



Sampling and Analysis Blueprint

February 5, 2020

Prepared For:

Madison Metropolitan Sewerage District
1610 Moorland Road
Madison, Wisconsin 53713

Prepared By:

TRC
708 Heartland Trail, Suite 3000
Madison, Wisconsin 53717

A handwritten signature in blue ink, appearing to read "Jeff Ramey".

Prepared by:

Jeff Ramey
Senior Chemist

A handwritten signature in black ink, appearing to read "Elizabeth Denly".

Reviewed and Approved by:

Elizabeth Denly, ASQ CMQ/OE
Quality Assurance & Chemistry Director /
PFAS Group Program Director



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ACRONYM LIST

AFFF	Aqueous film forming foam
CSM	Conceptual site model
DHS	Department of Health Services
DoD	Department of Defense
EPA	Environmental Protection Agency
ES	Wisconsin Administrative Code NR 140 Enforcement Standard
HA	Health Advisory
HDPE	High-density polyethylene
LC-MS/MS	Liquid chromatography with tandem mass spectrometry
MCL	Maximum Contaminant Level
MDLs	Minimum detection limits
mg/kg	Milligrams per kilogram
MMSD	Madison Metropolitan Sewerage District
ng/L	Nanograms per liter
NRB	Natural Resources Board
ORD	Office of Research and Development
PAL	Wisconsin Administrative Code NR 140 Preventive Action Limit
PFAS	Per- and polyfluoroalkyl substances
PFBS	Perfluorobutane sulfonate
PFCs	Perfluorinated compounds
PFCA	Perfluorocarboxylic acids
PFHpA	Perfluoroheptanoic acid
PFHxS	Perfluorohexane sulfonate
PFNA	Perfluorononanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulfonate
PFSA	Perfluorosulfonic acids
PT	Performance testing
PTFE	Polytetrafluoroethylene
QSM	Quality Systems Manual
qTOF	Quadrupole time of flight
RR	Remediation and Redevelopment

RSL	Regional Screening Level
SAB	Sampling and Analysis Blueprint
SIC	Standard Industrial Classification
SOPs	Standard operating procedures
SPE	Solid phase extraction
TAG	Technical Advisory Group
TOP	Total oxidizable precursor
TSS	Total Suspended Solids
UCMR3	Third Unregulated Contaminant Monitoring Rule
µg/kg	Micrograms per kilogram
VPLE	Voluntary Party Liability Exemption
WI Criteria	Wisconsin PFAS Aqueous (Non-Potable Water) and Non-Aqueous Matrices Method Expectations
WDNR	Wisconsin Department of Natural Resources
WWTPs	Wastewater treatment plants

Executive Summary

Per- and polyfluoroalkyl substances (PFAS) are a large group of over 4,000 synthetic chemicals that have been manufactured since the 1940s and are used in a variety of industrial and commercial products and processes. Some PFAS are resistant to degradation, are known to persist and be mobile once introduced into the environment and have the potential to bioaccumulate. In addition, some PFAS have demonstrated toxicity at very low concentrations.

Because of their prevalence and persistence, PFAS are frequently detected in our waste streams, including influents to municipal wastewater treatment plants (WWTPs). The concentrations of PFAS in the influents to WWTPs are found to be higher in urbanized areas and greatest in situations where WWTPs service significant industrial source(s) of PFAS.

The Madison Metropolitan Sewerage District (MMSD or District) operates the Nine Springs WWTP, which provides wastewater treatment to over 380,000 people in a 184 square mile service area. PFAS are expected to be detectable in MMSD's influent for this urbanized area. However, the levels are not anticipated to be on the high end of WWTPs that have been studied because significant industrial sources of PFAS have not been identified in MMSD's service area.

Regulatory standards are being developed by the Wisconsin Department of Natural Resources (WDNR) that will require careful sampling and analysis of PFAS. The ubiquity of PFAS in the environment and prevalence of PFAS in consumer goods and sampling equipment requires a prescriptive sampling and analysis plan to avoid, or determine, when cross contamination occurs. This includes avoiding polytetrafluoroethylene (PTFE) materials and many personal care products during sampling.

As of the date of the publishing of this Sampling and Analysis Blueprint (SAB), the only promulgated analytical methods for analyzing PFAS are for drinking water. However, commercial, municipal, and academic laboratories produce reliable and usable data for PFAS in matrices such as wastewater and biosolids. This is typically done by modifying the established analytical methods. A modified method allows a laboratory to produce data that lowers the reporting limits, expands the PFAS compound list, and adds different quantitative techniques (e.g., isotope dilution).

The WDNR created the WI Criteria, which is method performance criteria (not an analytical method), that will allow for future certified laboratories to produce results for 36 PFAS compounds in solid and non-potable liquid matrices. Laboratories were surveyed to assess their PFAS experience, capacity, and intent to certify and multiple laboratories are expected to be able to provide usable and reliable data to the District based on this survey. Screening methodologies for PFAS are also available but most are only appropriate for highly concentrated sources of PFAS.

To investigate PFAS, a phased characterization is recommended as described in the sampling and analysis plan. The initial sampling would characterize PFAS at the Nine Springs WWTP in the influent, effluent, biosolids, struvite, and polymers. The optional additional sampling, if necessary, would further characterize the initial sample points, determine high concentration and transformation points within the plant's processes, and investigate potential industrial sources in the District's service area.

1.0 Purpose and Scope

To gain a better understanding of the impacts of PFAS, MMSD along with TRC, has conducted a study featuring a literature search, SAB, a fate and transport report, and a conceptual site model (CSM) for PFAS at the District's Nine Springs Wastewater Treatment facility. This SAB is designed to inform the District on the state-of-the-science concerning PFAS. The objective of the SAB is to help the District understand the developing landscape for PFAS sampling and analysis. The SAB will assist the District in selecting and conducting a comprehensive PFAS sampling plan that includes sampling of their influent and effluent, at locations within the plant, and biosolids. The SAB provides procedures to be used for the potential sampling of industrial discharge permit holders and addresses other media.

The SAB includes a review and comparison of the latest published, draft, and developing analytical methodologies and screening techniques for PFAS. Several laboratories were surveyed in order to identify those with the capability of providing usable PFAS data to the District for the analysis of influent, effluent, in-plant process samples, and biosolids. This survey evaluated WDNR (NR 149) PFAS method criteria certification status, experience, and capacity for PFAS testing at 11 laboratories. A list of potential laboratory options is provided in this SAB.

This SAB also contains a sampling and analysis plan that outlines the recommended sampling and analysis of each media.

2.0 Background

Some of the earliest mentions of PFAS by the WDNR began over a dozen years ago in 2006 with the addition of perfluorinated compounds (PFCs) to the annual fish bioaccumulation pollutant studies in the Great Lakes and major river systems. PFAS data (although referred to as PFC at the time) were collected again in 2010 as part of the United States Environmental Protection Agency's (EPA) National Coastal Condition Assessment/ Great Lakes Human Health Fish Tissue Study. In 2016, the WDNR published a study using Great Lakes Restoration Initiative funding including these data sets from the WDNR and EPA. Perfluorooctane sulfonate (PFOS) was present in the highest concentrations and in the highest number of samples in the studies with rivers, and specifically, the Mississippi River showed higher results than the Great Lakes.

In 2002, the WDNR issued Remediation and Redevelopment (RR) Program guidance document, RR-682, that provided direction on using the EPA's Soil Screening Level website for determining residual contaminant levels. This calculator was replaced with the EPA Regional Screening Level (RSL) website that includes updates to the web calculator every six months, starting in 2008, and an updated guidance document [PUB-RR-890](#) issued in 2014 succeeded the previous guidance. The [RSL calculator](#) provides the only promulgated limits for PFAS used by the state of the Wisconsin and provides industrial and residential direct contact screening levels for PFOS, PFOA, and PFBS in soil. The residential screening levels in soil are 1.26 milligrams per kilogram (mg/kg), 1.26 mg/kg, and 1,260 mg/kg for PFOS, perfluorooctanoic acid (PFOA), and perfluorobutane sulfonate (PFBS), respectively. The soil screening levels are simply soil criteria and are not protective of groundwater leaching from solid materials such as impacted soil or land applied biosolids.

In February of 2018, the WDNR RR program released an update stating that PFAS meet the Wisconsin definition of a hazardous substance under Wisconsin (Wis.) Statute (Stat.) 292.01(5). Therefore, discharges of PFAS to the environment are subject to regulation under Wis. Stat. 292 and require immediate notification, investigation, and remediation under Wisconsin Administrative Code chapters NR 700-754. In September of 2018, the WDNR addressed the historical usage of PFAS at open Voluntary Party Liability Exemption (VPLE) sites by requesting a survey to be completed; new VPLE sites must now address historical PFAS usage. Early in 2019, the Bureau for Remediation and Redevelopment Tracking System was updated to include PFAS as a substance and the WDNR launched a PFAS website that is now a continually updated resource. The RR Program has convened a PFAS Technical Advisory Group (TAG) that is open for public attendance and has been meeting quarterly since February 22, 2019. The PFAS TAG has four subgroups including History and Use of PFAS, Fate and Transport of PFAS, Water Quality and Wastewater, and Waste and Materials Management. PFAS, and emerging contaminants in general, are also being taken into consideration in the current drafting of rule updates in the NR 700-754 emergency and permanent rule change processes.

On June 21, 2019, the Wisconsin Department of Health Services (DHS) provided recommendations for groundwater standards (Wisconsin Administrative Code NR 140 Enforcement Standard [ES] and Preventive Action Level [PAL]), as requested by the WDNR, for 27 groundwater contaminants (referred to as Cycle 10). PFOS and PFOA were included in the list of 27 contaminants. This is the first time in the last 10 years that the WDNR has proposed new or revised groundwater standards. The WDNR rulemaking process for Cycle 10 commenced in March of 2018 with the WDNR's formal request of DHS to provide groundwater recommendations and it is estimated by the WDNR that NR 140 groundwater standards for PFOS and PFOA will be promulgated in the Fall of 2021. The WDNR stated in their press release after promulgating groundwater quality standards that: "These standards are used for regulating facilities, practices, and activities that can affect groundwater. They apply to bottled water, approved agricultural chemicals, contamination site cleanup, regulation of solid waste landfills, and more."

The DHS has recommended a groundwater ES of 20 nanograms per liter (ng/L) or parts per trillion (ppt) for PFOS and PFOA individually or combined and a groundwater PAL of 2 ng/L for PFOS and PFOA individually or combined. These levels are amongst the lowest groundwater standards for these compounds in combination in the nation. According to DHS, the groundwater standards are set at a level to protect people, including sensitive populations such as pregnant women and infants, from health effects associated with PFOA and PFOS exposures, based on the most recent scientific findings. At these low levels, it will be challenging for laboratories to achieve reporting limits at or below the recommended PAL for PFOS and PFOA of 2 ng/L using the current analytical methodologies, due to the presence of matrix interferences.

In the absence of a promulgated PFAS analytical method for matrices other than drinking water, the WDNR's NR 149 laboratory certification program drafted the "WI PFAS SOP" with input from 17 laboratories, the Department of Defense (DoD), and EPA, to create a uniform set of method and performance criteria to report 36 PFAS compounds. On September 16, 2019, the WDNR released the "Wisconsin PFAS aqueous (non-potable water) and non-aqueous matrices method criteria," which is shortened to the WI Criteria for the purpose of this report, for a three-week public comment period and began accepting applications to certify laboratories for the criteria on October 29, 2019. The WI Criteria was finalized and released on December 16, 2019 (Wisconsin PFAS Aqueous [Non-Potable Water] and Non-Aqueous Matrices Method Expectations).

Concurrently, the WDNR has formally issued a request to the DHS to provide groundwater recommendations for 40 additional contaminants (referred to as Cycle 11), including 34 additional PFAS that do not currently have recommendations for NR 140 groundwater standards. The rulemaking process and timeline for Cycle 11 has a proposed end point for a published and effective date of Fall 2023. This Cycle 11 rulemaking procedure, and the previously mentioned Cycle 10, will follow separate prescribed rulemaking processes for the promulgation of permanent rules that will include the opportunity for public input through public meetings and hearings, natural Resources Board (NRB) meetings, and stakeholder input into the economic impact of the proposed rules in the required preparation of an Economic Impact Analysis. In a Waste and Materials Management PFAS TAG subcommittee meeting on November 7, 2019, DHS toxicologist Dr. Sarah Yang stated that DHS is moving forward on developing groundwater recommendations for 20 of the requested 34 Cycle 11 PFAS compounds and that 14 PFAS compounds did not have enough study data available to provide recommendations.

On July 22, 2019, the WDNR sent letters to 125 WWTPs, including the District, requesting the voluntary monitoring of PFAS for facilities with industrial pretreatment programs. The WDNR requested voluntary testing of influent and effluent for the WWTPs and listed known PFAS sources. The request was to sample influent and effluent at the WWTP within 90 days of receipt of the letter and to analyze for 36 PFAS compounds (including PFOS and PFOA). If the combined (additive) concentration of PFOS and PFOA in the influent or effluent was at or above 20 ng/L, the WDNR recommended that the WWTP conduct a review of industrial users to identify facilities that may be potential sources of PFOA and PFOS. In the letter, the WDNR committed to working with the WWTP to establish a sampling protocol of the wastewater from the probable PFAS industrial sources. Following the identification of an industrial PFAS source, WDNR staff offered to collaboratively work with municipalities and the industrial sources to reduce and eliminate PFAS discharges. Suggested source reduction efforts included product substitution, operational controls, pretreatment, and clean-up of historical contamination.

It is unknown at this time if an industrial or commercial source of PFAS is currently discharging to the District and the scope of an investigation would be subject to the individual dischargers and potential sources within the particular branch of the collection system being investigated. The WDNR used Standard Industrial Classification (SIC) Codes to group sources into designated categories and the SIC Codes for the WDNR designated known sources are listed below.

- **3471, 3479** Platers/metal finishers
- **26** Paper and packaging manufacturers
- **31, 23, 2273** Tanneries and leather/fabric/carpet treaters
- **Many SIC** Manufacturers of parts with PTFE (polytetrafluoroethylene, Teflon type)
- **Many SIC** Facilities that manufacture or use coatings
- **4953** Centralized waste treaters
- **0241** Dairy processing facilities and cheesemakers, where milk supply is sourced from livestock grazing on fields that have received PFAS-contaminated biosolids

- **2899, 3999** Fire-fighting equipment manufacturers
- **9711** Military bases
- **4581** Airports
- **284** Household cleaning product manufacturers
- **4953** Landfills (*listed as a receiver of PFAS and not a source*)

At a minimum, the District should be aware of the industries of interest identified by the WDNR and evaluate their industrial users to identify any that fall into the SIC Codes above. Note, the lack of specificity within some designations such as the SIC major category 26 – Paper and Packaging Manufacturers can be an overly broad designation to group all manufacturers under this category as a known source. Known sources of PFAS exist with paper and packaging manufacturers but, for example, SIC Code 2674 – Uncoated Paper and Multiwall Bags would not be a known or categorical source of PFAS.

In addition to developing groundwater standards, the WDNR has also started the rulemaking process of establishing NR 809 drinking water standards, NR 105 surface water quality standards, NR 106 effluent limitations, fish consumption advisories, and studying the impact of PFAS in the land application of biosolids. Per the rulemaking process, three scope statements were released for the proposed changes and a public hearing was held on November 12, 2019. The open comment period ended on November 19, 2019 and the NRB approved all three scope statements on January 22, 2020, officially commencing the 30-month rule-making process

In comparison to other states, the proposed groundwater ES of 20 ng/L for the sum of PFOS and PFOA that is recommended in Wisconsin is amongst the most conservative. Vermont is similar to Wisconsin, in that the groundwater standard is 20 ng/L; however, Vermont sums five individual PFAS compounds (PFOS, PFOA, perfluorononanoic acid [PFNA], perfluorohexane sulfonate [PFHxS], and perfluoroheptanoic acid [PFHpA]). Connecticut sums the same five PFAS compounds as Vermont, but uses a groundwater standard of 70 ng/L, which is the same concentration the EPA updated in 2016 as the established lifetime Health Advisory (HA) for PFOS and PFOA. Note, the EPA HA is not an enforceable regulatory standard and no federal standard for PFAS exists in any media.

The WDNR has been in contact with regulators from the neighboring states of Michigan and Minnesota concerning regulating PFAS and the development of standards. Minnesota released revised health-based advisory levels for drinking water and groundwater of 15 ng/L PFOS and 47 ng/L PFHxS in April of 2019 and has additional criteria for other PFAS. Michigan has groundwater surface water interface criteria for waters used as a drinking water source of 11 ng/L PFOS and 420 ng/L PFOA. Additionally, Michigan is in a formal rulemaking process for the establishment of maximum contaminant levels (MCLs) for drinking water for seven PFAS compounds with some of the lowest recommended health-based values: 6 ng/L PFNA; 8 ng/L PFOA; and 16 ng/L PFOS. Michigan estimates that the promulgation of their state drinking water MCLs will be completed in Spring 2020.

3.0 Analytical Methodology for PFAS

3.1 Individual Compound Analysis

The EPA promulgated an analytical methodology for PFAS in 2009 for drinking water, in part, to standardize a test method for the Third Unregulated Contaminant Monitoring Rule (UCMR3) that included the analysis of six PFAS compounds in public water supplies nationwide. The EPA methods for drinking water, 537 rev.1.1 (November 2009), 537.1 (November 2018) and 533 (December 2019), remain the only promulgated EPA methodologies at the time of publishing this SAB. In the absence of a standard methodology for analyzing non-potable water and solid samples, method modifications, including different method and performance criteria, have been utilized by commercial, private, public, and academic laboratories to provide quantifiable data for individual PFAS compounds. A list of the most pertinent finalized and draft analytical methods or established method criteria are in **Table 1**.

PFAS are typically analyzed using liquid chromatography with tandem mass spectrometry (LC-MS/MS) for aqueous and non-aqueous samples. Solid phase extraction (SPE) is the most predominant extraction technique for aqueous samples, allowing for the selection of an appropriate SPE cartridge sorbent, depending on the required analyte list. Isotope dilution with recovery correction is an analytical technique that corrects results for matrix interferences; this is a common modification to EPA Method 537 that laboratories utilize to provide more accurate data. EPA Method 537 is the method that most commercial, public, private, and academic laboratories are modifying to include isotope dilution and expanded analyte lists in the absence of a promulgated methodology for non-potable aqueous samples and solid samples. Commercial laboratories prepare standard operating procedures (SOPs) for inclusion in their Quality Assurance Manual for each method performed within their laboratory (standard or modified methods). In the case of modified methods, the SOPs for PFAS are typically proprietary and outline the processes that deviate from the published method such as isotope dilution or the technique for biosolids homogenization.

3.2 PFAS Screening

The way in which PFAS compounds are manufactured and exist in products presents a challenge to quantify individual PFAS compounds. There is believed to be over 4,000 individual PFAS compounds that exist in the class. PFAS are synthesized by two primary processes: telomerization and electrochemical fluorination. Electrochemical fluorination produces products that can biotransform into branched and linear perfluorocarboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs), such as PFOA and PFOS; whereas, products produced using telomerization biotransform into mainly linear PFCAs. Many commercial and industrial products, and aqueous film forming foam (AFFF), were historically and are currently produced as technical mixtures with a wide range of physical and performance characteristics and without a quantification of individual PFAS compounds. Individual compound analysis by mass spectrometry (the prevalent individual compound analysis) requires using a standard to quantify each target analyte. At the point of writing this SAB, standards for only a few hundred individual PFAS compounds are commercially available. Standards are currently available for the 36 PFAS compounds included in the WI Criteria.

Screening methodologies and tools have been established and are being developed to analyze for PFAS in order to gain an understanding of the mass of total PFAS or total fluorine that may exist in a particular sample. **Table 2** describes the screening techniques and tools that were evaluated for potential use by the District.

The use of screening tools tends to be applicable in the presence of high concentrations of total or organic fluorine in soil or aqueous samples and not as an inexpensive substitute for individual PFAS compound analysis. This may be more applicable to determining hot spots associated with the recent usage of AFFF containing PFAS, for example, and less applicable to lower concentrations of PFAS present in WWTP influent, effluent, or biosolids. However, total organic fluorine by combustion ion chromatography is utilized in Australia to screen soils received at landfills for PFAS and could potentially be applied to evaluate biosolids in the United States given the ability to screen as low as 100 micrograms per kilogram ($\mu\text{g}/\text{kg}$).

The total oxidizable precursor (TOP) Assay is effective in determining the potential total mass of PFAS present in samples by forcing oxidization of polyfluoroalkyl compounds and subsequent transformation into perfluorinated compounds. TOP Assay has applications in source characterization and remediation to determine the potential total PFAS mass. For the District, TOP Assay analysis of influent wastewater may be able to identify the mass of PFAS not identifiable with the current extraction and analytical methods. TOP Assay could also be applied at points within the District's processes to gain a better understanding of total PFAS mass flux before and after processes such as aerobic digestion.

Quadrupole time of flight (qTOF) is being utilized by academia and in EPA's Office of Research and Development (ORD) laboratory and may be the future of high-resolution mass spectrometry commercial analysis of PFAS. qTOF can determine the standard PFAS analytes, including precursors and common degradation products, and can be utilized in forensic evaluations to help determine sources of PFAS. This technology may be commercialized in the future. This would allow the District to use the analysis in addition to or instead of TOP Assay to determine total PFAS mass or to fingerprint multiple sources of PFAS.

3.3 Wisconsin Criteria

On September 16, 2019, the WDNR released the WI Criteria for public comment. The summary of the WI Criteria stated that "providing this criterion to laboratories allows the department to accredit laboratories until such time the EPA publishes their 1600 series isotopic dilution method". The WI Criteria was developed using the DoD Quality Systems Manual (QSM) version 5.2/5.3 method criteria for analyzing PFAS and using specific knowledge of the method criteria in development by the EPA. The 1600/EPA method 8328 will replace the WI Criteria once promulgated. Seventeen commercial, public, and academic laboratories also participated in the development of the draft WI Criteria. A major difference between the WI Criteria and the EPA methods under development is the anticipated PFAS compound list. The EPA 1600/EPA method 8328 is anticipated to include 25 PFAS compounds, whereas the WI Criteria contains 36 PFAS compounds. **Table 3** lists the 36 compounds from the WI Criteria. The list of 36 PFAS compounds for analysis requested in the WI Criteria is the largest list of PFAS analytes in the nation for aqueous and solid matrices. For example, the state of MI requests a minimum of 28 PFAS compounds to be analyzed for investigating surface water and wastewater.

Although the most recent update from the EPA's ORD targeted a draft release of the EPA 1600/ EPA method 8328 for fall of 2019, recent comments from EPA staff suggest the development of the 1600 series method will be a multi-year process. The WDNR began accepting applications to certify laboratories for the WI Criteria on October 29, 2019. It is anticipated that for the foreseeable future, the WI Criteria and list of 36 PFAS compounds will be the accredited criteria for laboratories analyzing PFAS in Wisconsin.

On October 25, 2019, TRC sent surveys to 11 analytical laboratories to determine their intent to certify for the WI Criteria under NR 149 with the purpose of developing a list of potential laboratories for PFAS analysis. The laboratories selected for the survey were local and national commercial and public laboratories with known experience analyzing for PFAS. The surveyed laboratories were requested to submit their intent to seek WI Criteria certification, expected date of WDNR audit, matrices included in the certification request, PFAS compound list included in the certification request, number of PFAS instruments in the laboratory, average number of PFAS samples analyzed per year, lead analyst's years of PFAS experience, experience with PFAS in Wisconsin, year laboratory first analyzed for PFAS, and any other comments the laboratory wanted to provide. The results of the laboratory survey are provided in **Table 4**.

At the time of the laboratory survey request, the WI Criteria were in draft form. The WDNR evaluated public comments and released the [final criteria](#) on December 16, 2019. As such, most laboratories had not yet applied for certification upon submission of their survey response. On October 29, 2019, the WDNR launched a website for the "[Laboratory certification for PFAS](#)" and to request applications from interested laboratories. Given the timeline of this SAB, requesting and reviewing individual SOPs from each surveyed laboratory for the WI Criteria was premature. Therefore, the review of the SOPs is an existing data gap that will need to be addressed in the future prior to final recommendations of laboratories and any District sampling. The laboratory SOP review will include evaluating the minimum detection limits (MDLs) for the 36 PFAS compounds and ensuring the sum of the MDLs for PFOS and PFOA can meet the ES of 20 ng/L for influent and effluent wastewater samples. The WDNR is accepting applications to certify for the following scenarios: drinking water for PFOS and PFOA and/or 18 PFAS compounds by EPA method 537.1 and aqueous and solid matrices for PFOS and PFOA or the PFAS group of up to 36 compounds by the WI Criteria. The WDNR expects to update their website in or around April 2020 with the certification of multiple laboratories for PFAS testing in solid, non-potable water, and drinking water matrices and at that time, certified laboratories would have approved SOPs available to review.

From evaluating the survey responses, several laboratories are seeking or intend to seek certification for the group of 36 PFAS compounds included in the WI Criteria and are capable of producing results given their established industry experience analyzing for PFAS.

- Eurofins TestAmerica Sacramento and Vista Analytical Laboratory appear to be capable of conducting non-potable water PFAS analysis pending audits by the WDNR and certification of the entire 36 PFAS compound list. Originally, the Wisconsin State Laboratory of Hygiene had sought to add all 36 compounds to their target analyte list for solids, tissue, and non-potable water; however, the results of their performance testing (PT) study resulted in the passing of only 31 PFAS compounds for each matrix as listed in **Table 4**. In particular, the results for five precursors did not pass the PT study and these compounds are of potential interest to the District as they are potential transformational perfluorooctyl precursors that could add additional PFOS and PFOA

mass generated through WWTP processes. Both Eurofins TestAmerica Sacramento and Vista Analytical Laboratory have the capability of running a TOP Assay to determine additional PFAS mass in non-potable samples.

- The same concerns for selecting a laboratory capable of passing PT studies for non-potable water analysis for the full 36 compound list exist for the analysis of solids/ biosolids/ sludge samples. Eurofins TestAmerica Sacramento, Eurofins Lancaster, and Vista Analytical Laboratory appear to be capable of conducting solids/biosolids/sludge PFAS analysis pending audits by the WDNR and certification of the entire 36 PFAS compound list. In addition, Alpha Analytical has substantial experience with PFAS biosolids analysis and could be an option as a non-Wisconsin certified laboratory. TestAmerica Sacramento and Eurofins Lancaster have PFAS experience and SOPs for leaching procedures (EPA 1312 Synthetic Precipitation Leaching Procedure and EPA 1315 Leaching Environmental Assessment Framework) which could provide the opportunity to analyze soil samples for leaching at areas in which the District's biosolids have been land applied. All four of the aforementioned laboratories have the capability of running a TOP Assay.

4.0 Sampling and Analysis of PFAS

PFAS are ubiquitous in the environment and exist in many sampling materials and equipment, clothing, and personal care products. Procedures for PFAS sampling with restricted use materials should be strictly adhered to for compliance sampling. Blank samples in any sampling program should be included to determine potential cross-contamination from sampling equipment, sampling personnel, and sample transportation. Below is a general procedure for sampling of PFAS that should be followed for the sampling of any media. Sampling at the District should be performed by trained and experienced personnel and follow a health and safety plan and specific sampling and analysis plan.

4.1 Sampling and Analysis Plan – Influent and Effluent Characterization

The District may decide to sample for PFAS at various points to characterize the PFAS coming into the WWTP, transformations of PFAS by plant processes, and PFAS in the aqueous or solid effluents. A general procedure for sampling PFAS in various media is provided in **Appendix A** and the detailed sampling and analysis plan for characterization in the influent and effluents is summarized in **Table 5**.

This sampling and analysis plan is intended to help close the data gaps presented in the PFAS Fate and Transport report TRC has prepared for the District in parallel with this SAB. Potential sampling locations at the Nine Springs WWTP are depicted on **Figure 1** and the potential sampling locations in relation to the Process Flow Diagram for Nine Springs WWTP are depicted on **Figure 2**. The name, number, and location of buildings at the Nine Springs WWTP are depicted on **Figure 3**. A photographic log of the potential sampling locations is included in **Appendix B**.

4.1.1 Influent and Effluent Sampling

The input of waste to the Nine Springs WWTP consists of the influent from five pumping stations (99.77%) and trucked-in waste (0.23%), which is combined at the influent well at the headworks. The combined waste undergoes primary treatment, secondary biological treatment and clarification, and the treated liquid effluent is then discharged to Badfish Creek or Badger Mill Creek. The accompanying Fate and Transport Report provides further details on the treatment processes used at the Nine Springs WWTP.

The influent of each individual pumping station should be sampled for flow, total suspended solids (TSS) and PFAS and analyzed by a laboratory that is certified for the WI Criteria and capable of producing the 36-compound list in Wisconsin (or required list at the time of testing) as described in Section 3.3. The results of the five influent samples should be normalized using the flow data to calculate an estimated influent concentration of PFOS+PFOA using the calculation below:

C = Concentration of PFOS+PFOA (ng/L)

F = Flow (gallons/ minute)

$$C_{Inf} = \frac{[(C_{PS02} * F_{PS02}) + (C_{PS07} * F_{PS07}) + (C_{PS08} * F_{PS08}) + (C_{PS11} * F_{PS11}) + (C_{PS18} * F_{PS18})]}{(F_{PS02} + F_{PS07} + F_{PS08} + F_{PS11} + F_{PS18})}$$

If the calculated influent is above 20 ng/L for PFOS and PFOA, the District could further investigate the influent of the individual pumping stations that are contributing PFOS and PFOA mass to the WWTP. Prior to sampling the influent, the District should document the service areas contributing to each pump station and isolate, if practicable, the flow from the District's service areas to avoid interconnecting the mixing points (such as in pumping station 7 and 18). This will help to isolate potential individual sources of PFAS within the District's service area. The investigation of industrial sources should follow the procedure in Section 4.2 and as outlined in the Fate and Transport Report.

The effluent conveyance should be sampled for PFAS and analyzed by a laboratory that is certified for the WI Criteria (or EPA 1600/8328) and capable of producing the 36-compound list in Wisconsin (or required list at the time of testing) as described in Section 3.3. The combined MDLs for PFOS and PFOA for the selected laboratory must be below the ES of 20 ng/L for the analysis of District effluent samples. This should be confirmed with the laboratory before ordering sampling containers.

The District has fixed ISCO samplers inline at each of the five individual pump stations. Composite sampling using the fixed ISCO samplers should be performed for influent. A portable ISCO sampler should be used to sample effluent to account for diurnal variation.

If future grab sampling, or sampling directly into the sample container, is needed, the method is described here. If the sample point is inaccessible, an extension rod can be used to extend the sample container to the sampling point. If the sampling point is stagnant water or flowing water in which the depth of the sample point is deeper than the height of the sample container, then the sample container should be immersed to sample from the water column. PFAS have a tendency to accumulate at the air-water interface due to their surfactant properties; for stagnant water, sampling only at the surface could result in high biased or non-representative results for the entire

column of water. However, collection of both submerged and surface samples should be considered to determine the mass of PFAS flux at locations where there is not turbulent flow.

If a composite or grab sample needs to be subsampled from a sampling container, such as a high-density polyethylene (HDPE) five-gallon bucket, then the sample container should be submerged in the sampling container.

The initial characterization in **Table 5** illustrates a sampling scope for the initial sampling of the District's influent and effluent. Although temporal changes to PFAS mass can be difficult to determine, the influent and effluent samples in each sampling event should be taken at the same time or within one week each other.

4.1.2 Biosolids Sampling

Solids that are separated from the wastewater during primary and secondary treatment are thickened and digested to create biosolids: Class A Cake and Class B Liquid (Metrogro®). (Further information on the biosolids can be found in the Fate and Transport Report.) The biosolids should be sampled for PFAS and sent to a laboratory capable of producing results for the 36-compound list in Wisconsin as described in Section 3.3.

Biosolids with high solids content, such as the Class A Cake, should be collected as a grab sample by simply using an HDPE or stainless-steel trowel to sample from the pile and place the sample in the sample container. A representative sample should be collected at a point below the surface of the pile. Metrogro®, which is a Class B liquid slurry, should be sampled from the gravity belt thickener using the container provided from the laboratory to directly catch the biosolids from the belt.

The initial characterization in **Table 5** includes PFAS characterization of the District's two types of biosolids: Class A Cake and Class B Liquid (Metrogro®). The optional additional characterization (as necessary) includes further characterization of the biosolids using TOP Assay and a temporal analysis of Metrogro®. For this additional characterization, it is recommended that one Metrogro® sample be submitted for immediate analysis and a second Metrogro® split sample be held in temperature conditions consistent with the Metrogro® holding area by the District for 180 days and then submitted for analysis. The scope of potential biosolids sampling is included in the optional additional characterization (as necessary) in **Table 5** and, if performed, should be conducted on the same day as the influent, effluent, and in plant samples.

4.1.3 Struvite Sampling

Struvite is an inorganic solid material harvested at the District by Ostara which is used to produce Crystal Green® fertilizer. The commercially available product is processed at the District to meet certain particle-size requirements. Any struvite that does not meet the particle-size specifications is collected in 55-gallon drums by the District. When the drums are full, the struvite is transferred to a sack. A sample of the out-of-specification struvite should be collected as a grab sample by using an HDPE or stainless-steel trowel to sample from the sack and placed in the sample container. A representative sample should be collected at a point below the surface of the bulk material. The initial characterization in **Table 5** includes PFAS characterization for one sample of struvite. The optional additional characterization (as necessary) includes further characterization of struvite using TOP Assay.

4.2 Additional Influent, Effluent, and In-Plant Characterization

Should the District determine the results of the initial characterization warrant further investigation, the District may also choose to collect samples at one or more of the points listed in the optional additional characterization in **Table 5**. The purpose for selecting the optional additional characterization may include:

- Understanding PFAS partitioning and transformations occurring in the Nine Springs WWTP
- Identifying specific sources of PFAS to the influent or isolate branch lines/areas with higher PFAS loading to the influent.
- Identifying PFAS contributions in the hauled waste

4.2.1 In-Plant Sampling and Analysis

In plant sampling can be used to evaluate PFAS partitioning and transformations occurring in the Nine Springs WWTP. The potential sample points in the treatment process are shown on **Figure 1** and **Figure 2**, and summarized in **Table 5**. Samples should be sent to a laboratory capable of producing results for the 36-compound Wisconsin list (not necessarily certified) and TOP Assay. Any of the optional in-plant sampling in **Table 5** should be conducted on the same day as the influent and effluent sampling. Sampling should follow the procedures in the SAB and additional notes in **Table 5**.

4.2.2 Upstream and Industrial Discharger Sampling and Analysis

The results of the influent testing of the five pumping stations in the initial characterization in **Table 5** may indicate that one or more areas within the District are contributing a mass of PFAS that warrants further investigation. The District may then choose to develop a specific sampling plan to identify the potential PFAS source(s) upstream of the pumping station(s) with elevated PFAS.

For the purpose of developing a scope for the optional additional characterization in **Table 5**, up to 10 upstream sampling locations are assumed at branch line(s) feeding into pumping station(s) with elevated PFAS and/or from the effluent from specific industrial or commercial discharger(s) feeding into the line(s). The samples may be collected as either grab or composite samples depending on location-specific factors including, but not limited to discharger(s) hours of operation, flow variability, and physical restrictions.

Upstream sampling locations should focus on the branches feeding pumping station(s) with elevated PFAS in the initial characterization and/or locations with highly probable PFAS contributors. The list of industries that may use PFAS is included in Appendix A in the Fate and Transport Report, and this list should be referred to when looking for potential PFAS contributors to the influent.

The general sampling procedures in **Appendix A** and the influent and effluent sampling procedure in Section 4.1.1 should be followed for this sampling. Industrial effluent samples should be collected at the compliance point and sent to a laboratory certified for the WI Criteria (or

EPA 1600/ 8328) and capable of producing the 36-compound list in Wisconsin (or required list at the time of testing) as described in Section 3.3. The combined MDLs for PFOS and PFOA for the selected laboratory must be below the ES of 20 ng/L for the analysis of industrial effluent samples.

4.2.3 Hauled Waste Sampling and Analysis

Because hauled waste accounts for approximately 0.23% of the total influent received by Nine Springs WWTP, sampling of this influent may not be needed. However, PFAS characterization of specific loads or general sources of hauled waste may be selected in the future, if needed to interpret the PFAS mass balance at the facility.

The sampling and analysis procedure for any hauled waste will depend of the material. Some materials will be able to use the general sampling procedures in **Appendix A** and the influent sampling procedure in Section 4.1.1, and others will need to be adapted to the unique matrix. Prior to sampling any hauled waste, the selected laboratory should be consulted to confirm the ability to analyze the sample and to identify their experience with these matrices.

4.3 Other Environmental Media Sampling

Sampling to address fate and transport in environmental media is not recommended at this time. The recommended approach by the WDNR is to focus on the reduction and elimination of PFAS sources, as explained in Section 2.0. Scope statements have been released by WDNR for the development of future State groundwater, surface water, and drinking water standards. If future sampling of environmental media is determined to be needed, adaptations to the general sampling procedure in **Appendix A** can be applied to sample media that are not currently required to be sampled, have been requested to be sampled for PFAS by the WDNR, or are of particular interest to the District. Surface water should be sampled using the same procedure in Section 4.1.1 for sampling wastewater by submersing the sample container below the surface of the water to collect a representative sample from the water column. Groundwater should be sampled using the general procedure in **Appendix A** and the usage of PFAS free sampling materials (pumps, permanent and temporary well installations, etc.) by sampling personnel and drillers experienced in sampling for PFAS. The analysis of groundwater and surface water should be performed by a laboratory that is certified for the WI Criteria (or EPA 1600/ 8328) and capable of producing the 36-compound list in Wisconsin (or required list at the time of testing) as described in Section 3.3. Drinking water sampling would follow the general procedure in **Appendix A** with the sample being collected directly into the sample container and using containers preserved with Trizma® supplied by the laboratory. Drinking water samples should be analyzed using EPA Method 537.1 for 18 PFAS compounds and by a laboratory that is certified for drinking water under NR 149.

The literature search in this study includes the review of publications where the sampling of other media such as sediment, air, and vegetation has been conducted using academic laboratories. The analysis of these other media would require the review of the extraction and analytical method procedures for PFAS by the laboratory for the selected matrix and include an agreement on the data quality objectives with the laboratory.

5.0 References

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Table 1: Comparison of Individual Compound Quantitative PFAS Methodologies or Established PFAS Method Criteria

Method or Criteria Reference	Matrix	Status	Date	Extraction Technique	Separation Technique	Analytical Technique	Isotope Dilution	# of Target PFAS Analytes	Summary
EPA Method 537 rev1.1	Drinking Water	Final	9/2009	Solid Phase Extraction	Liquid Chromatography	Tandem Mass Spectrometry	No	14	Drinking water method established for analyzing PFAS for UCMR 3 and has been updated to EPA method 537.1.
EPA Method 537.1	Drinking Water	Final	11/2018	Solid Phase Extraction	Liquid Chromatography	Tandem Mass Spectrometry	No	18	Updated the 14 compound list in EPA 537 rev 1.1 to include 4 additional replacement PFAS compounds. This method is frequently modified by laboratories to analyze non-potable aqueous and solid samples using isotope dilution for more than 18 PFAS compounds.
EPA Method 533	Drinking Water	Final	12/2019	Solid Phase Extraction	Liquid Chromatography	Tandem Mass Spectrometry	Yes	25	Draft methodology with an extraction technique targeted to capture short chain PFAS and additional replacement PFAS compounds. Eleven PFAS compounds are unique to method 533 and 14 overlap with method 537.1.
ASTM D7979-17	Water, Sludge, Influent, Effluent, and Wastewater	Final	2017	Direct Injection	Liquid Chromatography	Tandem Mass Spectrometry	No	21	A screening methodology that uses solvent extraction and external standard quantification. This method can be modified to include isotope dilution.
ASTM D7968-17	Soil	Final	2017	Solvent Extraction - Filtration - Direct Injection	Liquid Chromatography	Tandem Mass Spectrometry	No	21	A screening methodology that uses direct injection. This method can be modified to include isotope dilution.

Table 1: Comparison of Individual Compound Quantitative PFAS Methodologies or Established PFAS Method Criteria

Method or Criteria Reference	Matrix	Status	Date	Extraction Technique	Separation Technique	Analytical Technique	Isotope Dilution	# of Target PFAS Analytes	Summary
EPA Method 8327	Non-potable water and non-aqueous matrices	In response to public comments phase	6/1/2019	To be Determined	Liquid Chromatography	Tandem Mass Spectrometry	No	24	A screening methodology that uses direct injection that closely resembles ASTM D7979. Isotopically labeled internal standards are included as surrogates. The public comment period for this method ended 8/23/19.
DoD/ DOE QSM 5.1	Aqueous, Soil, Sediment	Final	1/3/2017	Solid Phase Extraction or Serial Dilution (aqueous) - Homogenization - Solvent Extraction (non-aqueous)	Liquid Chromatography	Tandem Mass Spectrometry	Yes	24+	Set of criteria for analysis, not a method. Criteria may be used to analyze any PFAS compound where a standard is available. Criteria in QSM 5.1 are common commercial laboratory modifications to EPA 537.1.
DoD/ DOE QSM 5.2	Aqueous, Solid, Biota, AFFF	Final	12/7/2018	Solid Phase Extraction or Serial Dilution (aqueous) - Homogenization - Solvent Extraction (non-aqueous)	Liquid Chromatography	Tandem Mass Spectrometry	Yes	24+	Updated from DoD QSM 5.1 set of criteria for analysis, not a method. Criteria may be used to analyze any PFAS compound where a standard is available. Criteria in QSM 5.2 are common commercial laboratory modifications to EPA 537.1.
DoD/ DOE QSM 5.3	Aqueous, Solid, Biota, AFFF	Final	5/3/2019	Solid Phase Extraction or Serial Dilution (aqueous) - Homogenization - Solvent Extraction (non-aqueous)	Liquid Chromatography	Tandem Mass Spectrometry	Yes	24+	Updated from DoD QSM 5.3 set of criteria for analysis, not a method. Criteria may be used to analyze any PFAS compound where a standard is available. Criteria in QSM 5.3 are common commercial laboratory modifications to EPA 537.1.

Table 1: Comparison of Individual Compound Quantitative PFAS Methodologies or Established PFAS Method Criteria

Method or Criteria Reference	Matrix	Status	Date	Extraction Technique	Separation Technique	Analytical Technique	Isotope Dilution	# of Target PFAS Analytes	Summary
Wisconsin PFAS Aqueous [Non-Potable Water] and Non-Aqueous Matrices Method Expectations	Non-potable water and non-aqueous matrices	Final	12/16/2019	Solid Phase Extraction (aqueous) Homogenization - Fortification - Solvent Extraction - Solid Phase Extraction (non-aqueous)	Liquid Chromatography	Tandem Mass Spectrometry	Yes	36	Set of criteria for analysis, not a method. Criteria were developed based on DoD QSM 5.2/5.3 criteria for analyzing PFAS along with criteria expected to be in draft EPA 1600/ method 8328 that will be promulgated in the future for the analysis of non-potable aqueous and solid samples.
EPA 1600/ EPA method 8328	Aqueous and Solids	Draft	TBD	TBD	Liquid Chromatography	Tandem Mass Spectrometry	Yes	25	Method is expected to be capable of analyzing non-potable aqueous and solid samples by incorporating some criteria in DoD QSM 5.3 and common commercial laboratory modifications to method 537.1.
ISO 25101	Aqueous	Final	3/1/2009	Solid Phase Extraction	Liquid Chromatography	Tandem Mass Spectrometry	No	2	Method only quantifies linear isomers of PFOS and PFOA in an aqueous matrix.
ISO 21675	Non-potable water	Final	10/2019	Solid Phase Extraction	Liquid Chromatography	Tandem Mass Spectrometry	No	30	Method specific for analyzing water matrices for PFAS.

Note:

TBD – To Be Determined

Table 2: PFAS Screening Techniques and Tools

Technique or Tool	Individual PFAS Compound Results	Limit of Detection	Summary
Particle-Induced Gamma Ray Emission (PIGE) Analysis	No	10 ug/L	Research method currently only available in Dr. Graham Peaslee's research laboratory at the University of Notre Dame. Uses excitation of light nuclei and detects the emission of characteristic gamma rays to determine total fluorine and has been successfully applied on the bench-scale. Development of this technique into a field-deployable technique is underway. Commercialization is estimated at over two years from now.
Total Fluoride by Ion Selective Electrode (ISE)	No	1 ppm	Inexpensive screening tool for total fluoride for solid or aqueous samples. Potentiometric determination of fluoride by multiple methods (Method 9214, ASTM D4646-87, ASTM D5233-92, ASTM D3987-85) yields results in the single digit parts per million range. Appropriate for screening highly concentrated sources, such as AFFF. Developing technologies could use ISE to screen in the field using an inexpensive portable electrode.
Total Organic Fluorine by Combustion Ion Chromatography (TOF-CIC)	No	0.05 mg F/kg	Destructive process that analyzes for organic fluorine by pyrohydrolysis and ion chromatography. References method LTM-INO-4370 that is derived from ASTM D7359-08. Analysis is commercially available in Australia and is used to screen soils for landfill acceptance. Estimated soil screening level is about 100 µg/kg. Limit of detection is based on organic fluorine mass which varies for each individual PFAS compound. Eurofins laboratory in Lancaster, PA is developing a version of this method that is expected to be commercially available in 2020.
Total Oxidizable Precursor (TOP) Assay	Yes	Method dependent	Artificial oxidization procedure using a strong oxidant and heat to forcibly oxidize and potentially hydrolyze precursor polyfluorinated PFAS. TOP assay includes the analysis of both a non-oxidized and oxidized sample by a method or modified method listed in Table 1. The oxidized sample represents the total potential PFAS mass of target analytes in the sample if oxidation of the precursor compounds were to occur in the environment. TOP assay will oxidize both target and non-target PFAS resulting in a better understanding of the total PFAS mass in a sample. The reporting limits for PFAS depend on the reporting limit of the selected PFAS analysis (typically 2-5 ng/L and 1-5 µg/kg per individual PFAS).
Quadrupole Time of Flight (qTOF) Mass Spectrometry	Yes	Compound Dependent (typically ~1 ng/L for perfluorinated compounds)	Utilized in academia and EPA's ORD; being developed commercially for high resolution mass spectrometry. qTOF readily captures the mass spectra of non-target analytes during the analytical run which allows for a forensic interpretation of the data. Research using qTOF is resulting in the development of library search capabilities to identify non-target analytes.

Table 3: Wisconsin 36 Compound List

13 Carboxylic Acids			12 Sulfonic Acids			7 Sulfonamides, Sulfonamidoacetic acids, Sulfonamidoethanols		
PFBA	Perfluorobutanoic acid	375-22-4	PFBS	Perfluorobutanesulfonic acid	375-73-5	FOSA	Perfluorooctanesulfonamide	754-91-6
PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeS	Perfluoropentanesulfonic acid	2706-91-4	N-MeFOSA	N-Methylperfluorooctanesulfonamide	31506-32-8
PFHxA	Perfluorohexanoic acid	307-24-4	PFHxS	Perfluorohexanesulfonic acid	355-46-4	N-EtFOSA	N-Ethylperfluorooctanesulfonamide	4151-50-2
PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpS	Perfluoroheptanesulfonic acid	375-92-8	N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
PFOA	Perfluorooctanoic acid	335-67-1	PFOS	Perfluorooctanesulfonic acid	1763-23-1	N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
PFNA	Perfluorononanoic acid	375-95-1	PFNS	Perfluorononanesulfonic acid	68259-12-1	N-MeFOSE	N-Methyl perfluorooctanesulfonamidoethanol	24448-09-7
PFDA	Perfluorodecanoic acid	335-76-2	PFDS	Perfluorodecanesulfonic acid	335-77-3	N-EtFOSE	N-Ethyl perfluorooctanesulfonamidoethanol	1691-99-2
PFUnA	Perfluoroundecanoic acid	2058-94-8	PFDoS	Perfluorododecanesulfonic acid	79780-39-5	4 Replacement Chemicals		
PFDoA	Perfluorododecanoic acid	307-55-1	4:2 FTS	4:2 Fluorotelomer sulfonic acid	757124-72-4	HFPO-DA	Hexafluoropropylene dimer acid	13252-13-6
PFTriA	Perfluorotridecanoic acid	72629-94-8	6:2 FTS	6:2 Fluorotelomer sulfonic acid	27619-97-2	DONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4
PFTeDA	Perfluorotetradecanoic acid	376-06-7	8:2 FTS	8:2 Fluorotelomer sulfonic acid	39108-34-4	9CI-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
PFHxDA	Perfluorohexadecanoic acid	67905-19-5	10:2 FTS	10:2 Fluorotelomer sulfonic acid	120226-60-0	11CI-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
PFODA	Perfluorooctadecanoic acid	16517-11-6						

Table 4: Laboratory Survey Results

Laboratory	Location	Intend to Certify	Date of Audit	Matrices Sought	Compound List	Compounds on WI PFAS List Excluded from Certification	# of PFAS Instruments	Average Number of Samples per Year	Analyst Years' Experience	WI PFAS Experience	Year of First PFAS Analysis	Additional Laboratory Comments
Eurofins TestAmerica	Sacramento, CA	Yes	TBD	All potable, non-potable and solids	36	None	Not Disclosed	50,000	13	Yes, WI 36 compound list	2006	Please note Eurofins TestAmerica considers this confidential business information that should not be disseminating outside of TRC. It is the laboratory's understanding that this information will be used solely to make a recommendation to TRC's client.
Vista Analytical Laboratory	El Dorado Hills, CA	Yes	Q4 2019	Potable water, Non-potable water, Solid and chemical waste (SCM), and Biological tissue (T)	36	None	4	10,000	3	Yes, >5 years	2008	Vista has extensive experience with wastewater, sludge and biosolids matrices.
SGS North America	Orlando, FL	Yes	Q1 2020	Non-potable water, soil, biosolids/ sludge, biota	36	MeFOSA, EtFOSA, MeFOSE, EtFOSE, PFHxDA, PFDoDA, PFDoDS, and 10:2-FTS will be added to current method for Q1 2020	3	10,000	6	Yes, DoD 24 compound list	2013	SGS North America has 3 labs performing PFAS Analysis. The facility in Sydney, BC (SGS Axys) will pursue tissue certification if offered by WI.
Wisconsin State Laboratory of Hygiene (WSLH)	Madison, WI	Yes	TBD	Non-potable water, soil, biosolids/ sludge, waste water, tissue (potentially avian serum)	31	Matrix dependent - Solids (FOSA, N-MeFOSA, N-MeFOSE, N-EtFOSA, N-EtFOSE) - Tissue (PFBA, N-MeFOSA, N-MeFOSE, N-EtFOSA, N-EtFOSE) - Waste Water (FOSA, N-MeFOSA, N-MeFOSE, N-EtFOSA, N-EtFOSE)	3	~500	3	Yes, <1 year, WI 36	2010	WSLH has worked with tissue and avian serum since about 2010. The analytical method has been updated based on the ISO method and the WDNR's 36 compound list. Method development is being continued by WSLH to expand from 31 compounds to the entire list of 36 compounds for each matrix. Drinking water by EPA 537.1 has been online since Spring of 2019.
GEL Laboratories, LLC	Charleston, SC	Yes	TBD	Non-potable water, soil, biosolids/ sludge, biota	36	None	3	300/ week maximum (received)	3	No	2017	GEL currently holds ISO 17025/DoD certification for 35 compounds.
ALS Environmental	Holland, MI	Yes	TBD	Non-potable water	36	None	1	TBD	1	No	2019	None
Pace Analytical	Minneapolis, MN	Yes	Q1 2020	Non-potable water, soils, sludge, drinking water, tissues, biosolids	36	None	4	2,600	12	No	1999	Pace has five laboratories with PFAS capabilities, including PFAS Mobile Lab capabilities (mobile lab is based in Madison, WI).
Merit Laboratories	Lansing, MI	Yes	TBD	Non-potable waste, soil/biosolids/ sludge	28-36	None	2	5,000	3	Yes, DoD 24 compound list	2018	Merit is a WBE certified small business analytical laboratory with NELAP(NY), ISO 17025, and DoD accreditation with ASTM D7979 (direct injection) capabilities.
Eurofins Lancaster	Lancaster, PA	TBD	TBD	TBD	TBD	TBD	6	>30,000	30	Yes	2007	Participated in Maine biosolids study
Alpha Analytical	Westborough, MA	No	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Participated in Maine biosolids study
Northern Lake Services	Crandon, WI	No	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Participated in analysis of PFAS samples for UCMR3 by EPA 537

Note:

N/A – Not applicable

Table 5: Sampling and Analysis Plan

Initial Characterization									
Map ID	Sample Name	Type	Sample Analysis & Quantity				Building Name (Number)	Data Gap	Notes
			PFAS	TOP Assay	TSS	Flow			
1A	Influent - Pump station 02	Influent	1	--	1	1	Headworks (2)	1	24-hour ISCO composite sample
1B	Influent - Pump station 07	Influent	1	--	1	1	Headworks (2)	1	24-hour ISCO composite sample
1C	Influent - Pump station 08	Influent	1	--	1	1	Headworks (2)	1	24-hour ISCO composite sample
1D	Influent - Pump station 11	Influent	1	--	1	1	Headworks (2)	1	24-hour ISCO composite sample
1E	Influent - Pump station 18	Influent	1	--	1	1	Headworks (2)	1	24-hour ISCO composite sample
2	Effluent	Effluent	1	--	1	1	Effluent (34)	4	From conveyance. To be collected as a composite sample over 24 hours and at same time or within one week of influent samples.
3A	Biosolids - Class A Cake	Biosolids	1	--	--	--	Biosolids End Use Facility (4)	4	From cake pile. To be collected at same time or within one week of influent samples.
3B	Biosolids - Class B (Metrogro®)	Biosolids	1	--	--	--	GBT (17)	4	From end of gravity belt thickener. To be collected at same time or within one week of influent samples.
4	Struvite (off-spec)	Struvite	1	--	--	--	Struvite Harvesting (40)	6	Off-spec product coming out of cyclones and stored on-Site in sacks
5A	Polymer 1	Polymer	1	1	--	--	WAS Thickening (41)	3	Pipe off tank, or manufacturer sample, if available.
5B	Polymer 2	Polymer	1	1	--	--	Sludge Dewatering (3)	3	Pipe off tank, or manufacturer sample, if available.
5C	Polymer 3	Polymer	1	1	--	--	GBT Polymer (6)	3	Pipe off tank, or manufacturer sample, if available.
--	Duplicates	Quality Control	4	--	--	--	--	--	One duplicate from influent; one duplicate from effluent, one duplicate from Class B (Metrogro®) biosolids; and one duplicate from polymer.
--	Field blanks	Quality Control	2	--	--	--	--	--	One field blank for every day of sampling, assumes two days at Nine Springs WWTP.
--	Equipment blanks	Quality Control	5	--	--	--	--	--	One equipment blank for influent; one equipment blank for effluent; one equipment blank for Class A Cake; one equipment blank for struvite; and one equipment blank for polymer.
Totals:			23	3	6	6			

Optional Additional Characterization (As Necessary)									
Map ID	Sample Name	Type	Sample Analysis & Quantity				Building Name (Number)	Data Gap	Notes
			PFAS	TOP Assay	TSS	Flow			
1A	Influent - Pump station 02	Influent	1	1	1	1	Headworks (2)	1	24-hour ISCO composite sample
1B	Influent - Pump station 07	Influent	1	1	1	1	Headworks (2)	1	24-hour ISCO composite sample
1C	Influent - Pump station 08	Influent	1	1	1	1	Headworks (2)	1	24-hour ISCO composite sample
1D	Influent - Pump station 11	Influent	1	1	1	1	Headworks (2)	1	24-hour ISCO composite sample
1E	Influent - Pump station 18	Influent	1	1	1	1	Headworks (2)	1	24-hour ISCO composite sample
2	Effluent	Effluent	1	1	1	1	Effluent (34)	4	From conveyance. To be collected as a composite sample over 24 hours and at same time or within one week of influent samples.
3A	Biosolids - Class A Cake	Biosolids	1	1	--	--	Biosolids End Use Facility (4)	4	From cake pile. To be collected at same time or within one week of influent samples.
3B	Biosolids - Class B (Metrogro®)	Biosolids	2	2	--	--	GBT (17)	4	From end of gravity belt thickener. To be collected at same time or within one week of influent samples. One sample to be submitted for immediate analysis; one sample to be held for 180 days then submitted for analysis.
4	Struvite (off-spec)	Struvite	1	1	--	--	Struvite Harvesting (40)	6	Off-spec product coming out of cyclones and stored on-Site in sacks
6	Primary sludge (from primary settling)	In-Plant (recycled waste)	1	1	--	--	Near Solids Gallery No. 1 (19)	5	Off hose from pipe in underground tunnel

Table 5: Sampling and Analysis Plan

Optional Additional Characterization (As Necessary)									
Map ID	Sample Name	Type	Sample Analysis & Quantity				Building Name (Number)	Data Gap	Notes
			PFAS	TOP Assay	TSS	Flow			
7	After primary settling	In-Plant (main process)	1	1	--	--	Outside of Blower Building No. 2 (7)	5	From conveyance
8	After secondary bio treatment	In-Plant (main process)	1	1	--	--	Outside of Aeration Gallery No. 2 (9)	5	From conveyance
9	WAS (from clarifier)	In-Plant (recycled waste)	1	1	--	--	Aeration Control Building No. 4 (25)	5	RAS 3 & 4, concentrated. 24-hour ISCO composite sampler.
10	Recycled Effluent	In-Plant (recycled waste)	1	1	--	--	Headworks (2)	5	From tap in screening room. To be collected as a composite sample over 24 hours and at same time or within one week of influent samples.
11	Combined WAS/Scum	In-Plant (main process)	1	1	--	--	DAF Thickener (20)	5	Solids from top of float tank
12	Liquids from DAF	In-Plant (recycled waste)	1	1	--	--	DAF Thickener (20)	5	Liquid sample from below solids in float tank
13	Before acid digestion	In-Plant (main process)	1	1	--	--	WAS Thickening (41)	5	Requires H2S gas meter, collected from same belt system as Class B biosolids (sample 3B)
14	After acid digestion	In-Plant (main process)	1	1	--	--	WAS Thickening (41)	5	Off hose in basement
15	Before thermophilic	In-Plant (main process)	1	1	--	--	GBT (17)	5	Off hose in basement
16	After thermophilic	In-Plant (main process)	1	1	--	--	Sludge Control Building No. 1 (11)	5	Off pipe
17	Liquid filtrate (recycled from end of struvite process)	In-Plant (recycled waste)	1	1	--	--	Struvite Harvesting (40)	6	Off pipe
18	Liquid filtrate (recycled to feed struvite process)	In-Plant (recycled waste)	1	1	--	--	Struvite Harvesting (40)	6	Off pipe
--	Influent - Upstream branch lines	Influent	10	--	--	10	To be determined	2	Sampling locations to be determined following initial characterization. Evaluate upstream branch lines and specific discharger(s) that feed into one or more of the five main pumping stations if elevated concentrations of PFAS are detected. To be collected as either grab or composite samples depending on location-specific factors.
--	Duplicates	Quality Control	8	--	--	--	--	--	One duplicate from influent; one duplicate from effluent, one duplicate from Class B (Metrogro®) biosolids; up to 3 duplicates from in-plant samples; and one duplicate from upstream influent.
--	Field blanks	Quality Control	5	--	--	--	--	--	One field blank for every day of sampling and/or general location, assumes up to 5 field blanks to account for multiple days of sampling and multiple locations outside Nine Springs WWTP for upstream influent sampling.
--	Equipment blanks	Quality Control	15	--	--	--	--	--	One equipment blank for influent; one equipment blank for effluent; one equipment blank for Class A Cake; one equipment blank for struvite. Up to 10 additional equipment blanks are included for the optional additional characterization of in-plant processes.
Totals:			61	23	6	16			

Notes:

- Specifications for fittings and tubing on ISCO samplers should be reviewed to determine composition and potential for cross-contamination. Alternatively, fittings and tubing can be replaced using PFAS-free components.
- If locations of aqueous samples are stagnant or have low flow, collection of a second sample may be warranted for a surface and submerged sample.
- In-plant samples should be collected directly from equipment; sample collection from hoses should be minimized to the extent practical to prevent potential cross-contamination.
- Data gaps are identified and described in the PFAS Fate and Transport Report prepared by TRC for the District in parallel with this SAB.



LEGEND

1

POTENTIAL SAMPLE LOCATION AND MAP ID

MAIN OPERATIONS OF NINE SPRINGS WASTEWATER TREATMENT PLANT

NOTES

1. BASE MAP IMAGERY FROM DANE COUNTY, 2017.

2. BASED ON FIELD OBSERVATIONS FROM JANUARY 22, 2020.

3. SAMPLING LOCATIONS ARE APPROXIMATE, REFER TO PHOTOGRAPHIC LOG AND SAMPLING AND ANALYSIS PLAN FOR ADDITIONAL DETAILS.

0200400
Feet

1" = 200'
1:2,400

PROJECT:

MADISON METROPOLITAN SEWERAGE DISTRICT
SAMPLING AND ANALYSIS BLUEPRINT
1610 MOORLAND ROAD, MADISON, WI 53713

TITLE:

POTENTIAL SAMPLING LOCATIONS

DRAWN BY:R. SUEMNICHT

CHECKED BY:M. URSIN

APPROVED BY:A. SELLWOOD

DATE:JANUARY 2020

PROJ. NO.:353946

FIGURE 1

TRC

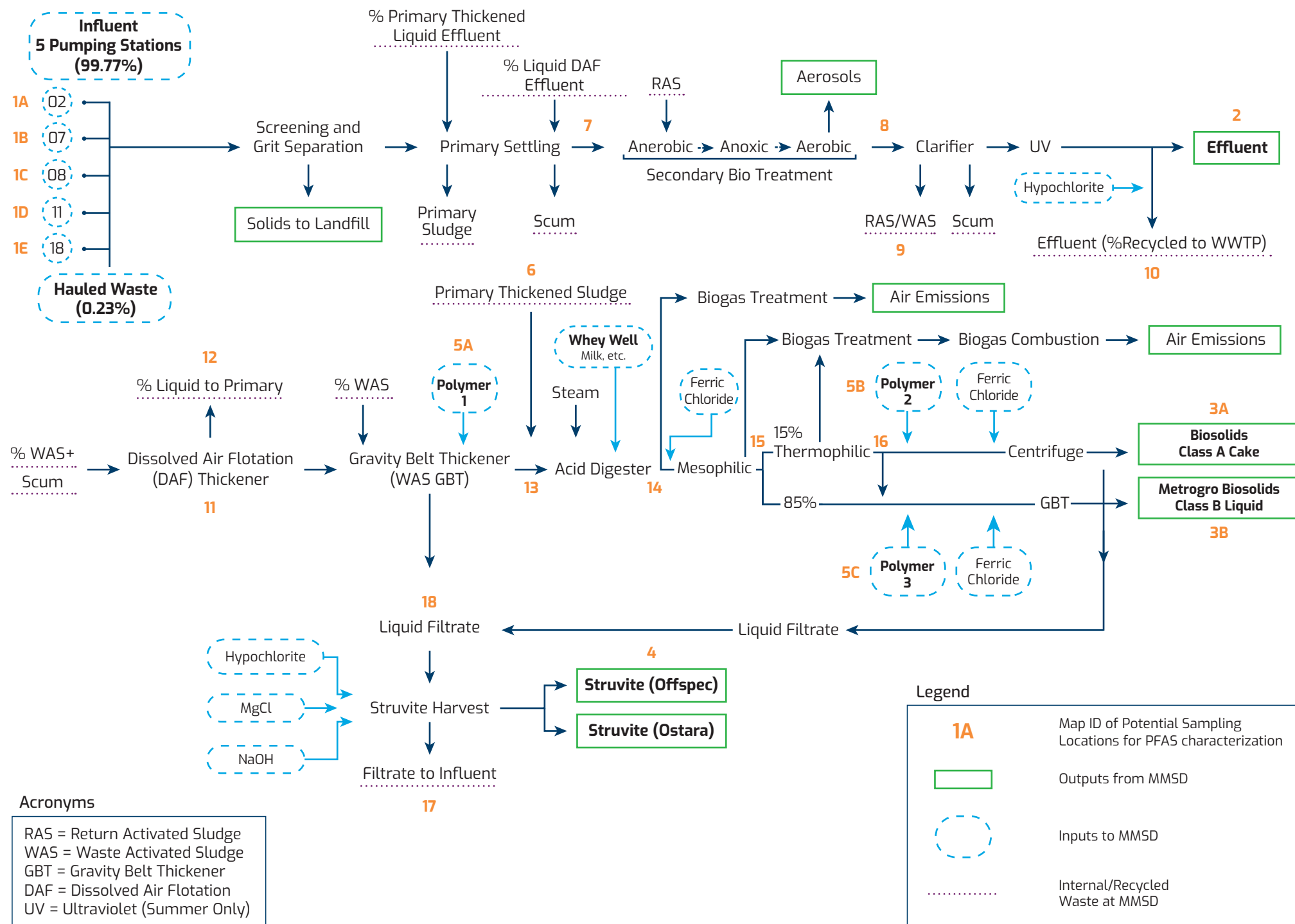
708 Heartland Trail, Suite 3000
Madison, WI 53717
Phone: 608.826.3600
www.trccompanies.com

FILE NO.:353946-001.mxd

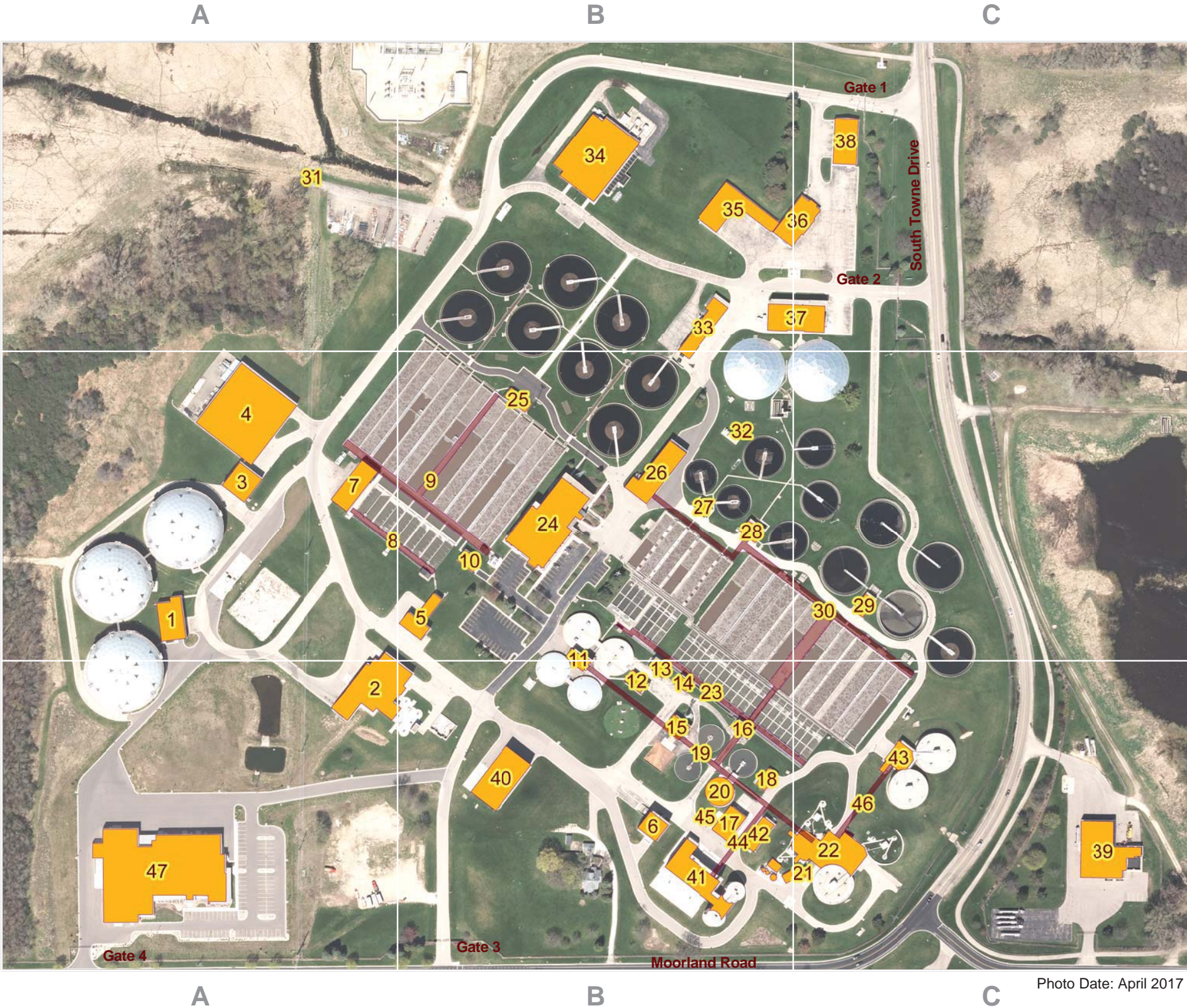
FIGURE 2

Nine Springs Wastewater Treatment Plant Process Flow Diagram

Madison Metropolitan Sewerage District



Building Number	Building Name	Grid Location
1	Metrogro Pumping Station	A2
2	Headworks Building	A3
3	Sludge Dewatering Building	A2
4	Biosolids End Use Facility	A2
5	Storage Building No. 3	B2
6	GBT Polymer Building	B3
7	Blower Building No. 2	A2
8	Primary Gallery No. 2	A2
9	Aeration Gallery No. 2	A2
10	Electrical Building U15	B2
11	Sludge Control Building No. 1	B3
12	Electrical Building U1	B3
13	Gas Control Building	B3
14	Primary Sludge Pumping Building No. 1	B3
15	Boiler Building	B3
16	Primary Sludge Pumping Building No. 2	B3
17	GBT Building	B3
18	DAF Building	B3
19	Solids Gallery No. 1	B3
20	DAF Thickener	B3
21	Oil Storage Building No. 1	C3
22	Sludge Control Building No. 2	C3
23	Primary Gallery No. 1	B3
24	Operations Building	B2
25	Aeration Control Building No. 4	B2
26	Blower Building No. 1	B2
27	Aeration Control Building No. 1	B2
28	Aeration Control Building No. 2	B2
29	Aeration Control Building No. 3	C2
30	Aeration Gallery No. 1	C2
31	Pumping Station No. 3	A1
32	Electrical Substation U2	B2
33	Service Building	B1
34	Effluent Building	B1
35	Storage Building No. 1	B1
36	Maintenance Shop No. 2	C1
37	Maintenance Shop No. 1	C1
38	Storage Building No. 2	C1
39	Vehicle Loading Building	C3
40	Struvite Harvesting Building	B3
41	WAS Thickening Building	B3
42	Electrical Building U3	B3
43	Sludge Control Building No. 3	C3
44	WAS Gallery	B3
45	Ferric Chloride Vault	B3
46	Solids Gallery No. 2	C3
47	Maintenance Facility	A3



Building Name -- Number	Grid Location
A	
Aeration Control Building No. 1 --27	B2
Aeration Control Building No. 2--28	B2
Aeration Control Building No. 3--29	C2
Aeration Control Building No. 4--25	B2
Aeration Gallery No. 1--30	C2
Aeration Gallery No. 2--9	A2
B	
Biosolids End Use Facility--4	A2
Blower Building No. 1--26	B2
Blower Building No. 2--7	A2
Boiler Building--15	B3
D	
DAF Building--18	B3
DAF Thickener--20	B3
E	
Effluent Building--34	B1
Electrical Building U1--12	B3
Electrical Building U15--10	B2
Electrical Building U3--42	B3
Electrical Substation U2--32	B2
F	
Ferric Chloride Vault--45	B3
G	
Gas Control Building--13	B3
GBT Building--17	B3
GBT Polymer Building--6	B3
H	
Headworks Building--2	A3
M	
Maintenance Facility--47	A3
Maintenance Shop No. 1--37	C1
Maintenance Shop No. 2--36	C1
Metrogro Pumping Station--1	A2
O	
Oil Storage Building No. 1--21	C3
Operations Building--24	B2
P	
Primary Gallery No. 1--23	B3
Primary Gallery No. 2--8	A2
Primary Sludge Pumping Building No. 1--14	B3
Primary Sludge Pumping Building No. 2--16	B3
Pumping Station No. 3--31	A1
S	
Service Building--33	B1
Sludge Control Building No. 1--11	B2
Sludge Control Building No. 2--22	C3
Sludge Control Building No. 3--43	C3
Sludge Dewatering Building--3	A2
Solids Gallery No. 1--19	B3
Solids Gallery No. 2--46	C3
Storage Building No. 1--35	B1
Storage Building No. 2--38	C1
Storage Building No. 3--5	B2
Struvite Harvesting Building--40	B3
V	
Vehicle Loading Building--39	C3
W	
WAS Gallery--44	B3
WAS Thickening Building--41	B3

Figure 3



Appendix A: General Sampling and Analysis Procedures

General Sampling and Analysis Procedures (Best practices for PFAS sampling)



1. Laboratory Selection: Selection depending on the matrix/ method of analysis:
 - a. Contact the laboratory and review the following:
 - i. Certification with NR 149 and confirm compound list
 - ii. SOP for each matrix
 - iii. Results of recent performance testing study
 - iv. Reporting Limits and MDLs for each of the Wisconsin 36 PFAS compounds
 - b. Communicate data quality objectives with the laboratory:
 - i. Confirm subsampling procedure for highly concentrated samples
 - ii. Confirm laboratory procedure for aqueous samples with high total solids (e.g., centrifuging or decanting for solids >1%, when is sample spiked, and are the solids analyzed)
 - iii. Confirm homogenization, fortification, and extraction procedure for solid samples
2. Request Bottleneckware: Use the selected laboratory's recommended containers (HDPE or polypropylene per WI Criteria) and preservation (required for drinking water samples):
 - a. Estimate the number of samples and include extra bottleneckware for breakage, etc.
 - b. Blank Scheme:
 - i. Equipment Blank(s): One blank for every set of equipment coming in contact with samples, per matrix. This blank is collected as a rinsate of the equipment using PFAS-free water. This blank should be collected after the decontamination process to be able to demonstrate effective decontamination and the lack of contamination coming from the equipment.
 - ii. Field Blank: One blank for every day of sampling. This blank is collected by pouring PFAS-free water into a sampling container while at the sampling site.
 - c. Request the laboratory provide PFAS-free water to be used for the equipment blank, field blank, and the final step of the decontamination process. The laboratory must be able to provide certification that the water is PFAS-free.
3. Sampling Procedure – Restricted Use Materials and Conditions:
 - a. Sampling staff should be directed to avoid using any equipment or materials containing PTFE, low density polyethylene, or other fluoropolymers during sample handling or mobilization/demobilization.
 - b. Sample volume collected should be as recommended by the laboratory.

- c. Avoid the use of:
 - i. Waterproof/water resistant paper products
 - ii. Post-it® notes
 - iii. Teflon® or Tyvek® materials
 - iv. Aluminum foil
 - v. PTFE tape
 - vi. Cosmetics
 - vii. Moisturizers and hand creams
 - viii. Personal care products including gel soaps and shampoos the day of sampling
 - ix. Insect repellants
 - x. Sunscreens
 - xi. Boots and other field clothing containing Gore-Tex™ or other waterproof/resistant material should not be worn (including rain gear). Polyurethane and polyvinyl chloride are acceptable.
 - xii. Clothing laundered with fabric softeners, new clothing, stain resistant clothing. Clothing made of cotton is preferred. Clothing should be well laundered since purchased.
 - xiii. Decon 90
 - xiv. Chemical (blue) ice packs. Use wet ice.
 - xv. Food and drink handling in the sampling area
- d. Equipment decontamination will include a final rinse with laboratory certified PFAS-free water in a new, clean 5-gallon bucket, or dispensed from an HDPE spray bottle.
- e. Sampling staff will wear nitrile gloves at all times while prepping sample containers, collecting samples, or handling samples. Avoid handling unnecessary items between donning gloves and handling or collecting the sample/sample containers. Don new gloves at any time during the sampling process if unnecessary items are contacted.
- f. Gloves should be changed between every sample.
- g. Field notes will be recorded on loose paper field forms maintained in aluminum or Masonite clipboards, or on a field tablet computer. Waterproof field books, plastic clipboards and spiral bound notebooks should not be used. Don new nitrile gloves between contacting the note-taking materials and handling the samples or sample containers.
- h. Individual sample bottles should be labeled using ballpoint pens or Sharpies®.

- i. A sample container should be opened immediately prior to sampling and capped immediately after sampling. Do not set the lid down.
- j. Samples bottles should be individually bagged with Ziploc® bags and returned to the sampling cooler on ice after sampling.
- k. The chain-of-custody should be filled out, signed, and placed in the cooler in a Ziploc® bag. Custody seals should be placed on the exterior of the cooler prior to delivering to the laboratory or shipping to the laboratory for next day receipt.

Appendix B: Photographic Log of Potential Sampling Locations

Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000
Photo No.	Date		
1	1/22/2020		
Description Map ID 1A - Overview of pumping station 02 in Headworks building. Influent sample to be collected from ISCO composite sampler at bottom of photo.			
Photo No.	Date		
2	1/22/2020		
Description Map ID 1B – Closeup of ISCO composite sampler for pumping station 02 in Headworks Building.			

Photographic Log







Client Name: Madison Metropolitan Sewerage District		Site Location: 1610 Moorland Road Madison, WI 53713	Project No.: 353946.0000
Photo No. 3	Date 1/22/2020		
Description Map ID 1B - Overview of pumping station 07 in Headworks building. Influent sample to be collected from ISCO composite sampler at bottom right of photo.			

Photo No. 4	Date 1/22/2020		
Description Map ID 1B – Closeup of ISCO composite sampler for pumping station 07 in Headworks Building.			



Photographic Log

Client Name: Madison Metropolitan Sewerage District		Site Location: 1610 Moorland Road Madison, WI 53713	Project No.: 353946.0000
Photo No. 5	Date 1/22/2020	<div> <div> Description Map ID 1C – Overview of pumping station 08 in Headworks building. Influent sample to be collected from ISCO composite sampler at bottom of photo. </div>  </div>	
Photo No. 6	Date 1/22/2020	<div> <div> Description Map ID 1C – Closeup of ISCO composite sampler for pumping station 08 in Headworks Building. </div>  </div>	

Photographic Log

Client Name: Madison Metropolitan Sewerage District		Site Location: 1610 Moorland Road Madison, WI 53713	Project No.: 353946.0000
Photo No. 7	Date 1/22/2020	<div>  </div>	
Description Map ID 1D – Overview of pumping station 11 in Headworks building. Influent sample to be collected from ISCO composite sampler at bottom of photo.			
Photo No. 8	Date 1/22/2020	<div>  </div>	
Description Map ID 1D – Closeup of ISCO composite sampler for pumping station 11 in Headworks Building.			

Photographic Log

Client Name: Madison Metropolitan Sewerage District		Site Location: 1610 Moorland Road Madison, WI 53713	Project No.: 353946.0000
Photo No. 9	Date 1/22/2020	<div> <div> Description Map ID 1E – Overview of pumping station 18 in Headworks building. Influent sample to be collected from ISCO composite sampler at bottom of photo. </div>  </div>	
Photo No. 10	Date 1/22/2020	<div> <div> Description Map ID 1E – Closeup of ISCO composite sampler for pumping station 18 in Headworks Building. </div>  </div>	

Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000





Photo No.	Date	
11	1/22/2020	
Description Map ID 2 – Effluent flows through a conveyance beneath the grate from the foreground to the background. Effluent sample to be collected by removing one of grates (cone is intended to approximate the location of sample collection). Located in the Effluent Building.		

Photo No.	Date	
12	1/22/2020	
Description Map ID 2 – Closeup of the conveyance discharge looking down from the platform from the top right of photo 11 in the Effluent Building.		


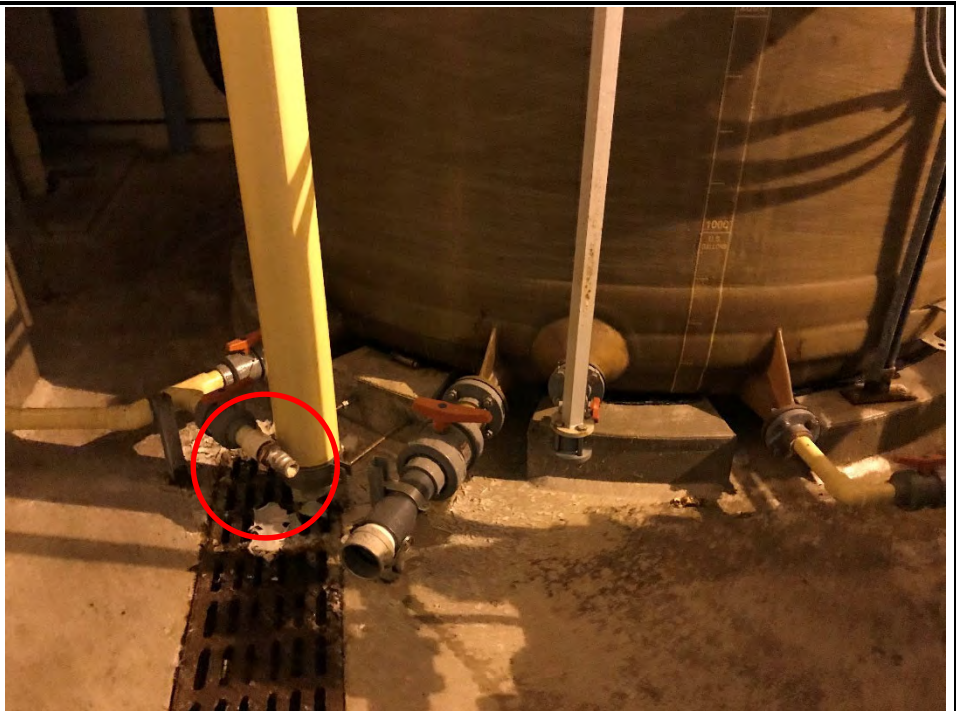
Photographic Log

Client Name: Madison Metropolitan Sewerage District		Site Location: 1610 Moorland Road Madison, WI 53713	Project No.: 353946.0000
Photo No. 13	Date 1/22/2020		
Description Map ID 3A – Biosolids-Class A Cake sampling location in southeast corner of Biosolids End Use Facility. Sample to be collected from a point below the surface of stockpile.			
Photo No. 14	Date 1/22/2020		
Description Map ID 3B – Biosolids-Class B Liquid (Metrogro®) sampling location in GBT Building. Sample to be collected from the end of the gravity belt thickener at center of photograph.			

Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000
Photo No.	Date		
15	1/22/2020		
Description Map ID 4 – Overview of the struvite sampling location in the Struvite Harvesting Building. The cyclones are used to separate off-specification struvite into 55-gallon drums. When full, the drums are dumped into the sack in the foreground. Sample to be collected from sack.			
Photo No.	Date		
16	1/22/2020		
Description Map ID 5A – Base of storage tank for Polymer 1 in WAS Thickening Building. Sample to be collected from pipe at base of tank (circled in red). Alternatively, a manufacturer-supplied sample could be analyzed.			

Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000
Photo No.	Date		
17	1/22/2020		
Description Map ID 5B – Base of storage tank for Polymer 2 in Sludge Dewatering Building. Sample to be collected from pipe at base of tank (circled in red). Alternatively, a manufacturer-supplied sample could be analyzed.			
Photo No.	Date		
18	1/22/2020		
Description Map ID 5C – Base of storage tank for Polymer 3 in GBT Polymer Building. Sample to be collected from pipe at base of tank (circled in red). Alternatively, a manufacturer-supplied sample could be analyzed.			

Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000







Photo No.	Date	
19	1/22/2020	
Description Map ID 6 – Primary sludge (from primary settling) sampling location in underground tunnel near Solids Gallery No. 1 Building. Sample to be collected from hose off pipe. End of hose circled in red at right.		

Photo No.	Date	
20	1/22/2020	
Description Map ID 7 – After primary settling sampling location outside Blower Building No. 2. Sample to be collected from conveyance flowing from left to right.		


Photographic Log

Client Name: Madison Metropolitan Sewerage District		Site Location: 1610 Moorland Road Madison, WI 53713	Project No.: 353946.0000
Photo No. 21	Date 1/22/2020		
Description Map ID 8 – After secondary bio treatment sampling location outside Aeration Gallery No. 2 Building. Sample to be collected from conveyance flowing from left to right.			
Photo No. 22	Date 1/22/2020		
Description Map ID 9 – WAS (from clarifier) sampling location inside Aeration Control Building No. 4. Sample to be collected from either ISCO composite sampler labelled as RAS 3 and RAS 4.			



Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000
Photo No.	Date		
23	1/29/2020		
Description Map ID 10 – Recycled effluent sampling location inside Headworks Building. Sample to be collected from spigot off pink effluent reuse pipe in the screening room.			
Photo No.	Date		
24	1/22/2020		
Description Map ID 11 – Combined WAS/Scum sampling location inside DAF Thickener Building. Sample to be collected from solids on top of process in the tank. Map ID 12 – Liquids from DAF Sampling location inside DAF Thickener Building. Sample to be collected from liquids beneath surface solids in the tank.			


Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000
Photo No.	Date		
25	1/22/2020		
Description Map ID 13 – Before acid digestion sampling location inside WAS Thickening Building. Room access requires an H2S gas meter and therefore the exact location was not directly observed. The belt system and sample collection will be the same as Map ID 3B.			
Photo No.	Date		
26	1/22/2020		
Description Map ID 14 – After acid digestion sampling location in basement inside WAS Thickening Building. Sample to be collected from hose off pipe. End of hose circled in red at right.			

Photographic Log

Client Name: Madison Metropolitan Sewerage District		Site Location: 1610 Moorland Road Madison, WI 53713	Project No.: 353946.0000
Photo No. 27	Date 1/22/2020		
Description Map ID 15 – Before thermophilic sampling location in basement inside GBT Building. Sample to be collected from hose off pipe. End of hose circled in red at right.			
Photo No. 28	Date 1/22/2020		
Description Map ID 16 – After thermophilic sampling location inside Sludge Control Building No. 1. Sample to be collected from spigot off pipe. Spigot is circled in red at right.			

Photographic Log

Client Name:		Site Location:	Project No.:
Madison Metropolitan Sewerage District		1610 Moorland Road Madison, WI 53713	353946.0000
Photo No.	Date		
29	1/22/2020		
Description Map ID 17 – Liquid filtrate (from end of struvite process) sampling location inside Struvite Harvesting Building. Sample to be collected from hose off pipe. End of hose circled in red at right.			
Photo No.	Date		
30	1/22/2020		
Description Map ID 18 – Liquid filtrate (recycled to feed struvite process) sampling location inside Struvite Harvesting Building. Sample to be collected from hose off pipe. End of hose circled in red at right.		