89-002. 287p., December.

US EPA. 2006. "On the Computation of a 95% Upper Confidence Limit of the Unknown Population Mean Based Upon Data Sets with Below Detection Limit Observations." EPA/600/R-06/022, 141p., March.

US EPA. 2011. "Exposure Factors Handbook: 2011 Edition." National Center for Environmental Assessment (NCEA), Office of Research and Development, EPA/600/R-090/052F, 1436p., September.

US EPA. 2014. "Estimated Fish Consumption Rates for the U.S. Population and Selected Subpopulations (NHANES 2003-2010) (Final Report)." EPA-820-R-14-002, 110p., April.

US EPA. 2021. "Human Health Toxicity Values for Perfluorobutane Sulfonic Acid (CASRN 375-73-5) and Related Compound Potassium Perfluorobutane Sulfonate (CASRN 29420-49-3)." Office of Research and Development (ORD), Center for Public Health and Environmental Assessment (CPHEA), EPA/600/R-20/345F, 169p., April.

US EPA. 2022a. "IRIS Toxicological Review of Perfluorobutanoic Acid (PFBA, CASRN 375-22-4) and Related Salts." Center for Public Health and Environmental Assessment, Office of Research and Development, EPA/635/R-22/277Fa, 146p., December.

US EPA. 2022b. "Draft Aquatic Life Ambient Water Quality Criteria for Perfluorooctane Sulfonate (PFOS)." Office of Water, Office of Science and Technology, Health and Ecological Criteria Division, EPA-842-D-22-002, 696p., April.

US EPA. 2022c. "Draft Aquatic Life Ambient Water Quality Criteria for Perfluorooctanoic Acid (PFOA)." Office of Water, Office of Science and Technology, Health and Ecological Criteria Division, EPA-842-D-22-001, 546p., April.

US EPA. 2023a. "Toxicological Review of Perfluorohexanesulfonic Acid [PFHxS, CASRN 335-46-4] and Related Salts (External Review Draft)." Office of Research and Development, Center for Public Health and Environmental Assessment, Integrated Risk Information System, EPA/635/R-23/148a, 459p., July.

US EPA. 2023b. "IRIS Toxicological Review of Perfluorohexanoic Acid (PFHxA, CASRN 307-24-4) and Related Salts." Center for Public Health and Environmental Assessment, Office of Research and Development, EPA/635/R-23/027Fa, 221p., April.

US EPA. 2024a. "Human Health Toxicity Assessment for Perfluorooctane Sulfonic Acid (PFOS) and Related Salts (Final)." Health and Ecological Criteria Division, Office of Science and Technology, Office of Water, EPA Document No. 815R24007, 514p., April.

US EPA. 2024b. "Human Health Toxicity Assessment for Perfluorooctanoic Acid (PFOA) and Related Salts (Final)." Health and Ecological Criteria Division, Office of Science and Technology, Office of Water, EPA Document No. 815R24006. 556p., April.

US EPA. 2024c. "Regional Screening Levels (RSLs)." May. Accessed at https://www.epa.gov/risk/regional-screening-levels-rsls.

US EPA. 2024d. "Maximum Contaminant Level Goals (MCLGs) for Three Individual Per- and Polyfluoroalkyl Substances (PFAS) and a Mixture of Four PFAS (Final)." Office of Water, Office of Science and Technology, Health and Ecological Criteria Division, EPA-815-R-24-004. 366p., April.

US EPA Region III. 1991. "Regional Guidance on Handling Chemical Concentration Data Near the Detection Limit in Risk Assessments (Interim Final)." November 4. Accessed at https://www.epa.gov/risk/regional-guidance-handling-chemical-concentration-data-near-detection-limit-risk-assessments.

US EPA Region VI; US EPA Region V. 2008. "RCRA Delisting Technical Support Document." Multimedia Planning and Permitting Division, Land and Chemicals Division, Report to US EPA, Office of Solid Waste. 202p., October 31.

Weston Solutions, Inc. 2023. "Instream PFAS Characterization Study Final Report, Mississippi River, Cottage Grove, Minnesota (Draft)." Report to 3M Co. W.O. No. 02181.002.230.0001, 467p., June 30.

Wisconsin Dept. of Natural Resources (WDNR). 2022. "Surface Water Quality Standards for PFOS and PFOA Rule Package Technical Support Document (Rule package WY-23-19, related to Chapters NR 102, 105, 106, and 219, Wis. Adm. Code)." 73p., January 21.

Attachment 1

Curriculum Vitae of Robyn Prueitt, Ph.D., DABT

A full copy of Dr. Prueitt's CV was provided as part of Exhibit G to 3M's August 2024 Comments and is incorporate by reference herein.



Curriculum Vitae of Tim Verslycke, Ph.D.

A full copy of Dr. Verslycke's CV was provided as part of Exhibit G to 3M's August 2024 Comments and is incorporated by reference herein.

EXHIBIT 2

Supplemental Arcadis Report



Technical Memorandum

SUBJECT

Supplemental Technical Review 3M Cottage Grove Advanced Wastewater Treatment System PFAS Treatment System Technical Memorandum

DATE

February 3, 2025

FROM

Joseph Quinnan, PE, PG, Senior Vice President/Director of Emerging Contaminants Keith Foster, Principal¹

Introduction and Purpose

The law firm of Thomas, Combs, and Spann retained Arcadis on behalf of 3M to provide this supplemental technical review and comment on the capabilities of the advanced wastewater treatment system (AWWTS) currently under construction at 3M Chemical Operations LLC's Cottage Grove facility (the Facility), specifically in connection with the permit limits proposed by the Minnesota Pollution Control Agency (MPCA) in both a draft permit MN0001449, issued in July 2024 (July Draft Permit; MPCA 2024a) and, more recently, a revised draft permit MN0001449, issued in December 2024 (Revised Draft Permit; MPCA 2024b). The AWWTS is being constructed at the 3M site in Cottage Grove, Minnesota to treat industrial wastewater before it is discharged to an unnamed creek in the Mississippi River watershed.

The following sections summarize Arcadis' technical review of the Revised Draft Permit with respect to the capabilities of the AWWTS, which were previously described in Arcadis' August 2024 Technical Review of 3M Cottage Grove Advanced Wastewater Treatment (AWWTS Technical Review; Arcadis, 2024), attached hereto as Ex. A. The analysis provided in the AWWTS Technical Review is not repeated in this Supplemental Technical Review but is incorporated by reference herein.

The AWWTS Technical Review included Arcadis' review of the Draft Permit (MPCA, 2024a), the per- and polyfluoroalkyl (PFAS) Treatability Study Report (Treatability Report) submitted to MPCA by Emerging Compounds Treatment Technologies (ECT2) and Barr on behalf of 3M (ECT2 and Barr 2021) as well as the Design Basis Report submitted to MPCA by ECT2 and Toltz, King & Day (TKDA; ECT2 and TKDA 2023. This supplemental technical review includes the following elements:

- A summary of applicable permitting considerations as specified in the Revised Draft Permit;
- An assessment of whether the Treatability Report supports that the AWWTS system can meet the new ultralow PFAS limits specified in the Revised Draft Permit; and
- A summary of the findings of this supplemental technical review.

¹ Corey Theriault, a co-author of the AWWTS Technical Review, has departed Arcadis and thus is not a co-author of this memorandum.



Review of the Revised Draft Permit

On December 18, 2024, the MPCA published the Revised Draft Permit for public comment. With respect to the AWWTS and PFAS treatment, two specific modifications were provided in the Revised Draft Permit:

- The WQBELs were recalculated to address certain issues identified during the original comment period, which increased the WQBELs for PFOA and PFHxS while the limit for PFOS remained unchanged; and
- Changes were made to the mass limits for the five PFAS parameters with limits at SD 001 and SD 002 to correspond to the new WQBELs. The mass limit values in the Revised Draft Permit were also corrected to grams per day (g/day) instead of milligrams per day, as previously calculated in the July Draft Permit.

Table 1 provides a summary of the updated WQBELs and the mass limit values for PFHxS, PFOS, and PFOA in the Revised Draft Permit.

Table 1 Revised Draft Permit WQBELs and Mass Limit Values

Analyte	SD 001			SD 002		
	WQBELs ¹		Mass Limits ²	WQBELs ¹		Mass Limits ²
	Daily Maximum (ng/L)	Calendar Month Average (ng/L)	Calendar Month Average (g/day)	Daily Maximum (ng/L)	Calendar Month Average (ng/L)	Calendar Month Average (g/day)
PFHxS	0.021	0.012	0.0030	0.021	0.012	0.004
PFOS	0.066	0.038	0.0093	0.066	0.038	0.0012
PFOA	0.080	0.046	0.0011	0.080	0.046	0.0015

Notes:

- 1 Revised Draft Permit WQBELs for sampling locations SD001 and SD002.
- 2 Revised Draft Permit mass limits for sampling locations SD001 and SD002.

Although the WQBELs for PFHxS and PFOA increased, they are still one to two orders of magnitude (OOM) lower than the best LOQs reported in the Treatability Report. See Ex. A at p. 5.

On December 18, 2024, the MPCA also published the National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit Program Fact Sheet for Permit Reissuance of Permit MN0001449 (Revised Fact Sheet; MPCA 2024c). Within the Revised Fact Sheet, it was noted that the design and antidegradation flows had been updated to 6.5 MGD for SD 001 and 8.7 MGD for SD 002 for a total of 15.2 MGD. These flows, in conjunction with the calendar month average WQBEL values were used to derive the mass limits for PFOA, PFOS and PFHxS.

The Revised Draft Permit and AWWTS Treatment Capabilities

As was discussed in the AWWTS Technical Review, the analytical results provided in the Treatability Report indicate that the combined treatment technologies of the AWWTS are effective at removing a variety of PFAS including short-chain compounds; however, the data did not provide support for a conclusion that the proposed



treatment system would meet the Draft Permit <u>WQBELs</u> for PFHxS, PFOS, and PFOA due to limitations in analytical capabilities (i.e., the LOQs in each of the laboratory results <u>reviewed in the Treatability Study</u> were higher than the WQBELs specified in the Draft Permit). See Ex. A at p. 27. In fact, in establishing Compliance Limits for PFOA, PFOS and PFHxS at SD 001 and SD 002, the MPCA acknowledged this challenge (MPCA 2024a). Although the WQBELs were increased in the Revised Draft Permit for PFOA and PFHxS, they remain below the LOQs in each of the laboratory results <u>reviewed in the Treatability Study</u>.

Furthermore, the mass limits that were recalculated in the Revised Draft permit rely on the WQBELs. That is, the mass limits are calculated simply by multiplying the design flow at each discharge point by the calendar month average WQBEL for the compound of interest. As a result, the mass limits pose the same operational and analytical challenges as the WQBELs, which are described in our previous report. And, for the same reasons, there is also no evidence in the record that the AWWTS can achieve the mass limits in the Revised Draft Permit. See Ex. A at p. 26-27.

Arcadis has also reviewed the Declaration of Michael J. Parent (hereinafter "Parent Declaration", Ex. 3 to 3M's Comments to the Revised Draft Permit and incorporated herein by reference) in which Dr. Parent calculated the estimated degree of treatment, expressed as percent (%) removal, that would be required by the AWWTS to achieve the mass limits in the Revised Draft Permit. The calculations show that even considering only a portion of the total water and PFAS to be treated (i.e., the load from the Woodbury wells) the degree of treatment needed ranges from >99.99% to >99.999% removal depending on the PFAS compound. See Parent Declaration p. 3. Arcadis has reviewed and agrees with the basis of these calculations and notes that while the AWWTS has demonstrated a degree of treatment capable of meeting the LOQs published in the Treatability Study, Arcadis is not aware of an application where these technologies have shown that they can provide a degree of treatment to the levels calculated in Parent Declaration.

Summary

As previously discussed in the AWWTS Technical Review, the AWWTS represents significant engineering and financial commitments and was designed to provide reliable, sustainable, and maximum extent practicable levels of treatment of the Cottage Grove Facility water. The AWWTS, a state-of-the-art and industry-exceeding PFAS treatment system, incorporates three field-implemented PFAS removal technologies that will treat, on average, approximately 15.2 MGD. While the AWWTS exceeds the industry standard for PFAS treatment, and was approved by the MPCA, the proposed WQBELs and mass limits in the Revised Draft Permit have not been demonstrated to be achievable for the Cottage Grove Facility water with the technologies included, and it is unclear what additional technologies could be installed at this scale that would provide this degree of treatment.

References

Arcadis. 2024. Final Technical Review of 3M Cottage Grove Advanced Wastewater Treatment System.

ECT2 and Barr. 2021. PFAS Treatability Study, 3M Cottage Grove, MN Facility.

ECT2 and TKDA. 2023. Design Basis Report for Cottage Grove Water Stewardship Water Quality Project-Regeneration System.



MPCA. 2024a. Draft National Pollutant Discharge Elimination System and State Disposal System Permit MN0001449. State of Minnesota. Available at https://scs-public.s3-us-gov-west-1.amazonaws.com/env_production/oid333/did200071/pid_209149/project-documents/Draft%20Permit%20-%20Public%20 Notice%20-%20MN0001449%20-%20202.pdf.

MPCA. 2024b. Draft National Pollutant Discharge Elimination System and State Disposal System Permit MN0001449. State of Minnesota. Available at https://scs-public.s3-us-gov-west-1.amazonaws.com/env_production/oid333/did200071/pid_210210/project-documents/Draft%20Permit%20-%20MN0001449%20-%202024.pdf

MPCA. 2024c.National Pollutant Discharge Elimination System (NPDES)/State Disposal System (SDS) Permit Program Fact Sheet. Permit MN0001449. State of Minnesota. Available at https://scs-public.s3-us-gov-west-1.amazonaws.com/env_production/oid333/did200071/pid_210210/project-documents/Fact%20Sheet%20-%20MN0001449%20-%202024.pdf

Submitted by,

Arcadis U.S., Inc.

Joseph Quinnan, PE, PG Senior Vice President

Director of Emerging Contaminants

Keith Foster Principal

EXHIBIT A

Arcadis Expert Report was attached as Exhibit D at 3M's August 30 Comments



3M Cottage Grove

Final

Technical Review of 3M Cottage Grove Advanced Wastewater Treatment System

August 2024

Final – Technical Review of 3M Cottage Grove Advanced Wastewater Treatment System

August 2024

Prepared By:

Arcadis U.S., Inc. 630 Plaza Drive, Suite 300 Highlands Ranch, CO 80129 United States

Phone: 205 930 5700

Our Ref: 30238952

Corey Theriault, PE

National Technical Manager

Keith Foster

Principal Geologist

Lauren March, PE

Project Chemical Engineer

Contents

1	Scop	pe of Work	1
2	Reg	ulatory Framework	2
2	.1	Overview of Draft Permit	2
3	Ove	rview of Treatment Systems	6
3	.1	Existing Treatment System Overview	6
3	.2	Proposed Treatment System Overview	8
	3.2.1	Systems A and B	8
	3.2.2	IX Resin Regeneration, Regenerant Recovery and Concentration System	. 11
4	Data	Summary	. 12
5	PFA	S Removal Technology Review	. 24
5	.1	Technology Selection and Industry Acceptance	. 24
5	.2	Documented Industry Performance	. 25
5	.3	Expected Technology Performance vs Draft Permit Conditions	. 26
6	Sum	mary	. 28
7	Refe	rences	. 29
Ta	able	es	
Tab	le 2-1	Intervention limits, WQBELs, CLs, and LOQs (MPCA 2024)	5
Tab	le 3-1	AWWTP PFAS Removal Technology Design Basis	9
Tab	le 4-1	Summary of the 16 PFAS Compounds Analysed by Enthalpy Analytical	. 12
Tab	le 4-2	Summary of PFAS Concentrations during the NCCW/SW Pilot Test Phases	. 14
Tab	le 4-3	Summary of PFAS Concentrations in Split Samples during the NCCW/SW Pilot Test Phases	. 15
Tab	le 4-4	Summary of PFAS Concentrations during the Phase 1/2 WW Pilot Test Phase	. 17
Tab	le 4-5	NCCW/SW RO PFAS Reject Efficiencies by Test Phase	. 18
Tab	le 4-6	Phase 1/2 WW RO PFAS Reject Efficiencies	19
Tab	le 4-7	Bed Volumes to First Detection of Breakthrough for NCCW/SW Test Phases	. 20
Tab	le 4-8	BVs to First Detection of Breakthrough for the Phase 1/2 WW Test Phase	. 21
Tab	le 4-9	Estimated Treated Effluent Water Quality Based on Treatability Study	. 22
Tab	le 5-1	PFAS Removal Performance by Technology	. 26
Tah	le 5-2	Pilot Test and Full-Scale Design Basis Parameters	26

Figures

Figure 2-1	Pilot Test Source Water Locations	4
Figure 2-2	Locations of Internal Waste Streams (WS) Stations in Process Flow	5
Figure 3-1	WWTP Process Flow Diagram	
Figure 3-2	Block Flow Diagram of Ttreatment Systems Included in Systems A and B	10

Appendices

Appendix A Arcadis Resumes

1 Scope of Work

The law firm of Hogan Lovells retained Arcadis¹ on behalf of 3M to provide technical review and comment on the capabilities of the advanced wastewater treatment system currently under construction at 3M Chemical Operations LLC's Cottage Grove facility (the Facility), specifically in connection with the intervention and compliance limits proposed by the Minnesota Pollution Control Agency (MPCA) draft permit MN0001449 (Draft Permit). The proposed treatment system is intended to be installed at a site in Cottage Grove, Minnesota to treat industrial wastewater before being discharged to an unnamed creek in the Mississippi River watershed.

The following sections comprise Arcadis' technical review of the per- and polyfluoroalkyl (PFAS) Treatability Study Report (Treatability Report) submitted to MPCA by Emerging Compounds Treatment Technologies (ECT2) and Barr on behalf of 3M (ECT2 and Barr 2021) as well as the Design Basis Report (BOD) submitted to MPCA by ECT2 and Toltz, King & Day (TKDA; ECT2 and TKDA 2023). MPCA approved these submissions. This technical review includes the following details:

- A summary of applicable permitting considerations as specified in the Draft Permit;
- An overview of the existing and proposed treatment systems;
- A summary and analysis of the Treatability Report data relevant to the Draft Permit;
- A comparison of the proposed treatment system to accepted industry standards;
- An assessment of whether the proposed treatment system can meet the ultra-low PFAS limits specified in the Draft Permit; and
- A summary of the technical review findings.

.

The CVs of the authors of this Report are attached as **Appendix A**.

2 Regulatory Framework

2.1 Overview of Draft Permit

Relevant to this analysis, the Draft Permit sets Facility discharge limits for perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA), and perfluorohexanesulfonic acid (PFHxS). Sampling locations are displayed in Figure 2-1 and Figure 2-2 (MPCA 2024). Section 4 of the Draft Permit provides a summary of stations and station locations including effluent to surface water stations SD001, SD002, and SD003. As shown on Figure 2-1, SD001 encompasses process and sanitary effluent; SD002 includes non-contact cooling water (NCCW), groundwater (GW), and industrial stormwater (ISW); and SD003 includes outfalls from SD001 and SD002 combined. Additionally, the Draft Permit includes a description of internal waste streams WS001 and WS002 as shown on Figure 2-2. WS001 is sampled after the process and sanitary anion exchange (IX) lag vessel and before mixing into SD001 at Building 151. WS002 is sampled after the NCCW, GW, and ISW IX lag vessel and before mixing into SD002 at Building 151. Note that the current treatment system includes no further treatment after WS001 and WS002.

Water Quality-based Effluent Limitations (WQBELs), Compliance Limits, and Intervention limits, are displayed in **Table 2-1**. Each of these limits have a specific role under the Draft Permit. The WQBELs are limit values derived by MPCA based upon its analysis of levels required to ensure achievement of the State's designated uses. The Compliance Limits are values adopted by MPCA that are deemed acceptable to demonstrate compliance with certain WQBELs that are below the limits of quantitation of MPCA's preferred laboratory analytical method (EPA Method 1633). The Intervention Limits are values applied at specific sampling locations, exceedances of which trigger specified actions by operators of the wastewater treatment system.

Section 5.69.128 of the Draft Permit defines compliance limits (CLs) as follows:

"Compliance limit (CL)" shall mean: The value deemed as in compliance with the Daily Maximum and Monthly Average PFAS limits. The monthly average and daily maximum PFOS WQBELs are below the reporting limits (limits of quantitation) achievable when analyzing treated effluent at Cottage Grove. For PFOS, a statistical analysis of the actual reporting limit wastewater at Cottage Grove sampling stations SD001 and SD002 is 2.2 ng/L. For PFOA and PFHxS, the actual reporting limit is 2.1 ng/L. For these three parameters, any effluent value less than or equal to the numbers above will be considered in compliance with the daily maximum limit; and any monthly average effluent value equal to or below the numbers above will be considered to be in compliance with the monthly average limits.

Section 5.33 of the Draft Permit provides the following intervention limit requirements:

- Sampling requirements in the case of an intervention limit is exceedance (e.g., resample the monitoring station within 2 days of receipt of sample results indicating exceedance);
- Evaluation of the significance and probable cause of the exceedance including a review of media changeout schedule;
- Proposed immediate corrective action to prevent future exceedances;
- Proposed change in monitoring schedule (e.g., increased sampling frequency, additional analytes, additional monitoring points); and.
- Submission of an intervention limit exceedance evaluation report within 30 days of receipt of sample results indicating exceedance

The Draft Permit indicates that an exceedance of an intervention limit does not constitute a permit violation; however, failure to respond to the intervention limit exceedances as described above constitutes a permit violation.

In summary, exceedances of PFHxS, PFOS, and PFOA above 2.1, 2.2, and 2.1 nanograms per liter (ng/L), respectively, would constitute a permit violation, as shown in **Table 2-1**. Section 5.73.198 of the Draft Permit provides additional effluent limitations and requirements and describes WQBELs as follows:

Water quality-based effluent limits shall be dependent on receiving water, discharge volume, in-stream flow volume, and discharge time, duration and location. The MPCA shall notify the Permittee if it is determined that additional requirements, more or less stringent limits, and/or monitoring are appropriate for a specific water body. The MPCA's letter notifying the Permittee of these additional requirements... shall then become a part of the enforceable requirements applicable through this permit for the specific discharge point and the Permittee shall comply with these requirements.

Note that the Treatability Report uses the term limit of detection (LOD), while the Draft Permit uses limit of quantitation (LOQ). Arcadis received the following communication from John Berry, representing ECT2, addressed to Christopher Bryan, representing 3M, which summarizes an explanation provided by representatives of Enthalpy Analytical on the use of LOD versus LOQ:

The LOQ is effectively determined by the range of concentrations calibrated on the instrument. The LOD can be determined in numerous ways, the most common of which is to spike samples and use statistical methods to determine a limit of detection. However, in some cases, such as when a method is new, an LOD study will not have yet been executed, in which case the LOD will be set to the same value as the LOQ. This is the case for the PFAS Treatability Study for Cottage Grove dated December 22, 2021.

The terms LOD and LOQ are not synonymous, but they may have the same value depending on the circumstances. For the purposes of this discussion, we will use LOQ to refer to analytical limits (i.e., any occurrence of LOD from the Treatability Report cited herein will be replaced with LOQ for terminology consistency).

It is important to note that the intervention limits and WQBELs specified in the Draft Permit are well below the CLs also specified in the Draft Permit as well as the LOQs found in the Treatability Report for PFOS, PFOA, and PFHxS. Currently, the lowest LOQs for PFAS compounds analyzed via common analytical methods (e.g., United States Environmental Protection Agency [USEPA] Method 1633, 537.1, 8421) are typically in the single digit parts per trillion (ppt) order of magnitude (OOM). This contrasts with the intervention limits and WQBELs specified by the Draft Permit, which are one to three OOMs lower, making them effectively unenforceable.

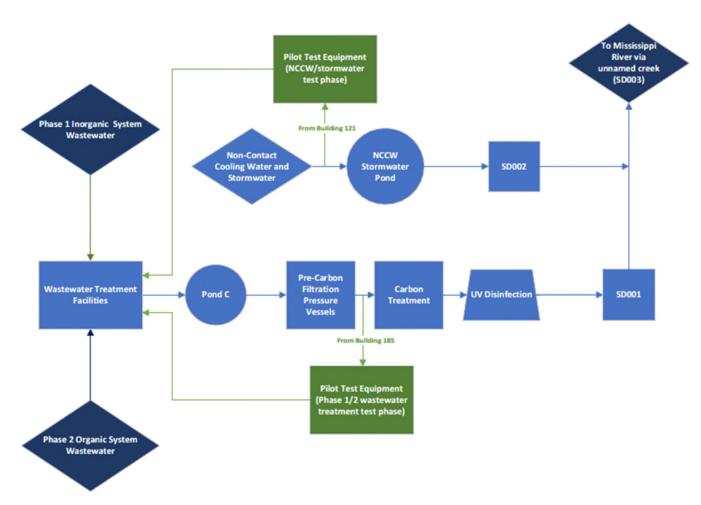


Figure 2-1 Pilot Test Source Water Locations
Source: ECT2 and Barr 2021, Figure 2.2

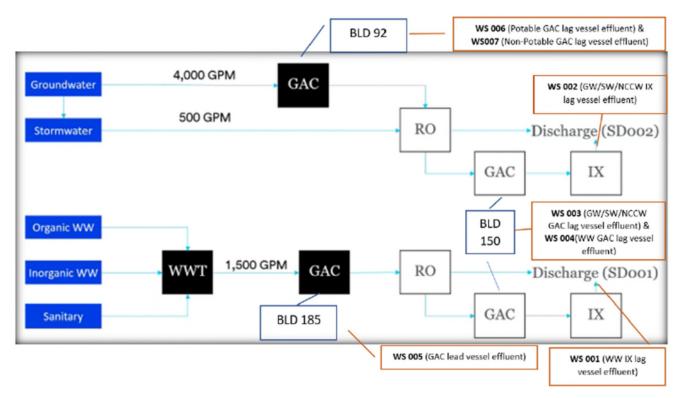


Figure 2-2 Locations of Internal Waste Streams (WS) Stations in Process Flow Source: MPCA 2024, Figure 7

Table 2-1 Intervention limits, WQBELs, CLs, and LOQs (MPCA 2024).

	Intervention Limits ¹		wc	QBELs ^{2,3}	Compliance Limits ³	LOQ Range⁴
Analyte	Daily Maximum (ng/L)	Calendar Month Average (ng/L)	Daily Maximum (ng/L)	Maximum Month Average		(ng/L)
PFHxS	0.0298	0.0171	0.0056	0.0032	2.1	<1.93 - <2,390
PFOS	0.27	0.155	0.066	0.038	2.2	<1.41 – <7,311
PFOA	0.117	0.069	0.022	0.013	2.1	<0.122 - <2,210

- 1 Draft Permit intervention limits for PFOA, PFOS, and PFHxS at sampling locations WS001 and WS002 as shown on Figure 2-2.
- 2 Draft Permit WQBELs for sampling locations SD001, SD002, and SD003 as shown on Figure 2-1.
- 3 Enforceable CLs, exceedances of which would constitute a violation of the Draft Permit.
- 4 LOQ ranges as specified in the Treatability Report.

3 Overview of Treatment Systems

This section provides a brief description of pertinent existing water treatment systems and a summary of the proposed PFAS treatment systems.

3.1 Existing Treatment System Overview

3M currently operates an existing wastewater treatment plant (WWTP). Process wastewater generated from production facilities, pilot production wastewaters, and sanitary wastewater are treated at the facility WWTP. These waters are treated at three separate WWTP systems, referred to as Phases, depending on their relevant liquid characteristics. The Phase 2 treatment system processes organic wastewater from manufacturing processes while the Phase 1 treatment system processes effluent from Phase 2, inorganic wastewater from manufacturing, and landfill leachate. The effluent of Phase 1 is then routed to a granular activated carbon (GAC) system, followed by ultraviolet light, before discharge at Outfall SD001. The Phase 3 treatment system previously treated scrubber wastewater from a former 3M hazardous waste incinerator at the Facility and currently treats drainage from drying beds, incinerator decommissioning waters, and select stormwater collected at the Facility. Effluent from the Phase 3 treatment system is routed to a separate GAC system to treat PFAS before discharge at Outfall SD001 (MPCA 2024).

In addition to the process streams identified above, 3M also manages NCCW, ISW, and GW at the Facility. Both NCCW and ISW were previously discharged to an unlined NCCW retention pond before discharge. Contaminated GW from the 3M Cottage Grove Facility, as well as the Woodbury Disposal site, is extracted from extraction wells and treated through a GAC system. Effluent from this GAC system is used throughout the Cottage Grove Facility for cooling water, process water, and other building/site water requirements. The following block diagram (**Figure 3-1**) shows the current WWTP process flows (MPCA 2024).

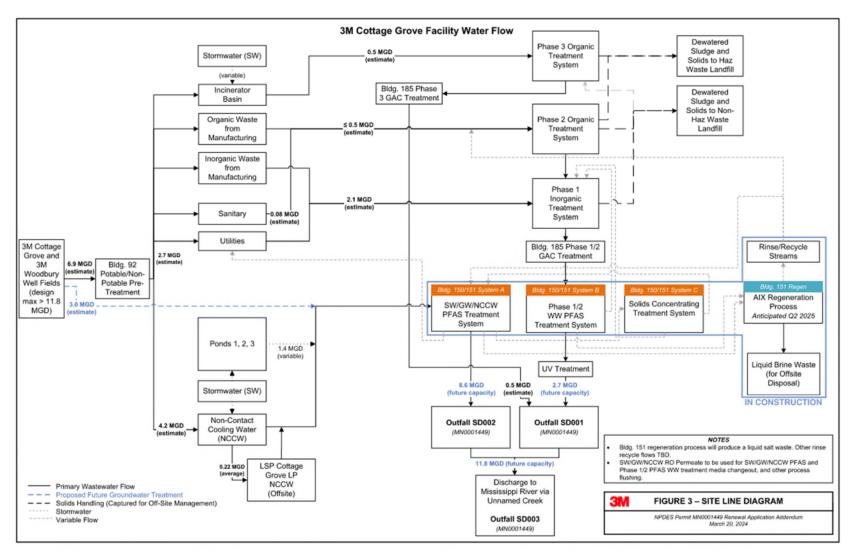


Figure 3-1 WWTP Process Flow Diagram
Source: MPCA 2024, Figure 5

3.2 Proposed Treatment System Overview

3M has proposed to install an advanced wastewater treatment system (AWWTS), which encompasses two discrete PFAS treatment systems (Systems A and B) and a third IX resin regeneration and regenerant recovery and concentration system. A fourth system (System C) includes a solids concentrating treatment system for System A solids management. Together, these treatment systems will treat approximately 11 million gallons per day (MGD) of GW (from the Cottage Grove Facility and Woodbury disposal site well fields), ISW, NCCW (System A) and Phase 1/2 treatment system effluent (System B). Because the design basis of System C is focused on solids management and not direct PFAS treatment, it is not further discussed herein. As discussed herein, the AWWTS incorporates a best-in-class approach to consistent treatment of PFAS and management of PFAS waste materials, based on the particular characteristics of the composition of the 3M wastewater. When first operated, the AWWTS will represent almost four years of testing, design, and construction at a cost of approximately \$275,000,000.

The following sections provide a narrative of the process streams and a description of the process units/technologies included in the design for each system.

3.2.1 Systems A and B

Influent water for System A includes GW, ISW, and NCCW with a design flow rate of 8.28 MGD. Influent water for System B includes WWTP Phase 1/2 effluent with a design flow rate of 2.95 MGD. Although the resulting treatment processes are generally the same for both systems, due to the different characteristics of the water in the process streams, 3M designed two separate treatment systems to allow for optimum design and operability. Had the source waters been combined and routed to a singular system, the unique differences in the water chemistry, flow rates, and pre-treatment requirements may have resulted in inconsistent operation of the combined system. In general, both systems include the following unit processes (ECT2 and Barr 2021):

Pre-filtration:

- Pre-filtration for System A, which appears to contemplate potential treatment for algal growth in NCCW pond; and
- Pre-filtration for System B includes the existing glass filter media before the existing GAC treatment system for Phase 1/2.
- Ultra Filtration (UF):
 - UF is being used to protect the reverse osmosis (RO) membranes from excessive fouling. UF backwash streams will be sent to a solids-concentration system, and concentrated solids will be returned to the existing WWTP.

RO:

- Three RO stages are included in the design to enable a wider range of PFAS recovery in light of the PFAS composition of 3M's effluent.
- RO concentrate will be treated using GAC and regenerable IX resin. The treated RO concentrate will be combined with the RO permeate and discharged to Outfalls SD001 and SD002, respectively.

GAC:

 In the treatment configuration utilized, GAC adsorption will be optimized to remove primarily longchain PFAS from the RO concentrate stream before IX treatment. Short chain PFAS compounds will also be removed during this treatment step, however, the design intent of this step is for the removal of long chain PFAS compounds.

IX Resin:

- o In the treatment configuration utilized, regenerable IX resin will be optimized to remove short-chain PFAS from the RO concentrate. Long chain PFAS compounds will also be removed during this treatment step, however, the design intent of this step is for the removal of short chain PFAS compounds.
- Each IX resin "train" will consist of three adsorbent vessels connected in series. The first vessel will contain SORBIX A3F IX resin. The second and third vessels will contain a "secondary high-capacity microporous media."

Table 3-1 provides a summary of the PFAS removal technologies included in the AWWTP (MPCA 2024):

Table 3-1 AWWTP PFAS Removal Technology Design Basis

Parameter	System A	System B
	Reverse Osmosis System	
Recovery (% to permeate)	85%	85%
Active Area (ft²)	400	400
Stages / Total Elements Per Skid (5 skids)	3 / 252	3 / 108
Total active area per skid (ft²)	100,800	43,200
Design Flux (GFD) / Design Flow Rate (gpm)	14 / 5,750	11.6 / 2,050
	GAC	
Treatment Trains/ Vessels per Train	4/2	2/2
Vessel Diameter (ft)	10	10
Mass of GAC/vessel (lbs)	20,000	20,000
Empty Bed Contact Time/vessel (mins)	30	30
Total Design Flowrate (gpm)	576	192
Surface Loading Rate (gpm/ft²)	2.4	2.4
	IX Resin	
Treatment Trains/ Vessels per Train	7/3	3/3
Vessel Diameter (ft)	6	6
Volume of IX resin/vessel (ft³)	360	360
Empty Bed Contact Time/vessel (mins)	20	20
Total Design Flowrate (gpm)	675	270
Surface Loading Rate (gpm/ft²)	4.8	4.8

Abbreviations and Acronyms:

ft = feet

 ft^2 = square feet

ft3= cubic feet

GFD = gallons per square foot per day

gpm = gallons per minute

mins = minutes

Source: MPCA 2024

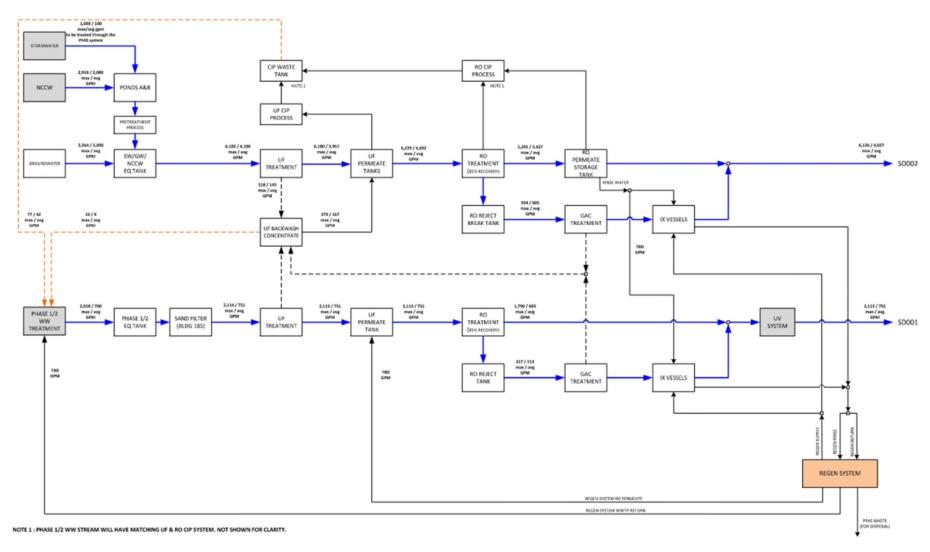


Figure 3-2 Block Flow Diagram of Treatment Systems Included in Systems A and B Source: ECT2 and Barr 2021, Large Figure 5

3.2.2 IX Resin Regeneration, Regenerant Recovery and Concentration System

As noted above, use of GAC, IX resin, and RO is based on site-specific features and the PFAS composition in 3M's effluent. To remove PFAS from the RO concentrate, the concentrate passes through both GAC and regenerable IX resin, as indicated in Section 4.2.1. To provide consistent and reliable treatment of PFAS, while minimizing waste disposal of PFAS-laden adsorbent materials (i.e., single-use IX resin or GAC), a regenerable IX resin was selected. This process also allows for a unique operational approach, as the timing between IX regenerations can be tailored to specific PFAS compound effluent concentrations. For the AWWTS, this operational approach is centered around regenerating IX resin once short-chain PFAS compounds, such as trifluoroacetic acid (TFA) and perfluorobutyric acid (PFBA), are likely to first be detected in the IX resin effluent. Operating under this approach offers several benefits, two of which are:

- Removing and treating the bulk of the PFAS mass, which is primarily composed of short-chain PFAS
 compounds; and
- 2. Ensuring treatment of longer-chain PFAS, such as PFHxS, PFOS, and PFOA, as these compounds are removed more efficiently than their shorter-chain counterparts.

This innovative process has been deployed to a limited extent in the United States (Wastewater Digest 2021) and Australia (Wastewater Digest 2020), typically at rates on the order of 50 to 200 gpm. The AWWTS represents a significant expansion in scale of this technology, of which there are no other known regenerable IX systems of this size in the world outside of 3M.

The regeneration process consists of removing individual treatment vessels from operation and pumping a mixture of solvent, water, and salt through the vessels to desorb and remove the PFAS compounds, thereby restoring the capacity of the resin to continue to treat PFAS. After the PFAS compounds have been removed, the IX resin is rinsed with treated water (RO permeate) and the vessels are then placed back in normal treatment service for continued PFAS removal. The spent regenerant solution is then processed through a solvent recovery system to recover the solvent for reuse in the system. The still bottoms (STB) are processed through a STB RO unit and a brine concentrating unit, which concentrates the salt and PFAS into a smaller volume that is subsequently collected and disposed of off-site.

RO permeate from the STB RO system will be routed back to the head of the WWTP for further treatment. The STB RO reject will be processed through a thin film evaporator. The evaporator boils off water and other light-end materials from the STB RO reject, producing a concentrated liquid of salt and PFAS. Evaporator overhead vapors are condensed, subcooled, and recycled back to the Phase 1 WWTP with the RO permeate from the STB RO. The concentrated brine exits the evaporator and is pumped to a storage tank for off-site disposal at a hazardous waste site. **Figure 3-2** is a block flow diagram showing how the regeneration system is incorporated into the design for Systems A and B (ECT2 and Barr 2021, Large Figure 5).

4 Data Summary

In connection with its application for a construction permit, 3M submitted the Treatability Report to MPCA. As stated in Section 1.2 of the Treatability Report, the purpose of the Treatability Study was to assess the efficacy of treating PFAS in wastewater at the Facility using commercially available technologies. The Treatability Study was not designed to discern operational limits for PFAS in the treatment system effluent. This section provides a summary of the pilot study data presented in the Report (ECT2 and Barr 2021).

Two different source waters were tested during the pilot testing phase of the Treatability Study, including NCCW/SW effluent (SD002), which was sampled before the NCCW and SW pond, and phase 1/2 WW, which was sampled between the pre-carbon filtration pressure vessels and existing carbon treatment system, as shown on **Figure 2-1**. **Table 4-1** presents a summary of the 16 PFAS compounds analyzed by Enthalpy Analytical. The dominant compounds detected, making up more than 90 percent of the PFAS mass in the NCCW/SW and Phase 1/2 WW streams, include TFA, trifluoromethanesulfonic acid (TFMS), perfluorophosphonic acid (PFPA), bis(trifluoromethane)sulfonimide (HQ-115), and PFBA.

Table 4-1 Summary of the 16 PFAS Compounds Analysed by Enthalpy Analytical

Group No.1	Abbreviation	Full Name
Group 1		
1	TFA	Trifluoroacetic acid
2	TFMS	Perifluoromethanesulfonate
3	2,2,3,3-TFPA	2,2,3,3-Tetrafluoropropionic acid
4	2,3,3,3-TFPA	2,3,3,3-Tetrafluoropropionic acid
5	PFPA	Perfluoropropionic acid
6	HQ-115	Methanesulfonamide, 1,1,1-trifluoro-N-[(trifluoromethyl)sulfonyl-
7	PFBA	Perfluorobutyric acid
8	PFPeA	Perfluoropentanoic Acid
Group 2	'	
9	PFBS	Perfluorobutanesulfonate
10	PFPeS	Perfluoropentanesulfonate
11	PFHxA	Perfluorohexanoic acid
12	PFHpA	Perfluoroheptanoic acid
13	PFHxS	Perfluorohexanesulfonate
14	PFHpS	Perfluoroheptanesulfonate
15	PFOA	Perfluorooctanoic acid
Group 3		
16	PFOS	Perfluorooctanesulfonate

Notes:

Source: ECT2 and Barr 2021, Table 2.4.

¹ Groups 1, 2, and 3 were established in the Treatability Plan based on the number of carbon atoms, the number of fluorinated carbons, and the physical characteristics of the PFAS. These groups were established to estimate the treatability of specific PFAS for which publicly available treatability information is not available.

Table 4-2 summarizes the PFAS results from samples collected at multiple locations including the pilot influent UF feed, UF permeate, RO permeate, RO concentrate, lag GAC effluent, lag CalRes ion exchange resin effluent (IX2), and lag SORBIX ion exchange resin effluent (IXR2) during the NCCW/SW pilot test phase. At a high level, these results show the efficacy of PFAS removal using the different technologies. PFHxS, PFOS, and PFOA are highlighted yellow in **Table 4-2** for clarity. Note that all three compounds identified in the Draft Permit are shown as non-detect (ND) in the pilot influent UF feed, rendering the results inconclusive as far as the pilot treatment system's ability to achieve the Draft Permit compliance limits. Of the three compounds, PFOS has the lowest LOQ range of <200 – <2,000, which is two OOMs higher than the Draft Permit Compliance Limit shown in **Table 2-1**. This indicates that, even if there was PFHxS, PFOS, and/or PFOA detected in the pilot influent UF feed and the IX2 and IXR2 effluent remained ND, there is no assurance that the pilot effluent would meet the Draft Permit compliance limits for discharge.

Table 4-3 provides a summary of split samples taken during the NCCW and SW test phases and analyzed by 3M's Global EHS Laboratory (3M Lab). HQ-115 and PFBA were detected separately in the two RO permeate samples collected. For the Enthalpy Analytical samples shown in **Table 4-3**, no PFAS were detected in the corresponding RO permeate split samples. Eighteen different PFAS (FBSA, FOSA, HQ-115, PECHS, PFBA, PFBS, PFES, PFHpA, PFHpS, PFHxS, PFHxA, PFOA, PFPA, PFPeA, PFPeS, PIBA, TFA, and TFMS) were detected at concentrations above LOQs in the RO concentrate samples. For the corresponding Enthalpy Analytical split samples, only six PFAS were detected (PFPA, PFBA, PFPeA, PFBS, HQ-115, and TFMS). These observations may be attributed in part to the lower LOQs found in the 3M Lab samples.

PFHxS, PFOS, and PFOA are highlighted yellow in **Table 4-3** for clarity. In contrast to the Enthalpy results, in which all three compounds identified in the Draft Permit were reported as ND in the pilot influent UF feed sample, only one of the three compounds (PFOS) was reported as ND in the pilot influent feed, rendering the results inconclusive as far as the pilot treatment system's ability to achieve the Draft Permit PFOS compliance limit. However, PFHxS and PFOA were detected in the influent at 54.6 and 62.8 ng/L, respectively, and were ND in the RO permeate, IX2, and IXR2 samples. These results indicate that the pilot treatment system may be able to achieve the Draft Permit PFHxS and PFOA compliance limits. These results provide more assurance than the Enthalpy results, but the LOQs for all three compounds in the 3M Lab results are higher than the corresponding compliance limits. For instance, of the two compounds detected in the influent, PFOA has the lowest LOQ range of <9.6 – <19.2, which is four to nine times higher than the Draft Permit compliance limit shown in **Table 2-1**. This indicates that it is inconclusive whether the pilot effluent would meet the Draft Permit compliance limits for discharge.

Table 4-4 shows a summary of the PFAS results from samples collected at multiple locations during Phase 1/2 WW pilot test phase. PFHxS, PFOS, and PFOA are highlighted yellow in **Table 4-4** for clarity. Note that two of the three compounds identified in the Draft Permit, PFHxS and PFOA, are shown as ND in the pilot influent UF feed, rendering the results inconclusive as far as the pilot treatment system's ability to achieve the Draft Permit compliance limits for those two compounds. PFOS was detected in the pilot influent UF feed at up to 1,360 ng/L. The PFOS LOQs for the IXR2 samples ranged from <1.60 ng/L to <200 ng/L. Because 1.6 ng/L is below the Draft Permit compliance limit for PFOS, this provides some assurance that the pilot effluent would meet the Draft Permit compliance limits for discharge of PFOS as shown in **Table 2-1**.

Table 4-2 Summary of PFAS Concentrations during the NCCW/SW Pilot Test Phases

				NCCW/SW Co	ncentration F	Ranges (minimum	and maximum)		
PFAS	Units	LOQ Range	Pilot Influent UF feed	UF Permeate	RO Permeate ⁶	RO Concentrate	Lag GAC Effluent (GAC2)	Lag CalRes Effluent (IX2)	Lag SORBIX Effluent (IXR2)
Sum of 16 Analyzed PFAS ⁷	ng/L		ND-27,000	7,790–99,000	ND-6,200	47,400–795,000	ND-225,000	ND-21,900	ND-52,000
'				Gr	oup 1				
TFA	ng/L	<2.29-<69,853	ND	ND	ND	ND- 14,900	ND- 4,750	ND-17,900	ND
TFMS	ng/L	<346-<10,000	ND-10,800	1,600-11,400	ND-1,310	14,900-174,000	ND-195,000	ND	ND
2,2,3,3-TFPA	ng/L	<1,000-<17,897	ND	ND	ND	ND	ND	ND	ND
2,3,3,3-TFPA	ng/L	<752-<14,840	ND	ND	ND	ND	ND	ND-2,790	ND-1,890
PFPA	ng/L	<8.42-<51,058	ND- 7,520	1,390-5,910	ND	ND- 44,900	ND- 51,700	ND-16,000	ND-21,200
HQ-115	ng/L	<2.61-<10,000	ND- 27,000	ND- 82,700	ND-6,200	13,500-480,000	ND-8	ND	ND
PFBA	ng/L	<191-<1,910	ND- 8,060	398-8,450	ND- 70	7,890-76,600	ND- 76,100	ND	ND-38,100
PFPeA	ng/L	<212-<2,120	ND- 561	ND- 717	ND	1,240-10,100	ND	ND	ND
				Gr	oup 2				
PFBS	ng/L	<444-<4,440	ND-12,900	ND-17,700	ND	ND- 17,100	ND	ND- 19	ND
PFPeS	ng/L	<31.1-<2,580	ND	ND- 41	ND	ND- 811	ND	ND- 36	ND
PFHxA	ng/L	<241-<2,410	ND	ND- 61	ND	ND- 2,660	ND	ND	ND
PFHpA	ng/L	<152-<1,520	ND	ND	ND	ND- 40	ND	ND	ND
PFHxS	ng/L	<239-<2,390	ND	ND	ND	ND- 5,610	ND	ND	ND
PFHpS	ng/L	<169-<1,690	ND	ND	ND	ND- 222	ND	ND	ND
PFOA	ng/L	<221-<2,210	ND	ND	ND	ND- 11,200	ND	ND	ND
				Gr	oup 3				
PFOS	ng/L	<200-<2,000	ND	ND	ND	ND- 11,800	ND	ND	ND

- 1. Data are from Enthalpy Analytical.
- 2. ng/L = nanograms per liter (equivalent to parts per trillion or ppt)
- 3. LOQ = limit of detection
- 4. ND = non-detect or below LOQ
- 5. Bold values are concentrations detected above the LOQ.
- 6. During test phase NCCW_D only (95% RO recovery). TFMS, HQ-115, PFBA were detected in the RO permeate.
- 7. Sum of 16 Analyzed PFAS only includes the PFAS detected at concentrations above the LOQ.

Source: ECT2 and Barr, 2021, Table 3.3

Table 4-3 Summary of PFAS Concentrations in Split Samples during the NCCW/SW Pilot Test Phases

		NCCW/SW Concentration Ranges (minimum and maximum)—Split Samples Only								
PFAS	Units	LOQ Range	Pilot Influent UF Feed (n=1)	UF Permeate (n=4)	RO Permeate (n=2)	RO Concentrate (n=5)	Lag CalRes Effluent (IX2) (n=1) ⁵	Lag SORBIX Effluent (IXR2 (n=1) ⁵		
<u> </u>				Group 1						
TFA	ng/L	<200	3,360	3,040-3,320	ND	17,000-21,600	19,800	19,800		
TFMS	ng/L	<25.0	3,160	1,280-3,140	ND	8,560-14,600	ND	ND		
2,2,3,3-TFPA	ng/L	<500	ND	ND	ND	ND	ND	ND		
2,3,3,3-TFPA	ng/L	<1,000	1,210	ND	ND	ND	ND	ND		
PFPA	ng/L	<50.0	3,180	1,800-3,300	ND	9,840-16,200	ND	4,320		
PFES	ng/L	<25.0	73.2	ND- 71	ND	74.8–322	ND	ND		
HQ-115	ng/L	<10.0	236	256-4,440	ND-111	1,430-74,000	ND	ND		
PFBA	ng/L	<10.0	8,000	482-8,120	ND- 25.2	8,700-36,800	ND	ND		
PIBA	ng/L	<100	123	ND-109	ND	139–334	ND	ND		
PFPeA	ng/L	<10.0	502	ND- 526	ND	560-2,140	ND	ND		
'	-	'		Group 2		<u>'</u>				
PFBS	ng/L	<9.0-<10.0	ND	ND	ND	ND	ND	ND		
FBSA	ng/L	<10.1	ND	ND	ND	ND-13.4	ND	ND		
PFBS	ng/L	<10.0	142	ND-147	ND	546-13,800	ND	ND		
PFPeS	ng/L	<9.4	45	ND- 44.2	ND	96.8–256	ND	ND		
MeFBSA	ng/L	<39.4-<44.0	ND	ND	ND	ND	ND	ND		
FBSE	ng/L	<45.6-<51.0	ND	ND	ND	ND	ND	ND		
MeFBSAA	ng/L	<44.8-<50.0	ND	ND	ND	ND	ND	ND		
MeFBSE	ng/L	<17.9-<20.0	ND	ND	ND	ND	ND	ND		
PBSA	ng/L	<9.0-<10.0	ND	ND	ND	ND	ND	ND		
FBSEE-Diol	ng/L	<44.8-<50.0	ND	ND	ND	ND	ND	ND		
FBSEE-DA	ng/L	<9.0-<10.0	ND	ND	ND	ND	ND	ND		
FBSAA	ng/L	<100	ND	ND	ND	ND	ND	ND		
PBSA-DC	ng/L	<10.7-<12.0	ND	ND	ND	ND	ND	ND		
PFHxA	ng/L	<10.0	173	ND-182	ND	204–740	ND	ND		
PFHpA	ng/L	<10.0	27.2	ND- 28	ND	34.4–82.2	ND	ND		

Table 4-3 Summary of PFAS Concentrations in Split Samples during the NCCW/SW Pilot Test Phases

		NCCW/SW Concentration Ranges (minimum and maximum)—Split Samples Only								
PFAS	Units	LOQ Range	Pilot Influent UF Feed (n=1)	UF Permeate (n=4)	RO Permeate (n=2)	RO Concentrate (n=5)	Lag CalRes Effluent (IX2) (n=1) ⁵	Lag SORBIX Effluent (IXR2) (n=1) ⁵		
PFHxS/PFHS	ng/L	<10.0-<20.0	54.6	ND- 35.6	ND	94.2–300	ND	ND		
PFHpS	ng/L	<10.0	ND	ND	ND	ND- 23.6	ND	ND		
PHSA-C	ng/L	<89.5-<100	ND	ND	ND	ND	ND	ND		
PFOA	ng/L	<9.6-<19.2	62.8	ND- 69.4	ND	173–324	ND	ND		
		,		Group 3			,			
FOSA/PFOSA	ng/L	<10.0	ND	ND	ND	ND- 45.2	ND	ND		
PFOS	ng/L	<8.3-<9.3	ND	ND	ND	ND	ND	ND		
PECHS	ng/L	<9.2	14.5	ND	ND	31.2–76.2	ND	ND		

- 1. Data from 3M Global EHS Laboratory.
- 2. No data available from GAC effluent.
- 3. n =the number of split samples collected at the specified location.
- 4. ND = non-detection
- 5. Sample collected after 212 bed volumes treated across the lag vessel.

Source: ECT2 and Barr, 2021, Table 3.14.

Table 4-4 Summary of PFAS Concentrations during the Phase 1/2 WW Pilot Test Phase

				Phase 1/2 WW	PFAS Concentra	tion Ranges (minimum	and maximum)		
PFAS	Units	LOQ range	Pilot Influent UF Feed	UF Permeate	RO Permeate	RO Concentrate	Lag GAC Effluent (GAC2)	Lag CalRes Effluent (IX2)	Lag SORBIX Effluent (IXR2)
Sum of 16 Analyzed PFAS ⁵	ng/L		97,800– 202,000	74,800–181,000	1,420–3,180	1,064,000-2,31,000	6,500–1,780,000	ND-11,000	ND-12,400
'				'	Group 1				
TFA	ng/L	<700-<23,461	ND	ND	ND	ND	ND	ND	ND
TFMS	ng/L	<18.4-<1000	65,900– 166,000	46,900–145,000	1,050-3,090	827,000-1,850,000	ND-1,770,000	ND	ND- 290
2,2,3,3-TFPA	ng/L	<373-<19,129	ND	ND	ND	ND	ND	ND	ND
2,3,3,3-TFPA	ng/L	<122-<31,656	ND	ND-1,610	ND	ND- 7,300	ND- 7,920	ND	ND
PFPA	ng/L	<20.8-<63,771	ND- 2,420	ND- 10,100	ND- 34	ND- 44,000	ND- 105,000	ND-11,000	ND-12,400
HQ-115	ng/L	<0.734-<102	17,000–24,100	13,400-20,800	92–157	128,000-259,000	ND-8	ND- 20	ND- 21
PFBA	ng/L	<8.17-<1,053	1,500-3,160	1,740-2,960	ND- 10	12,400-26,500	ND	ND	ND
PFPeA	ng/L	<12.5-<1,062	ND	ND-111	ND- 5	ND- 680	ND	ND	ND
					Group 2		<u> </u>		
PFBS	ng/L	<4.43-<2,219	2,870-16,200	3,570–15,200	ND- 84	34,800-143,000	ND	ND	ND
PFPeS	ng/L	<1.75-<1,288	ND	ND	ND- 80	ND- 848	ND	ND- 37	ND
PFHxA	ng/L	<0.718-<2,087	ND	ND	ND	ND- 127	ND	ND	ND
PFHpA	ng/L	<0.612-<1,056	ND	ND	ND	ND	ND	ND	ND
PFHxS	ng/L	<1.93-<1,194	ND	ND- 33	ND	ND- 5,540	ND	ND	ND
PFHpS	ng/L	<2.17-<3,375	ND	ND	ND	ND- 102	ND	ND	ND
PFOA	ng/L	<0.122-<221	ND	ND- 34	ND	ND- 5,080	ND	ND	ND
					Group 3				
PFOS	ng/L	<1.41-<7,311	ND-1,360	ND	ND	ND- 8,940	ND	ND	ND

- 1. ng/L = nanograms per liter (equivalent to parts per trillion or ppt)
- 2. LOQ = limit of detection
- 3. ND = non-detect or below LOQ
- 4. Bold values are concentrations detected above the LOQ.
- 5. The Sum of 16 Analyzed PFAS only includes the PFAS detected at concentrations above the LOQ.

Source: ECT2 and Barr 2021, Table 3.9. Data are from Enthalpy Analytical.

Table 4-5 summarizes the PFAS rejection efficiencies of the RO membrane for the eight PFAS compounds found at concentrations above the LOQ in the UF permeate during the three NCWW test phases (ECT2 and Barr 2021). PFAS reject efficiencies refer to the mass of PFAS eliminated from the RO permeate by the RO membrane as defined in Section 1.4 of the Treatability Report (see Equation 1). Where the RO permeate PFAS concentration was below the LOQ, the reject efficiency was calculated using the nominal LOQ value. In these cases, the reject efficiency is shown as greater than (>) the calculated rejection efficiency, meaning that the actual reject efficiency is likely greater than the value calculated using the LOQ.

(1) Reject Efficiency
$$\% = \frac{RO\ Influent\ PFAS\ Conc. - RO\ Permeate\ PFAS\ Conc.}{RO\ Influent\ PFAS\ Conc.} \times 100\%$$

Table 4-5 NCCW/SW RO PFAS Reject Efficiencies by Test Phase

DEAS Paintin	Test Phase						
PFAS Rejection Efficiencies ³	NCCW_A (n=7) ⁴	NCCW_B (n=5) ⁴	NCCW_D (n=1) ⁴				
PFPA	>49.6%->75.5%	>50.7%->74.0%	7				
PFBA	>94.4%->97.7%	>52.0%->86.9%	99.7%				
PFPeA	>24.6% ->70.4%	5	>0%6				
PFHxA	5	5	7				
PFBS	6	>97.5%6	7				
PFPeS	5	5	7				
HQ-115	>64.9%->98.8% ⁶	>35.9%->91.5%	97.0%				
TFMS	>66.6%->91.2%	>37.5%-86.0%	95.5%				

Notes:

- The "-- " symbol indicates not applicable; the reject efficiency could not be calculated because the RO influent (UF permeate)
 PFAS concentration was below the LOQ.
- 2. The ">" symbol indicates that the concentration in the RO permeate was below the LOQ.
- 3. This table summarizes only data reported by Enthalpy Analytical.
- 4. The number of samples shown (n) indicates the number of paired samples collected within 4 hours of each other from the RO influent (UF permeate) and the RO permeate.
- The reject efficiency could not be calculated in at least one sample because the RO influent (UF permeate) PFAS concentration was below the LOQ.
- 6. >0% indicates that the reported concentration in the RO permeate was below the LOQ, and the concentration in the RO influent was equivalent to the nominal LOQ value.
- 7. The PFAS were detected in the RO influent, and concentrations were below the LOQ in the RO permeate, but the PFAS reject efficiency is not reported because the nominal LOQ value in the RO permeate was greater than the detected concentration in the RO influent.

Source: ECT2 and Barr 2021, Table 3.4

Table 4-6 summarizes the PFAS reject efficiencies of the RO membrane during the 1/2 WW testing phase. However, only PFAS compounds that were observed at concentrations above LOQs in the UF permeate are summarized. Where the RO permeate PFAS concentration was below the LOQ, the reject efficiency was calculated using the nominal LOQ value. In these cases, the rejection efficiency is shown as greater than (>) the calculated rejection efficiency, meaning that the actual rejection efficiency is likely greater than the value calculated using the LOQ.

Table 4-6 Phase 1/2 WW RO PFAS Reject Efficiencies

PFAS Rejection Efficiencies	Phase 1/2 WW Test Phase (n=3) ²
2,3,3,3-TFPA	3,4
PFPA	>51.0%–98.2% ³
PFBA	>84.4%–99.7%³
PFPeA	95.5% ^{3,4}
PFOA	>55.6%³
PFBS	>85.4%–99.1%
PFPeS	3
PFHxS	3,4
HQ-115	98.8%–99.4%
TFMS	96.9%–98.1%

- The ">" symbol indicates that the concentration in the RO permeate was below the LOQ.
- 2. The number of samples shown (n) indicates the number of paired samples collected simultaneously from the RO influent (UF permeate) and the RO permeate.
- In at least one sample, the reject efficiency could not be calculated because the RO influent (UF permeate) PFAS concentration was below the LOQ.
- 4. In at least one sample, PFAS was detected in the RO influent, and concentrations were below the LOQ in the corresponding RO permeate. The PFAS reject efficiency is not reported because the nominal LOQ value in the RO permeate was greater than the detected concentration in the RO influent.

Source: ECT2 and Barr 2021, Table 3.10

Table 4-7 summarizes the number of bed volumes to the first detection of breakthrough for each of the NCCW/SW test phases. Table 4-5 and Table 4-7 do not include PFOS, PFOA, or PFHxS because the analytical results for these compounds were ND throughout the NCCW/SW phases of testing. This indicates that the resin and GAC changeout schedule should be driven by the breakthrough of compounds shown in Groups 1 and 2, which are anticipated to break through before the compounds specified in the Draft Permit. As such, it is not recommended to monitor PFOA, PFOS, and PFHxS to determine the performance of the GAC and/or IX systems. Arcadis recommends considering compounds that were shown to have low bed volumes (BVs) before breakthrough and also detected at high concentrations in the influent stream; TFMS, PFPA, and PFBA make up about 70 percent of the total detections in the influent stream as analyzed by 3M Global EHS Laboratory in Table 4-3. Additionally, breakthrough of TFMS was observed for GAC1 and GAC2, breakthrough of PFBA was observed at all sample locations aside from IX2, and breakthrough of PFPA was observed at all sample locations. Thus, TFMS, PFPA, and PFBA would be appropriate surrogate compounds to drive the media changeout schedule; however, operational experience and/or additional data may suggest monitoring of additional compounds.

Table 4-7 Bed Volumes to First Detection of Breakthrough for NCCW/SW Test Phases

PFAS ⁵	Lead GAC (GAC1)	Lag GAC (GAC2)	Lead CalRes (IX1)	Lag CalRes (IX2)	Lead SORBIX (IXR1)	Lag SORBI) (IXR2)				
NCCW_A – BVs to Media Column Breakthrough, up to 1,639 BVs across the Lead Vessel										
Group 1										
TFMS	295	148	not observed	not observed	not observed	not observed				
2,3,3,3-TFPA	not observed	not observed	487	244	487	244				
PFPA	295	148	679	580	487	340				
HQ-115	1,838	not observed	not observed	not observed	not observed	not observe				
PFBA	295	148	1,159	not observed	679	484				
PFPeA	1,159	not observed	not observed	not observed	not observed	not observe				
N	CCW_B - BVs to	Media Column B	reakthrough, up	to 471 BVs acros	s the Lead Vesse					
			Group 1							
TFA	6	6	not observed	not observed	INT	not observe				
2,3,3,3-TFPA	6	6	135	116	135	164				
PFPA	6	6	471	not observed	183	212				
PFBA	6	6	not observed	not observed	231	not observe				
N	CCW_D - BVs to	Media Column B	reakthrough, up	to 238 BVs acros	s the Lead Vesse					
			Group 1							
TFA	INT	INT	293	INT	not observed	not observe				
TFMS	46	23	not observed	not observed	not observed	not observe				
HQ-115	94	147	not observed	not observed	not observed	not observe				
PFPA	46	23	293	INT	not observed	not observe				
PFBA	94	119	not observed	not observed	not observed	not observe				
			Group 2	1						
PFBS	not observed	not observed	not observed	INT	not observed	not observe				
PFPeS	not observed	not observed	not observed	INT	not observed	not observe				

- 1. Not observed = breakthrough was not observed up to the BVs tested.
- 2. INT = intermittent detections, but a consistent breakthrough curve was not apparent.
- 3. BV is a unitless measure of the volume of water treated through a media filter; it is equal to the volume of water treated divided by the volume of the media bed. As a result, BVs shown for lag columns are half those shown for lead columns on a given date because the same flow has gone through twice as much media by the time it reaches the lag column effluent compared to lead column effluent. However, BVs shown for IX do not consider upstream GAC volume.
- 4. The first breakthrough is defined as the first detection above LOQ, with subsequent measurements consistently as high or higher.
- 5. For PFAS not listed in this table, breakthrough was not observed during the test phases.
- 6. BVs to breakthrough of the GAC columns are not shown for NCCW_B because the media beds were not changed out between test phases NCCW_A and NCCW_B. If breakthrough was observed during NCCW_B, the BV to breakthrough is shown under NCCW_A to reflect continuous GAC operation through the two phases.

Source: ECT2 and Barr 2021, Table 3.6

Table 4-8 summarizes the number of bed volumes to the first detection of breakthrough for the Phase 1/2 WW test phase. **Table 4-8** shows that no breakthrough of PFOS, PFOA, or PFHxS was observed at any of the sample points throughout testing. This indicates that the resin and GAC changeout schedule should be driven by the breakthrough of compounds shown in Group 1, which are anticipated to break through before the compounds specified in the Draft Permit. As such, PFOA, PFOS, and PFHxS are not appropriate compounds to use for determining the performance of the GAC and/or IX systems. Arcadis recommends considering compounds that were shown to have low BVs before breakthrough and also detected at high concentrations in the influent stream; TFMS and PFPA were detected in the influent stream at 3,160 ng/L and 3,180 ng/L, respectively, as analyzed by 3M Global EHS Laboratory in **Table 4-3**. Additionally, breakthrough of TFMS was observed for GAC1, GAC2, IXR1, and IXR2, while breakthrough of PFPA was observed at all sample locations. Thus, TFMS and PFPA would be appropriate surrogate compounds to drive the media changeout schedule.

Table 4-8 BVs to First Detection of Breakthrough for the Phase 1/2 WW Test Phase

PFAS⁵	Lead GAC (GAC1)	Lag GAC (GAC2)	Lead CalRes (IX1)	Lag CalRes (IX2)	Lead SORBIX (IXR1)	Lag SORBIX (IXR2)			
	BVs to Media Column Breakthrough, up to 496 BVs across the Lead Vessel								
			Group 1						
TFMS	8	28	not observed	not observed	434	INT			
2,3,3,3-TFPA	INT	INT	not observed	not observed	not observed	not observed			
PFPA	112	49	INT	INT	242	200			
HQ-115	INT	INT	INT	INT	INT	INT			
PFBA	194	not observed	not observed ⁶	not observed	not observed	not observed			
PFPeA	not observed ⁶	not observed	not observed ⁶	not observed	not observed	not observed			
	'		Group 2						
PFBS	not observed	not observed	not observed ⁶	not observed	not observed	not observed			
PFPeS	not observed	not observed	not observed ⁶	INT	INT	not observed			
PFHpA	not observed ⁶	not observed	not observed ⁶	not observed	not observed	not observed			
PFHxS	not observed ⁶	not observed	not observed ⁶	not observed	not observed	not observed			
PFHpS	not observed	not observed	not observed ⁶	not observed	not observed	not observed			
PFOA	not observed ⁶	not observed	not observed ⁶	not observed	not observed	not observed			
			Group 3						
PFOS	not observed	not observed	not observed ⁶	not observed	not observed	not observed			

Notes:

- 1. Not observed = breakthrough was not observed up to the BVs tested.
- 2. INT = intermittent detections, but a consistent breakthrough curve was not apparent.
- 3. BV is a unitless measure of the volume of water treated through a media filter. It is equal to the volume of water treated divided by the volume of the media bed. As a result, BVs shown for lag columns are half those shown for lead columns on a given date because the same flow has gone through twice as much media by the time it reaches the lag column effluent compared to lead column effluent. However, BVs shown for IX do not consider upstream GAC volume.
- 4. The first breakthrough is defined as the first detection above LOQ, with subsequent measurements consistently as high or higher.
- 5. For PFAS not listed in this table, breakthrough was not observed during the test phase.
- 6. One sample had low detections of multiple PFAS, but seven of eight did not have later detections or breakthroughs, suggesting possible sample contamination. As a result, any PFAS only detected in this sample were judged not to have broken through. These samples were from lead GAC column at 56 BVs and lead CalRes column at 386 BVs.

Source: ECT2 and Barr 2021, Table 3.12

Table 4-9 presents an initial estimate of the full-scale system's treatment capacity in terms of effluent water quality. To generate this estimate, it was assumed that the full-scale system effluent would consist of 85 percent RO permeate water and 15 percent IX lag vessel effluent. As shown in the highlighted rows of the table, the lowest LOQs for PFHxS, PFOS, and PFOA are 5 ng/L, 15 ng/L, and 4 ng/L, respectively – all of which are higher than the Draft Permit compliance limits shown in **Table 2-1**. This indicates that, based on the Treatability Study, we do not have assurance that the proposed full-scale treatment system will meet the Draft Permit compliance limits.

Table 4-9 Estimated Treated Effluent Water Quality Based on Treatability Study

Source Water (Test Phase)	NCCW	/SW (NCCV	V_B)	Phas	se 1/2 WW (W	'W)				
# of BVs	98	212	212	97	241	241				
IX Resin	SORBIX/CalRes	SORBIX	CalRes	SORBIX/CalRes	SORBIX	CalRes				
	General Chemistry ¹									
Calcium		62			54					
Iron+ Manganese		<0.055			<0.055					
TOC		3.6			3.5					
TDS		367			1,150 ⁷					
TSS		<10			14 ³					
рН		5.9-8.6			6.3–8.6					
			PFAS⁴							
Sum of 16 Detected PFAS ⁵		4,218	3,570	1,807	3,385	2,069				
			Group 1 ⁶							
TFA	< 700	< 3,140 ⁶	< 3,140 ⁶	< 700	< 2,150 ⁶	< 1,775 ⁶				
TFMS	< 1,000	< 498	< 498	< 1,811 ⁶	< 276	< 276				
2,2,3,3-TFPA	< 1,000	< 500	< 500	< 2,406	< 500	< 500				
2,3,3,3-TFPA	< 752	< 1,000	< 1,000	< 740	< 1,000	< 1,000				
PFPA	< 700	< 691 ⁶	< 50	< 700	< 1,039 ⁶	<98 ⁶				
HQ-115	< 1,000	< 83	< 83	133 ⁶	< 104	< 104				
PFBA	< 191	<116	<11 ⁶	< 260	< 10	< 10				
PFPeA	< 212	< 10	< 10	< 17	< 10	< 10				
			Group 2 ⁶							
PFBS	< 444	<16 ⁶	<16 ⁶	< 9	< 36	< 36				
PFPeS	< 258	< 9	< 9	< 2	< 9	< 9				
PFHxA	< 241	< 10	< 10	< 2	< 10	< 10				
PFHpA	< 152	< 10	< 10	< 24	< 10	< 10				
PFHxS	< 239	< 239 < 10 < 10		< 5	< 10	< 10				
PFHpS	< 169	< 10	< 10	< 6	< 10	< 10				
PFOA	< 221	< 18	< 18	< 15	< 18	< 18				

Table 4-9 Estimated Treated Effluent Water Quality Based on Treatability Study

Source Water (Test Phase)	NCCW	//SW (NCC\	W_B)	Phase 1/2 WW (WW)						
Group 3 ⁶										
PFOS	< 200	< 9	< 9	< 4	< 9	< 9				

- 1. Effluent concentrations are estimated as a weighted average of RO permeate concentrations and IX lag column effluents and not intended to include regeneration waste. BVs indicated are for lag vessels. The early BV is generally before breakthrough and thus similar for both resins, while IX effluent concentrations varied among resins at higher BVs.
- 2. General chemistry is based on water quality sampling events for NCCW_B and WW test phases and is not expected to vary significantly by IX BV.
- 3. Effluent total suspended solids (TSS) concentration is biased by IX effluent TSS concentration measured at 59 to 71 milligrams per liter (mg/L). That concentration is unlikely to have passed through all four media vessels and may reflect precipitation of minerals between sampling and analysis.
- 4. PFAS data for end-of-pilot samples (236 BVs for NCCW phase and 241 BVs for WW phase) reflect 3M data, which typically had lower detection limits than Enthalpy data. The initial sample for each water source is Enthalpy data because 3M did not collect data for these events.
- 5. Sum of 16 PFAS detected only includes parameters detected above Enthalpy LOD for that sample.
- 6. Values for which one of the source readings was above LOD are bolded. For weighted averages with a different LOD, the LOD indicated here is the weighted average of LODs. For weighted averages with one sample above LOD, the LOD indicated here is the weighted average of the LOD and the detection.
- 7. Estimated total dissolved solids (TDS) for treated Phase 1/2 WW includes 60 mg/L of NaCl added with regeneration waste brine recycled back to Phase 1/2 WW influent.

Source: ECT2 and Barr 2021, Table 3.16

In summary, the analytical results provided in the Treatability Report indicate that these combined treatment technologies are effective at removing a variety of PFAS, including short-chain compounds; however, these data do not provide sufficient evidence to support a conclusion that the proposed treatment system will meet the Draft Permit WQBELs or Intervention Limits for PFHxS, PFOS, and PFOA due to limitations in analytical capabilities (i.e., the LOQs in almost all of the laboratory results reviewed in the Treatability Study were higher than the limits specified in the Draft Permit). Furthermore, the Treatability Report results suggest that PFHxS, PFOS, and PFOA are not appropriate compounds to monitor for purposes of ensuring compliance with the ultimate discharge limits. Instead, Arcadis recommends monitoring compounds that were shown to have low BVs before breakthrough and detected at high concentrations in the influent stream (i.e., TFMS, PFPA, and PFBA) to drive the media changeout schedule.

5 PFAS Removal Technology Review

This section provides a comparison of the proposed treatment system to accepted industry standards, documented industry performance of the proposed technologies, and an assessment of whether the proposed treatment system can meet the ultra-low PFAS limits specified in the Draft Permit.

5.1 Technology Selection and Industry Acceptance

Arcadis reviewed the design and capability of 3M's proposed AWWTS and compared it against other available technologies for treating PFAS in water. In many PFAS treatment applications the industry standard is to use a single technology such as GAC, IX resin, or RO (among others) to remove PFAS. In drinking water, the industry standard has been to use GAC or IX resin. EPA has also noted that while RO also meets the definition of Best Available Technology under the Safe Drinking Water Act, EPA "does not anticipate water systems will select this technology to comply with the rule, largely due to the challenges presented by managing the treatment residuals from this process." 89 Fed. Reg. 32532, 32654 (Apr. 26, 2024). As the water chemistry becomes more complicated due to site-specific issues such as (i) effluent with a much more complex PFAS composition (including type and concentration), (ii) the presence of co-contaminants (e.g., metals, volatile organic compounds, 1,4-dioxane) and/or (iii) background chemistry (e.g., elevated concentrations of salts, organic matter, solids), a treatment train approach involving multiple technologies has also been deployed in limited situations. In addition, because of the inherent challenges involved in treating PFAS, including treating PFAS to parts per trillion levels and minimizing the handling of concentrated PFAS waste (e.g., spent GAC, IX resin, concentrated brine), the use of multiple technologies can provide a more reliable and sustainable treatment process.

In the case of the AWWTS, 3M selected RO as the primary PFAS treatment technology. RO was selected in large part due to its ability to remove a broad spectrum of PFAS (i.e., both short- and long-chain) compounds present in 3M wastewater effluent that are atypical compared to most water treatment scenarios, including drinking water. A challenge with RO resides in the PFAS removal mechanism relying on size exclusion which rejects PFAS, ions, and some water and results in a low-volume (when compared to the RO permeate) stream of concentrated PFAS brine (salt) liquid waste. To remove PFAS from this low-volume concentrated PFAS stream, 3M has proposed to install GAC treatment followed by regenerable IX resin. The GAC treatment train is optimized and operated to remove long-chain PFAS compounds, such as PFOS and PFOA, while the regenerable IX resin is optimized and operated to remove short-chain PFAS compounds such as TFA and PFBA. In conventional IX resin systems, once the IX resin is no longer capable of providing PFAS treatment, the IX resin is removed, sent off site for disposal, and new IX resin is placed within the treatment vessel. In the case of the AWWTS, a regenerable IX resin system is proposed to be installed, which offers three significant benefits:

- The IX resin's ability to treat PFAS can be restored in place, ensuring continuous treatment.
- Spent regenerant solution (see Section 4.2.2) is distilled and re-used.
- PFAS-containing waste is further concentrated, and its volume is reduced.

The Interstate Technology and Regulatory Council (ITRC), in their 2023 PFAS Technical/Regulatory Guidance Document (ITRC 2023), has identified RO, GAC, and IX resin as field-implemented liquids treatment technologies. The ITRC goes on further to explain field-implemented technologies as being:

 Implemented in the field by multiple parties at multiple sites, and the results have been well-documented in practice or peer reviewed literature; and Applied to a variety of PFAS-impacted media including drinking water (regardless of source), surface water, groundwater, wastewater, stormwater, or landfill leachate.

An evaluation prepared for the MPCA by Barr Engineering Co. and Hazen and Sawyer entitled Evaluation of Current Alternatives and Estimated Cost Curves for PFAS Removal and Destruction from Municipal Wastewater, Biosolids, Landfill Leachate, and Compost Contact Water (Barr and Hazen & Sawyer 2023) evaluated multiple PFAS separation and destruction technologies. In this evaluation, RO, GAC, and regenerable IX resin were retained as technologies to evaluate for PFAS-impacted liquids treatment due to their deployment at field scale, commercial availability, and demonstrated performance of removing at least 90 percent of at least one selected PFAS compound. The study set treatment targets at 5 ng/L, which is 127 to 138 percent higher than the proposed compliance limits and 3,126 to 29,140 percent higher than the proposed intervention limits. The study recognized that "targeting analytical reporting limits for removal in this Report is aggressive" and that "many beneficial projects may target mass removal of total PFAS or long-chain PFAS" (Barr and Hazen & Sawyer 2023).

Thus, given the summary of the pilot test data described in Section 3 herein, and the acknowledgement by the technical community (e.g., ITRC and MPCA) of these technologies' real-world use in treating PFAS, 3M's decision to use all three of these technologies for the AWWTP offers a robust and industry-exceeding solution to meet the particulars of 3M's effluent composition. Furthermore, based on our expertise in designing, constructing, operating, and evaluating PFAS treatment systems, we are not aware of another equivalent-sized (i.e., 11 MGD) system that encompasses three discrete PFAS removal technologies and combines them in as innovative and sustainable a manner as the Cottage Grove AWWTS. That is, most PFAS systems simply focus on the removal of PFAS from water but do not contemplate waste treatment, management, and minimization to the scale done so by the AWWTS.

5.2 Documented Industry Performance

The focus of the review in this section is in relation to PFOA, PFOS, and PFHxS; the three PFAS compounds having proposed compliance and/or intervention limits that are at concentrations below their respective AWWTS influent concentrations (see Section 3). Other PFAS compounds will also be treated by the AWWTS; however, the analysis herein of documented industry performance for RO, GAC, and regenerable IX resin has been limited to PFOA, PFOS, and PFHxS.

The way by which technologies remove PFAS governs how they are evaluated in their treatment efficacy. RO, which treats PFAS via size exclusion, is typically evaluated by "percent rejection" or "rejection efficiency" as shown in Equation 1 in Section 4 – Data Summary.

It is generally accepted that this percent rejection efficiency will continue until the RO membranes require replacement at the end of their useful service lives, which is typically on the order of 2 to 5 years. GAC and regenerable IX resins, which remove PFAS by adsorptive processes, are typically evaluated on the duration of time during which the adsorbents remove PFAS to a level below a specified limit, such as a laboratory reporting or permit limit. This is commonly referred to as "bed volumes to breakthrough." A BV is a measurement of volume of influent water equal to the volume of adsorbent media within an adsorbent reactor vessel. BVs to breakthrough for GAC and regenerable IX resins depend on the nature (chain length and functional group) and concentrations of the PFAS compounds, but typically are on the order of thousands to hundreds of thousands of BVs for PFOA, PFOS, and PFHxS. Typically, these sorts of BV capacities represent an operational timeline of months to a few years.

Table 5-1 was developed from information provided in the Barr and Hazen & Sawyer 2023 evaluation and summarizes the PFAS removal by technology.

Table 5-1 PFAS Removal Performance by Technology

PFAS Compound	RO (% Rejection)	GAC (BVs)	Regenerable IX (BVs)	
PFHxS	>80 – 99	3,000 – 100,000	21,000	
PFOS	>71 – 99	3,000 – 100,000	21,000	
PFOA	>77 – 98	3,000 - 100,000	13,000	

Source: Barr and Hazen & Sawyer 2023

5.3 Expected Technology Performance vs Draft Permit Conditions

As discussed in Section 4, 3M undertook a pilot study that evaluated treating PFAS-impacted water with RO, GAC, and regenerable IX resins. Samples were taken from the influent and effluent of each technology, which provides perspective on how the AWWTS is reasonably expected to perform at scale barring no significant differences, none of which are expected, of the treatment conditions (water chemistry and hydraulics) between the pilot study and full-scale systems. The differences between the pilot test conditions and the design basis are summarized in **Table 5-2** (ECT2 & Barr 2021 and MPCA 2024).

Table 5-2 Pilot Test and Full-Scale Design Basis Parameters

Unit Process	Design Parameter	Pilot Test System A	Design Basis System A	Pilot Test System B	Design Basis System B
RO	Flux	14 GFD	14 GFD	12 GFD	11.6 GFD
	RO Recovery	85%	85%	85%	85%
GAC	EBCT	60 minutes across two vessels			
	Surface Loading Rate	0.9 gpm/ft ²	2.4 gpm/ft ²	0.9 gpm/ft ²	2.4 gpm/ft ²
IX Resin	EBCT	60 minutes across two vessels			
	Surface Loading Rate	0.9 gpm/ft ²	4.8 gpm/ft ²	0.9 gpm/ft ²	4.8 gpm/ft ²

Note:

EBCT = empty bed contact time

Source: ECT2 & Barr 2021, MPCA 2024

Based on **Table 5-2**, there does not appear to be significant differences between the hydraulic conditions of the pilot tests and the full-scale design. The 1.5 gallons per minute per square foot (gpm/ft²) and 3.9 gpm/ft² differences in surface loading rates for GAC and IX resin, respectively, between the pilot tests and full-scale design basis are not expected to impact PFAS removal performance.

As the pilot test and full-scale design conditions are relatively similar, it is thus expected that the treatment performance of the unit processes will be similar. **Table 4-2** and **Table 4-4** provide a summary of the pertinent analytical data, as analyzed by Enthalpy Analytical, collected during the pilot tests. **Table 4-3** summarizes the results

of split samples analyzed by 3Ms Global EHS Laboratory. As shown in the data, effluent concentrations of PFOA, PFHxS, and PFOS in the RO permeate (future discharge locations SD 001 and SD 002), lag GAC (future discharge locations WS 003 and WS 004), and lag IX resin (future discharge locations WS 001 and WS 002) were routinely below their LOQs. It should be noted that, in many instances, the LOQs were elevated. A variety of factors can cause PFAS LOQs to be elevated and may include general water quality characteristics that interfere with the instrumentation's ability to measure PFAS in the single-digit parts per trillion concentration range.

As discussed in Section 3.1, the WQBELs for PFOA, PFHxS, and PFOS are below the LOQs of current commercially available PFAS analytical techniques, rendering their implementation impractical. In fact, in establishing the compliance limits for SD 001 and SD 002, the MPCA acknowledged this challenge and revised the compliance limits to be based on the achievable LOQs rather than WQBELs (MPCA 2024). However, this does not alleviate the fact that the WQBELs are below what has been demonstrated to be achievable by this or any other available technology. The same challenge applies to the intervention limits at WS 001 through WS 004, which are also lower than the LOQs for PFOA, PFHxS, and PFOS and therefore unmeasurable. Further, the Treatability Report does not support that the AWWTS can meet the compliance or intervention limits. The results in the Treatability Report do, however, indicate that these combined treatment technologies are effective at removing a variety of PFAS including short-chain compounds. It is inconclusive whether the proposed AWWTS will meet the Draft Permit WQBELs or intervention limits for PFHxS, PFOS, and PFOA due to limitations in analytical capabilities (i.e., the LOQs in almost all of the laboratory results were higher than the compliance and intervention limits specified in the Draft Permit).

The intervention limits, particularly with respect to WS 001 and WS 002 (IX resin effluent), present additional significant operational challenges due to the proposed regeneration schedule of the IX resin. Per the Design Basis Report (ECT2 and TKDA 2023), an estimated 18.2 discrete vessel regenerations will occur, on average, each week (12.6 for System A and 5.6 for System B). Once a vessel has been regenerated, it is placed back into service for normal water treatment operations. Because of this non-static operational philosophy, responding to intervention limit exceedances, even if the analytical methodologies were capable of reporting to these concentrations, would likely be infeasible due to the 3- to 4-week laboratory processing and reporting time required for PFAS samples. For instance, roughly 37.8 to 50.4 discrete vessel regenerations would occur on System A over a 3- to 4-week period. If there were to be an exceedance of the intervention limit, evaluating the root causes of the exceedance would be nearly impossible due to the turn-over in vessel orientation and duty. This regeneration schedule has been deliberately constructed to regenerate the IX resin once concentrations of short-chain PFAS are likely to be detected in the IX resin effluent. This has significant consequences for the treatment of PFOA, PFOS, and PFHxS, as these compounds did not break through the IX resin, except for one sample, during the treatability study. Thus, if the IX resin is regenerated at the onset of breakthrough of shortchain PFAS compounds, their longer-chain counterparts (i.e., PFOA, PFOS, and PFHxS) can be expected to be treated to levels below the LOQ.

6 Summary

The AWWTS represents approximately four years of testing, engineering, and construction at an approximate cost of \$275,000,000, and was designed to provide reliable, sustainable, and maximum extent practicable levels of treatment of the Cottage Grove Facility water. The AWWTS, a state-of-the-art and industry-exceeding PFAS treatment system, incorporates three field-implemented PFAS removal technologies that will treat, on average, approximately 11 MGD. At the time of this evaluation, there are no other known water treatment systems of this complexity operating at this scale outside of 3M. While the AWWTS exceeds the industry standard for PFAS treatment, the proposed WQBELs and intervention limits in the Draft Permit have not been demonstrated to be achievable for the Cottage Grove Facility water with the technologies included. The proposed WQBELs and intervention limits are lower than the LOQs for commercially available analytical techniques and thus are not measurable. The results in the Treatability Report do, however, indicate that these combined treatment technologies are effective at removing a variety of PFAS including short-chain compounds. Further, the innovative design incorporates IX resin and allows for on-site regeneration of the IX resin, which has been designed to be performed at the onset of short-chain PFAS breakthrough, thus ensuring the sustained treatment of PFOA, PFOS, and PFHxS to below current LOQs.

7 References

- Barr and Hazen & Sawyer. 2023. Evaluation of Current Alternatives and Estimated Cost Curves for PFAS Removal and Destruction from Municipal Wastewater, Biosolids, Landfill Leachate, and Compost Contact Water. Available at https://www.pca.state.mn.us/sites/default/files/c-pfc1-26.pdf.
- ECT2 and Barr. 2021. PFAS Treatability Study, 3M Cottage Grove, MN Facility.
- ECT2 and TKDA. 2023. Design Basis Report for Cottage Grove Water Stewardship Water Quality Project-Regeneration System.
- ITRC. 2023. Per- and Polyfluoroalkyl Substances (PFAS) Technical/Regulatory Guidance. September. Available at https://pfas-1.itrcweb.org/wp-content/uploads/2023/12/Full-PFAS-Guidance-12.11.2023.pdf.
- MPCA. 2024. Draft National Pollutant Discharge Elimination System and State Disposal System Permit MN0001449. State of Minnesota. Available at https://scs-public.s3-us-gov-west-1.amazonaws.com/env_production/oid333/did200071/pid_209149/project-documents/Draft%20Permit%20-%20Public%20 Notice%20-%20MN0001449%20-%20202.pdf.
- Wastewater Digest. 2020. RAAF Base Tindal, NT Australia. December 1. Available at https://www.wwdmag.com/water/article/10939321/ect2-raaf-base-tindal-nt-australia.
- Wastewater Digest. 2021. Pease Site 8: Regenerable Resin System for Groundwater Remediation. May 6, 2021. Wastewater Digest. Available at https://www.wwdmag.com/wastewater-treatment/article/10939725/ect2-pease-site-8-regenerable-resin-system-for-groundwater-remediation.

Appendix A

Arcadis Resumes

Copies of the resumes of the authors of the Arcadis Expert Report were provided as part of Exhibit D to 3M's August 2024 Comments and are incorporated herein by reference.

EXHIBIT 3

Parent Declaration

Declaration of Michael J. Parent, Ph.D.

3M Chemical Operations LLC's Comments to December 18, 2024 Draft NPDES/SDS Permit No. MN0001449 for 3M Cottage Grove Facility Cottage Grove, Washington County, Minnesota February 3, 2025

I, Michael J. Parent, declare and state as follows:

- 1. I am the Vice President of the Chemical Program at 3M Company. I have a Ph.D. in chemical engineering, which I received in 1999. I have worked full-time at 3M since 1999. As part of my role at 3M, I have studied 3M's design of its new advanced wastewater treatment systems being installed at 3M's Cordova, II, Cottage Grove, MN, and Decatur, AL facilities. The design considers, among other factors, the sources and amount of water to be treated, as well as the known or expected concentrations of PFAS expected to be present.
- 2. For the Cottage Grove, MN site, all of the water used or treated by the site is pumped from a series of wells located on the 3M Cottage Grove site itself as well as four remediation wells located about 5 miles from the plant site in Woodbury, MN, commonly referred to as the Woodbury Disposal Site wells¹. **Table 1** shows separately the relative amounts of water pumped from the 3M Cottage Grove site wells and the Woodbury remediations wells.

Table 1 Total Volume Pumped, thousand gallons

Table I	rotar volume i umpeu, mousanu ganons						
	2021	2022	2023				
Woodbury remediation wells. MPARS permit 1967-0674	1,110,261	1,078,208	1,111,524				
3M Cottage Grove Site Wells. MPARS permit 1960-0687	1,207,339	1,097,243	1,141,179				
Total	2,317,599	2,175,450	2,252,704				
Woodbury Wells, as % of total water pumped	47.9%	49.6%	49.3%				

¹ Settlement Agreement and Consent Order, *In the matter of Releases and Discharges of Perfluorochemicals at and From Sites in Washington County, Minnesota, and Certain Related Matters* (May 22, 2007) (SACO). The SACO was attached as Exhibit L to 3M's August 2024 Comments and is incorporated herein.

3. As part of the design process for the new the advanced wastewater treatment system, a review was completed in May 2021 that assembled the recent water pumped from each of the wells. **Table 2** shows this data for the four Woodbury wells. The design flows are the flow rates that were used as requirements for treatment for the advanced wastewater treatment system being installed in 3M's Cottage Grove site.

Table 2

Woodbury	2020 Average Operating Flow (gpm)	Design Flow Rate (gpm)		
B1	201	700		
B2	137	150		
B3	663	800		
B4	1313	1500		

4. Also, as part of the design process for the new the advanced wastewater treatment system, a review was completed in May 2021 that assembled the average and maximum concentrations of several PFAS analytes measured in the water produced by each well. **Table 3** shows this data for the four Woodbury wells.

Table 3

Woodbury Remediaton Wells

	Aver	age Conce	ntration, r	ng/mL	Maxin	num Conc	entration,	ng/mL
Analyte	B1	B2	В3	B4	B1	B2	В3	B4
Perfluorobutanesulfonic Acid (PFBS)	0.678	0.000	0.223	2.593	3.470	0.006	0.861	13.500
Perfluorobutyric Acid (PFBA)	0.913	0.496	0.613	1.307	1.790	0.632	0.980	4.550
Perfluorodecanoic Acid (PFDA)	ND	ND	ND	ND	ND	ND	ND	ND
Perfluorododecanoic Acid (PFDoA)	ND	ND	ND	ND	ND	ND	ND	ND
Perfluoroheptanoic Acid (PFHpA)	0.079	ND	0.017	0.407	0.152	ND	0.104	2.285
Perfluorohexanesulfonic Acid (PFHxS)	0.719	0.000	0.856	11.521	2.610	0.007	3.960	62.900
Perfluorohexanoic Acid (PFHxA)	0.479	0.000	0.067	0.722	0.974	0.007	0.191	3.960
Perfluorononanoic Acid (PFNA)	ND	ND	ND	ND	ND	ND	ND	ND
Perfluorooctanesulfonamide (PFOSA)	ND	ND	ND	ND	ND	ND	ND	ND
Perfluorooctanesulfonic Acid (PFOS)	0.044	ND	0.149	2.593	0.180	ND	0.735	32.200
Perfluorooctanoic Acid (PFOA)	0.836	0.000	0.173	2.287	2.330	0.005	0.664	17.000
Perfluoropentanoic Acid (PFPeA)	0.290	ND	0.042	0.343	0.532	ND	0.095	1.610
Perfluoroundecanoic Acid (PFUnA)	ND	ND	ND	ND	ND	ND	ND	ND

5. Using the data in **Table 2** and **Table 3** above, the mass loading that would correspond with the 2020 average operating flows and the design flow rate along with the average and maximum PFAS concentrations for PFOA, PFOS, and PFHxS can be calculated using the following equation:

$$\begin{aligned} \textit{Mass Loading Value} \; \left(\frac{g}{day} \right) \\ &= \textit{Sample Conc} \; \left(\frac{ng}{mL} \right) * \frac{3785 \; mL}{1 \; gal} * \frac{1 \; g}{10^9 \; ng} * \textit{Flow rate} \left(\frac{gal}{min} \right) * \frac{1440 \; min}{day} \end{aligned}$$

The data for each well in both flow scenarios is shown in **Table 4** and **Table 5**:

Table 4 Based on 2020 Ave Operating flows Average Concentration, g/day Maximum Concentration, g/day В4 В4 Analyte В1 B2 **B**3 **B1 B2 B**3 Perfluorohexanesulfonic Acid (PFHxS) 0.7880.000 3.093 82.446 2.859 0.005 14.310 450.136 Perfluorooctanesulfonic Acid (PFOS) 0.048 ND 0.539 18.559 0.197 ND 2.656 230.435 Perfluorooctanoic Acid (PFOA) 0.916 0.000 0.624 16.364 2.553 0.004 2.399 121.658

Table 5 Based on Design Flow Rates Maximum Concentration, g/day Average Concentration, g/day Analyte **B1 B2 B3 B4** B1 B₂ **B3 B4** Perfluorohexanesulfonic Acid (PFHxS) 514.245 2.744 0.000 3.732 94.188 9.958 0.006 17.267 Perfluorooctanesulfonic Acid (PFOS) ND 0.650 21.202 0.687 ND 3.205 263.254 0.168Perfluorooctanoic Acid (PFOA) 3.190 0.000 0.753 18.694 8.890 0.004 2.895 138.985

6. Comparing the calculated mass for each analyte shown in the tables above to the mass limits for PFOA, PFOS, and PFHxS listed in the Revised Draft Permit for 3M's Cottage Grove facility, the degree of treatment can be calculated, expressed in percent (%) removal, that would be needed in order to treat the water from the Woodbury wells and comply with the mass limits in the draft NPDES permit for the 3M Cottage Grove site. **Table 6** shows the low and high estimates for these degrees of treatment needed.

Table 6

	PFHxS		PF	os	PFOA		
Outfall	Low	High	Low	High	Low	High	
SD 001	99.9997%	99.99994%	99.995%	99.9997%	99.994%	99.9993%	
SD 002	99.9995%	99.99993%	99.994%	99.9996%	99.992%	99.9990%	

In summary, for PFHxS for both SD 001 and SD 002, a degree of treatment of at least >99.999% would be needed and a degree of treatment of >99.9999% may be needed. Similarly, for both PFOS and PFOA for both SD 001 and SD 002, a degree of treatment of at least >99.99% would be needed and a degree of treatment of >99.999% may be needed.

7. This analysis ignores any other source of PFAS other than the Woodbury wells.

8. Referring again to the following equation,

Mass Loading Value
$$\left(\frac{g}{day}\right)$$
 = Sample Conc * Flow rate

and substituting the Design Flow for SD 001 of 6.5 MGD and the mass limit for PFOA at SD 001 of 0.0011 g/day the necessary concentration to comply with the mass limit can be calculated, as

Sample Conc
$$\left(\frac{ng}{mL}\right) = \frac{Mass\ Loading\ Value\ \left(\frac{g}{day}\right)}{Flow\ rate(MGD)} * \frac{1\ MG}{10^6\ gal} * \frac{1\ gal}{3.785\ L} * \frac{10^9\ ng}{g}$$

Sample Conc
$$\left(\frac{ng}{mL}\right) = \frac{0.0011 \left(\frac{g}{day}\right)}{6.5 \, MGD} * \frac{1 \, MG}{10^6 gal} * \frac{1 \, gal}{3.785 \, L} * \frac{10^9 \, ng}{g} = 0.045 \, g/day$$

the calculated mass loading value is essentially equal to the WQBEL for PFOA at SD 001 of 0.045 g/day. **Table 7** contains the same calculation for PFOA, PFOS, and PFHxS at both SD 001 and SD 002. In all cases, the calculated maximum concentration that would comply with the mass limit at the design flow is essentially the same as the WQBEL for that parameter and quite a bit lower than the concentration Compliance Limit for each parameter.

Table 7

Location	Analyte	Revised Draft Permit Mass Limit, g/day	Design Flow Rate, MGD	Calculated Max. Conc. to comply w/ Mass Limit at Design Flow, ng/L	WQBEL, ng/L	Concentration Compliance Limit, ng/L
	PFHxS	0.0003	6.5	0.012	0.012	2.1
SD 001	PFOS	0.00093	6.5	0.038	0.038	2.2
	PFOA	0.0011	6.5	0.045	0.046	2.1
	PFHxS	0.0004	8.7	0.012	0.012	2.1
SD 002	PFOS	0.0012	8.7	0.036	0.038	2.2
	PFOA	0.0015	8.7	0.046	0.046	2.1

9. Another way to consider this is to determine what flow reduction would be necessary to ensure compliance with the mass limits. As an example, assuming a month with four sampling events at SD 001 (as required by the Revised Draft Permit) in which all of the PFOA values were reported as ≤2.1 ng/L. Each of these samples would be in compliance with the daily maximum and monthly average concentration Compliance Limit for PFOA of 2.1 ng/L. However, considering the permitted flow rate of 6.5 MGD for SD 001, the monthly average mass discharge (calculated to be 0.0517 g/day) would significantly exceed the mass-based limit for SD 001 (0.0011 g/day). Please note that this calculation assumes that averaging for mass-based values is similar to averaging for concentration-based values as described in the 5.70.83 of the Revised Draft Permit. **Table 8** shows this scenario for the permitted flow rate of 6.5 MGD and several reduced flows. To reach a point of compliance, the flow rate would need to be reduced to 0.14 MGD or less, which is only 2.2 % of the design flow rate for SD 001 and 3.2 % of the design flow rate for the Woodbury wells.

Table 8

Samples		6.5	MGD		3.25 MGD			
Samples	Concentration (ng/L)		Mass (g/day)		Concentration (ng/L)		Mass (g/day)	
1	<	2.1	<	0.0517	<	2.1	<	0.0258
2	<	2.1	<	0.0517	<	2.1	<	0.0258
3	<	2.1	<	0.0517	<	2.1	<	0.0258
4	<	2.1	<	0.0517	<	2.1	<	-
Monthly Average:	<	2.1	<	-	<	2.1	<	-
In Compliance?		Yes		No		Yes		No

Samples		1.6	MGD		0.14 MGD			
Samples	Concentration (ng/L)		Mass (g/day)		Concentration (ng/L)		Mass (g/day)	
1	<	2.1	<	0.0127	<	2.1	<	-
2	<	2.1	<	0.0127	<	2.1	<	-
3	<	2.1	<	0.0127	<	2.1	<	-
4	<	2.1	<	-	<	2.1	<	-
Monthly Average:	<	2.1	<	-	<	2.1	<	0.0011
In Compliance?		Yes		No		Yes		Yes

10. As another example, assume there is a month with four sampling events at SD 001 (as required by the Revised Draft Permit). Out of those four samples, three PFOA values were reported as ≤2.1 ng/L and one sample with a value reported as 2 ng/L. Each of these samples would be in compliance with the daily maximum and monthly average concentration Compliance Limit for PFOA of 2.1 ng/L. However, assuming the design flow rate for SD 001 (6.5 MGD) the calculated monthly average mass discharge (0.0123 g/day) would significantly exceed the mass-based limit for SD 001 (0.0011 g/day). **Table 9** shows this scenario for the permitted flow rate of 6.5 MGD and several reduced flows. To reach a point of compliance, the flow rate would need to be reduced to 0.6 MGD or less, which is only 9.2 % of the design flow rate for SD 001 and 13.2 % of the design flow rate for the Woodbury wells.

Table 9

Samples		6.5	MGD		3.25 MGD			
Samples	Concentration (ng/L)		Mass (g/day)		Concentration (ng/L)		Mass (g/day)	
1	<	2.1	'	0.0517	<	2.1	٧	0.0258
2	<	2.1	v	0.0517	<	2.1	'	0.0258
3	<	2.1	٧	0.0517	<	2.1	v	0.0258
4		2		0.0492		2		0.0246
Monthly Average:		0.5		0.0123		0.5		0.0062
In Compliance?		Yes		No		Yes		No

Samples		1.6	MGD		0.6 MGD			
Samples	Concentration (ng/L)		Mass (g/day)		Concentration (ng/L)		Mass (g/day)	
1	<	2.1	'	0.0127	<	2.1	'	0.0048
2	<	2.1	<	0.0127	<	2.1	<	0.0048
3	<	2.1	'	0.0127	<	2.1	'	0.0048
4		2		0.0121		2		0.0045
Monthly Average:		0.5		0.0030		0.5		0.0011
In Compliance?		Yes		No		Yes		Yes

11. In sections 5.70.113 and 6.63.35, the Revised Draft Permit details a requirement for an annual O&M Deviation & WWTP Optimization Report which requires an "evaluation of the WS 001 - WS 002 PFAS treatment performance relative to various concentration thresholds for a series of the PFAS parameters. **Table 10** contains the specified threshold concentrations. ².

Table 10

Parameter	Threshold Conc., ng/L
PFHpS	10
PFHxA	10
PFPeS	9.4
PFPeA	10
PFPrA	370
2233-TFPA	500

Parameter	Threshold Conc., ng/L
TFA	10700
TFMS	25
PFBS	71241
PFBA	686477
PFHxS	0.112
PFOS	0.35
PFOA	0.426

The fact sheet describes that these threshold concentrations were calculated for PFBA, PFBS, PFOS, PFOA, and PFHxA "using a dilution ratio" but it does not describe how the other threshold values were obtained. **Table 11** shows the threshold concentrations for the remaining PFAS parameters and includes values for estimated treated effluent water quality from the Pilot Study.

TABLE 11	Revised Draft Permit	Est. treated effluent water quality based on Pilot Study NCCW/SW - CalRes			
Location	WS 001 WS 002	SD 002	Equiv. conc. at WS 002		
Portion of total SD 001 Flow	18.76%	100%	18.76%		
Parameter	Conc, ng/L	Conc, ng/L	Conc, ng/L		
PFHpS	10	10	53		
PFHxA	10	10	53		
PFPeS	9.4	9	48		
PFPeA	10	10	53		
PFPrA	370	50	266		
2233-TFPA	500	500	2665		
TFA	10700	3140	16736		
TFMS	25	498	2654		

² Barr Engineering, *PFAS Treatability Study* (Dec. 22, 2021) (hereinafter the "Pilot Study") was attached to 3M's August 2024 Comments as Exhibit B and incorporated herein.

The threshold values at WS 001 and WS 002 are at a point in the process flow that has about 18.76% of the flow for the corresponding outfall. The values reported in the Pilot Study (Table 3.16) for the "estimated treated effluent water quality based on the treatability study" is reporting the estimated effluent concentration at the outfall (e.g., SD 002) using the weighted average of the RO effluent (81.24% of total flow at SD 002) and the ion exchange effluent (e.g., WS 002 which is 18.76% of total flow at SD 002). For all of the PFAS parameters except for PFPrA, the threshold value in the Revised Draft Permit is less than the flow adjusted values from the treatability study expressed in the table as "equivalent concentration at WS 002".

Additionally, there are statements in the Pilot Study (pp. 25-26) regarding the observed phenomena of breakthrough from the lag ion exchange vessel in less bed volumes (BVs) than breakthrough of the lead vessel. This indicates poor adsorption of the analytes mentioned (2,3,3,3-TFPA, PFPrA, and PFBA) which indicates potential difficulty removing these PFAS species.

On this 3rd day of February, I declare under penalty of perjury that the foregoing is true and correct to the best of my knowledge and belief.

Michael J. Parent, Ph.D.

EXHIBIT 4

Henningsgaard Notes

3M - MN0001449 - 1163 Low Flow Determination Notes Bruce Henningsgaard - 7-17-2023

When we determine low flows for a receiving water, we usually use a series of USGS gauges and convert the results at the gauge(s) to the discharge point based on drainage area. This was easily accomplished for the Mississippi River where the unnamed creek enters. However, we have decided to apply effluent limits based on discharging to the unnamed creek. There are no gauges on the unnamed creek so the usual method of determining low flows was not an option.

Another method we sometimes use, when gauges are not available, is USGS's StreamStats program. This web-based system allows us to zoom in on a location and get a number of watershed-based bits of information including low flows and drainage area. I used StreamStats to determine the drainage area at 3M's discharge point and at the flow monitoring sites on Battle Creek and Fish Creek (more on those creeks later). I also used StreamStats to generate low flows for the unnamed creek. The results were much higher than expected, especially when compared to the three flow data points we have from 2015. This difference prompted me to talk with Carol Sinden who reminded me of the Equal Yield process where we get low flows from a neighboring watershed and use the Equal Yield spreadsheet to transfer the results to the desired watershed. This is the process that was used to determine the low flows at 3M's discharge point.

There are two neighboring watersheds near the unnamed creek watershed: Battle Creek and Fish Creek. MCES has continuously monitored the flow in Battle Creek since January 1, 1996, and in Fish Creek since January 1, 1995, through 2022. Emily Brault used the flow data to determine the low flows for each creek. These low flows were then used in the Equal Yield process, along with the corresponding drainage areas, to determine the low flows in the unnamed creek.

The low flows in Battle Creek and Fish Creek were very similar to each other. I ran the Equal Yield spreadsheet for both Battle Creek and Fish Creek and the results for the unnamed stream were basically the same, or within 0.01 cfs. The unnamed stream 30Q10 low flows were used to determine ammonia effluent limits. Since the low flows were essentially equal, it did not matter which set of low flows were used to determine the ammonia limits. The ammonia limits were the same either way.

The low flows for the unnamed creek are lower than for Battle Creek and Fish Creek, even though unnamed creek has a larger drainage area. This makes sense when looking at two issues.

- 1. Approximately 1.5 to 2 miles upstream of the discharge points there is an unnamed lake (Ravine Park Lake) (82-0087-00). Flow out of this lake during dry periods could have an impact on the downstream flows.
- 2. The Washington Conservation District did some limited flow monitoring on the unnamed creek back in 2015. The flows on those days are lower when compared to the flows on the same days in Battle Creek and Fish Creek.

These two items indicate that our low flow results are reasonable.

EXHIBIT 5

StreamStats Report

StreamStats Report - Unnamed Creek

Region ID: MN

Workspace ID: MN20240313180328986000

Clicked Point (Latitude, Longitude): 44.79030, -92.90530

Time: 2024-03-13 13:03:49 -0500



Unnamed creek at the confluence with the railroad track running along the western/northwestern boundary of the facility.

Collapse All

Parameter Code	Parameter Description	Value	Unit
DRNAREA	Area that drains to a point on a stream	18.86	square miles
LAKEAREA	Percentage of Lakes and Ponds	0	percent
LONG_OUT	Longitude of Basin Outlet	-92.905312	degrees
SOILA	Percentage of area of Hydrologic Soil Type A	13.1	percent
SSURGOC	Percentage of area of Hydrologic Soil Type C from SSURGO	11.2	percent
STORNWI	Percentage of strorage (combined water bodies and wetlands) from the Nationa Wetlands Inventory	3.52	percent

➤ Low-Flow Statistics

Low-Flow Statistics Parameters [Low flow Region BC 2015 5170]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	2.98	2960
STORNWI	Percentage of Storage from NWI	3.52	percent	2.44	48
SSURGOC	SSURGO Percent Hydrologic Soil Type C	11.2	percent	2.61	100

Low-Flow Statistics Flow Report [Low flow Region BC 2015 5170]

Statistic	Value	Unit
7 Day 10 Year Low Flow	7.22	ft^3/s

Low-Flow Statistics Citations

Ziegeweid, J.R., Lorenz, D.L., Sanocki, C.A., and Czuba, C.R.,2015, Methods for estimating flow-duration curve and low-flow frequency statistics for ungaged locations on small streams in Minnesota: U.S. Geological Survey Scientific Investigations Report 2015–5170, 23 p. (http://dx.doi.org/10.3133/sir20155170)

> Peak-Flow Statistics

Peak-Flow Statistics Parameters [Minnesota Peakflow B 2023 5079]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	0.23087298	1393.716374
LAKEAREA	Percent Lakes and Ponds	0	percent	0	18.18143582
SOILA	Percent Hydrologic Soil Type A	13.1	percent	0	54.49756
LONG_OUT	Longitude of Basin Outlet	-92.905312	decimal degrees	-96.1197813	-91.6585024

Peak-Flow Statistics Flow Report [Minnesota Peakflow B 2023 5079]

PIL: Lower 90% Prediction Interval, PIU: Upper 90% Prediction Interval, ASEp: Average Standard Error of Prediction, SE: Standard Error (other -- see report)

Statistic	Value	Unit	PIL	PIU	SE	ASEp
66.7-percent AEP flood	175	ft^3/s	99.2	309	33.6	35.6
50-percent AEP flood	235	ft^3/s	133	414	33.6	35.6
20-percent AEP flood	416	ft^3/s	229	754	35.5	37.6
10-percent AEP flood	553	ft^3/s	297	1030	37.1	39.4
4-percent AEP flood	746	ft^3/s	385	1450	39.3	41.8
2-percent AEP flood	902	ft^3/s	454	1790	41	43.7
1-percent AEP flood	1060	ft^3/s	521	2160	42.6	45.5
0.2-percent AEP flood	1490	ft^3/s	692	3210	45.8	49.2

Peak-Flow Statistics Citations

Christopher A. Sanocki and Sara B. Levin2023, Techniques for Estimating the Magnitude and Frequency of Peak Flows on Small Streams in Minnesota, excluding the Rainy River Basin, Based on Data through Water Year 2019, U.S. Geological Survey Scientific Investigations Report 2023-5079, 15 p. (https://doi.org/10.3133/sir20235079)

> Flow-Duration Statistics

Flow-Duration Statistics Parameters [Flow duration Region BC 2015 5170]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	2.98	2960
STORNWI	Percentage of Storage from NWI	3.52	percent	2.44	48
SSURGOC	SSURGO Percent Hydrologic Soil Type C	11.2	percent	2.61	100

Flow-Duration Statistics Flow Report [Flow duration Region BC 2015 5170]

Statistic	Value	Unit
0.01 Percent Duration	493	ft^3/s
0.1 Percent Duration	265	ft^3/s
2 Percent Duration	81.9	ft^3/s
5 Percent Duration	49.4	ft^3/s
10 Percent Duration	26.8	ft^3/s
25 Percent Duration	20.7	ft^3/s
50 Percent Duration	15.6	ft^3/s
75 Percent Duration	13.8	ft^3/s
90 Percent Duration	11.1	ft^3/s
95 Percent Duration	9.27	ft^3/s
99 Percent Duration	7.43	ft^3/s
99.9 Percent Duration	4.62	ft^3/s
99.99 Percent Duration	3.88	ft^3/s

Flow-Duration Statistics Citations

Ziegeweid, J.R., Lorenz, D.L., Sanocki, C.A., and Czuba, C.R.,2015, Methods for estimating flow-duration curve and low-flow frequency statistics for ungaged locations on small streams in Minnesota: U.S. Geological Survey Scientific Investigations Report 2015–5170, 23 p. (http://dx.doi.org/10.3133/sir20155170)

> Seasonal Flow Statistics

Seasonal Flow Statistics Parameters [Low flow Region BC 2015 5170]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	2.98	2960
STORNWI	Percentage of Storage from NWI	3.52	percent	2.44	48
SSURGOC	SSURGO Percent Hydrologic Soil Type C	11.2	percent	2.61	100

Seasonal Flow Statistics Flow Report [Low flow Region BC 2015 5170]

Statistic	Value	Unit
Oct to Nov 7 Day 10 Year Low Flow	8.98	ft^3/s
30_Day_10_Year_Low_Flow_Oct_to_Nov	11	ft^3/s
7_Day_10_Year_lowflow_Dec_to_Mar	8.24	ft^3/s
30_Day_10_Year_Low_Flow_Dec_to_Mar	9.59	ft^3/s
Apr to May 7 Day 10 Year Low Flow	12.5	ft^3/s
Apr to May 30 Day 10 Year Low Flow	14.2	ft^3/s
7_Day_10_Year_lowflow_Jun_to_Sep	7.36	ft^3/s
30_Day_10_Year_Low_Flow_Jun_to_Sep	8.58	ft^3/s
122_Day_10_Year_Low_Flow_Jun_to_Sep	14.5	ft^3/s

Seasonal Flow Statistics Citations

Ziegeweid, J.R., Lorenz, D.L., Sanocki, C.A., and Czuba, C.R.,2015, Methods for estimating flow-duration curve and low-flow frequency statistics for ungaged locations on small streams in Minnesota: U.S. Geological Survey Scientific Investigations Report 2015–5170, 23 p. (http://dx.doi.org/10.3133/sir20155170)

> Bankfull Statistics

Bankfull Statistics Parameters [Interior Plains D Bieger 2015]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	0.19305	59927.7393

Bankfull Statistics Parameters [Central Lowland P Bieger 2015]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	0.200772	59927.66594

Bankfull Statistics Parameters [USA Bieger 2015]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	0.07722	59927.7393

Bankfull Statistics Flow Report [Interior Plains D Bieger 2015]

Statistic	Value	Unit
Bieger_D_channel_width	32.9	ft
Bieger_D_channel_depth	2.62	ft
Bieger D channel cross sectional area	86.3	ft^2

Bankfull Statistics Flow Report [Central Lowland P Bieger 2015]

Statistic	Value	Unit
Bieger_P_channel_width	36.7	ft

Statistic	Value	Unit
Bieger_P_channel_depth	3.05	ft
Bieger_P_channel_cross_sectional_area	80.5	ft^2

Bankfull Statistics Flow Report [USA Bieger 2015]

Statistic	Value	Unit
Bieger_USA_channel_width	34.8	ft
Bieger_USA_channel_depth	2.25	ft
Bieger_USA_channel_cross_sectional_area	83.5	ft^2

Bankfull Statistics Flow Report [Area-Averaged]

Statistic	Value	Unit
Bieger_D_channel_width	32.9	ft
Bieger_D_channel_depth	2.62	ft
Bieger_D_channel_cross_sectional_area	86.3	ft^2
Bieger_P_channel_width	36.7	ft
Bieger_P_channel_depth	3.05	ft
Bieger_P_channel_cross_sectional_area	80.5	ft^2
Bieger_USA_channel_width	34.8	ft
Bieger_USA_channel_depth	2.25	ft
Bieger_USA_channel_cross_sectional_area	83.5	ft^2

Bankfull Statistics Citations

Bieger, Katrin; Rathjens, Hendrik; Allen, Peter M.; and Arnold, Jeffrey G.,2015, Development and Evaluation of Bankfull Hydraulic Geometry Relationships for the Physiographic Regions of the United States, Publications from USDA-ARS / UNL Faculty, 17p. (https://digitalcommons.unl.edu/usdaarsfacpub/1515? utm_source=digitalcommons.unl.edu%2Fusdaarsfacpub%2F1515&utm_medium=PDF&utm_campaign=PDFCoverPages)

> Maximum Probable Flood Statistics

Maximum Probable Flood Statistics Parameters [Crippen Bue Region 6]

Parameter Code	Parameter Name	Value	Units	Min Limit	Max Limit
DRNAREA	Drainage Area	18.86	square miles	0.1	10000

Maximum Probable Flood Statistics Flow Report [Crippen Bue Region 6]

Statistic	Value	Unit
Maximum Flood Crippen Bue Regional	60900	ft^3/s

Maximum Probable Flood Statistics Citations

Crippen, J.R. and Bue, Conrad D.1977, Maximum Floodflows in the Conterminous United States, Geological Survey Water-Supply Paper 1887, 52p. (https://pubs.usgs.gov/wsp/1887/report.pdf)

USGS Data Disclaimer: Unless otherwise stated, all data, metadata and related materials are considered to satisfy the quality standards relative to the purpose for which the data were collected. Although these data and associated metadata have been reviewed for accuracy and completeness and approved for release by the U.S. Geological Survey (USGS), no warranty expressed or implied is made regarding the display or utility of the data for other purposes, nor on all computer systems, nor shall the act of distribution constitute any such warranty.

USGS Software Disclaimer: This software has been approved for release by the U.S. Geological Survey (USGS). Although the software has been subjected to rigorous review, the USGS reserves the right to update the software as needed pursuant to further analysis and review. No warranty, expressed or implied, is made by the USGS or the U.S. Government as to the functionality of the software and related material nor shall the fact of release constitute any such warranty. Furthermore, the software is released on condition that neither the USGS nor the U.S. Government shall be held liable for any damages resulting from its authorized or unauthorized use.

USGS Product Names Disclaimer: Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

Application Version: 4.19.4

StreamStats Services Version: 1.2.22

NSS Services Version: 2.2.1