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# Study of Per- and Polyfluoroalkyl Substances in Bay Area POTWs: Phase 1 Sampling and Analysis Plan

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# **Study of Per- and Polyfluoroalkyl Substances in Bay Area POTWs: Phase 1**

## **Sampling and Analysis Plan**

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# 1. Introduction

This Sampling and Analysis Plan (SAP) details the plan associated with the Per- and Polyfluoroalkyl Substances Monitoring for Bay Area Publicly-Owned Treatment Works, Phase 1: Study design, coordination of sample collection, data quality assurance and reporting. This study was developed to investigate per- and polyfluoroalkyl substances (PFAS) in matrices from Bay Area publicly-owned treatment works (POTWs) to inform the monitoring strategy and program decisions for the Regional Monitoring Program for Water Quality in San Francisco Bay (RMP) and address monitoring needs for the State Water Board. The study is a two-part study, and this plan details the tasks associated with Phase 1 of the study. Phase 1 will analyze samples from a representative set of Bay Area POTWs to measure concentrations of PFAS in wastewater influent, effluent, biosolids, and reverse osmosis concentrate (ROC). Phase 2 (currently planned for summer 2021) will be informed by results from Phase 1 and will also include sampling of tertiary recycled water.

The objective of Phase I is to analyze samples from a representative set of Bay Area POTWs to measure concentrations of PFAS in wastewater influent, effluent, biosolids, and ROC. Since some Bay Area POTWs are moving towards recycling water, sampling ROC will provide data on PFAS concentrations in ROC and inform management actions. The POTWs included in this SAP were carefully selected to be representative of various characteristics of Bay POTWs, providing a representative sample set to analyze the range of PFAS concentrations in wastewater matrices and various characteristics that may influence PFAS concentration that could be later investigated. Service populations represented in the sample set range from entirely residential to those with notable industrial discharges. This sampling set was selected based on consideration of the following factors:

- **Discharge volume:** Sampling at the largest facilities is prioritized in order to capture dominant flows to the Bay. A few medium and small size facilities are also represented.
- **Service population and industries:** Chosen facilities include those with minimal industrial sources as well as those with a greater percentage of flows coming from industrial sources, particularly sources related to fabricated metals, electronic manufacturing, airports, and military bases.
- **Participation in previous Bay RMP PFAS study in 2014:** All facilities that participated in the previous PFAS study are included to evaluate changes in specific PFAS concentrations.
- **Treatment type:** Different secondary treatment technologies, including advanced secondary treatment processes, are included to understand potential impacts of the treatment processes on PFAS.
- **Geographic location:** Selected facilities are geographically diverse and represent all subembayments.

The specific characteristics of each facility can be found in Appendix C. All facilities will collect grab samples of each matrix. A subgroup of facilities will concurrently collect composite samples to compare to grab samples and inform an understanding of the differences between the sampling methodologies. All samples will be analyzed for targeted PFAS compounds listed in Section 8 (Laboratory and Analytical Methods). Terminal PFAS in influent and biosolids samples after oxidation will also be analyzed to

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understand the presence of important precursors and indicate the presence of other PFAS that may need to be identified and quantified. These efforts will help inform sampling design for Phase 2.

The specific objectives of the sampling effort are:

1. Collect influent, effluent, and biosolids samples for PFAS target and Total Oxidizable Precursors (TOP) analyses from the following POTWs:
  - Central Contra Costa Sanitary District (CCCSD)
  - City of San Mateo Wastewater Treatment Plant (CSM)
  - Dublin San Ramon Services District (DSRSD)
  - East Bay Municipal Utility District Main Wastewater Treatment Plant (EBMUD)
  - Fairfield-Suisun Sewer District (FSSD)
  - Novato Sanitary District (NSD)
  - Oceanside Water Pollution Control Plant (OSP), SFPUC
  - Palo Alto Regional Water Quality Control Plant (PA)
  - San Francisco International Airport Mel Leong Treatment Plant (SFO-S)
  - San Francisco International Airport Mel Leong Treatment Industrial Plant (SFO-I)
  - San Jose-Santa Clara Regional Wastewater Facility (SJ-SC)
  - Southeast Water Pollution Control Plant (SEP), SFPUC
  - Union Sanitary District (USD)
  - Vallejo Flood & Wastewater District (VFWD)
2. Collect effluent samples at East Bay Dischargers Authority (EBDA) for PFAS target analyses. EBDA receives treated wastewater effluent from several POTWs, including Union Sanitary and Dublin San Ramon Services District, which are included in this study. EBDA also receives effluent from the City of San Leandro, Oro Loma Sanitary District, Castro Valley, City of Hayward, Livermore-Amador Valley Water Management Agency, serving the Cities of Pleasanton and Livermore and DSRSD. Sampling discharges at EBDA provides the opportunity to sample the combined flows from all these POTWs at one sampling location. Sampling at the individual facilities (USD and DSRSD) will provide information about PFAS changes through the treatment process and evaluation of the results with the service population.
3. Collect reverse osmosis concentrate (ROC) at Valley Water (VW) and DSRSD for PFAS target analyses. Valley Water produces purified water at its Advanced Water Purification Facility (AWPF) by treating secondary effluent from SJ-SC through microfiltration, reverse osmosis, and ultraviolet disinfection. Reverse osmosis concentrate (ROC) is the reject water from the AWPF, which is currently mixed with advanced secondary treated effluent from SJ-SC before discharge to the Bay. In this study, ROC from the AWPF is collected for PFAS analysis. DSRSD receives ROC from Zone 7, which is mixed with effluent from DSRSD effluent before it is transported to EBDA. In this study, ROC from Zone 7 which will be collected at DSRSD and analyzed for PFAS separately from DSRSD secondary treated effluent. Schematic diagrams of sampling locations relative to treatment processes and flows are shown in Appendix D.

The RMP's Quality Assurance Program Plan (QAPP) (Yee et al., 2019) for field sampling design and analysis and Department of Defense (DoD) Table B-15 of Quality Systems Manual (QSM), version 5.3 for laboratory analysis of PFAS (DOD and Department of Energy (DOE), 2019), will be applied to this study unless otherwise stated. A short summary of relevant QAPP program requirements are included in this SAP for easy reference and review.

## 2. Key Personnel and Approvals

The personnel who will approve this SAP before it is finalized are shown in Table 1.

**Table 1. Key Personnel Approvals for this SAP.**

<b>Name</b>	<b>Affiliation</b>	<b>Duties</b>	<b>Initial and Date to Indicate Approval of Plan</b>
Diana Lin	SFEI	Project Manager/Lead Scientist	DL
Rebecca Sutton	SFEI	Lead Scientist	RAS 2020-11-23
Don Yee	SFEI	RMP QA Officer	DY 2020-11-16
Adam Wong	SFEI	RMP Data Manager	AW-2020-11-23
Lorien Fono	BACWA	Executive Director	LF-2020-12-2
Wendy Linck	State Water Board	Senior Engineering Geologist	WL-2020-12-1
Richard Grace	SGS AXYS	Director - Sales, Marketing, and Service	RG
Sean Campbell	SGS AXYS	Business and Technical Consultant	SC

The personnel who should be contacted in case of any questions regarding this SAP are shown in Table 2.

**Table 2. Key Personnel for PFAS sampling 2020 Contact**

<b>Name</b>	<b>Affiliation</b>	<b>Duties</b>	<b>Contact Information (email/phone/cell)</b>
Diana Lin	SFEI	Project Manager/Lead Scientist	diana@sfei.org (510) 746-7385 / (714) 932-8085
Miguel Mendez	SFEI	Environmental Analyst	miguelm@sfei.org (510) 746-7319 / (773) 698-5472

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Adam Wong	SFEI	RMP Data Manager	adamw@sfei.org (510) 746-7309
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The personnel who should be contacted at each participating POTW in case of any questions regarding PFAS monitoring are shown in Table 3.

**Table 3. POTW Contact Information**

<b>Name</b>	<b>Affiliation</b>	<b>Title/Duties</b>	<b>Contact Information (email/phone)</b>
Blake Brown	CCCSD	Senior Chemist	bbrown@centralsan.org (925) 229-7237
Mary Lou Esparza	CCCSD	Laboratory Superintendent	mesparza@centralsan.org (925) 335-7751
Tim Potter	CCCSD	Environmental Compliance Superintendent	tpotter@centralsan.org (925) 229-7380
Xiongbing Liang	CSM	Laboratory Supervisor	xliang@cityofsanmateo.org (650) 522-7388
Connie Sanchez	DSRSD	Senior Chemist	sanchez@dsrsd.com 925-875-2325
Angie Berumen	EBDA	City of San Leandro Lab Manager	ABerumen@sanleandro.org (510) 577-6042
Jackie Zipkin	EBDA	General Manager	jzipkin@ebda.org (510) 206-3820
Alicia Chakrabarti	EBMUD	Manager of WW Environmental Services	alicia.chakrabarti@ebmud.com (510) 287 2059
Nicole Van Aken	FSSD	Laboratory Manager	nvanaken@fssd.com (707) 428-9153
Liz Falejczyk	NSD	Environmental Services Supervisor, Veolia Water at NSD	lizf@novatosan.com (415) 892-1694 (ext 119)
Jennie Pang	OSP & SEP	Regulatory Specialist (SFPUC WWE)	jypang@sfwater.org (415) 934- 5762

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Samantha Bialorucki	PA	Lab Manager	Samantha.Bialorucki@CityofPaloAlto.org (650) 329-2334
Samantha Engelage	PA	Senior Engineer	Samantha.Engelage@CityofPaloAlto.org (650) 329-2123
Suguna Pillay	PA	Senior Chemist	Suguna.Pillay@CityofPaloAlto.org
Jennifer Acton	SFO-S & SFO-I	Acting Utility Manage	Jennifer.acton@flysfo.com (650) 821-8380
Eric Dunlavey	SJ-SC	Wastewater Compliance Manager	eric.dunlavey@sanjoseca.gov (408) 635-4017
Tim Grillo	USD	Research and Support Coach	timg@unionsanitary.ca.gov (510) 477-7561
Dan Jackson	USD	Lab Director	danj@unionsanitary.ca.gov (510) 517-1413
Anita Setty	VFWD	Environmental Services Superintendent	asetty@vallejowastewater.org (707) 652-7825
Medi Sinaki	VW	Senior Engineer	msinaki@valleywater.org (408) 630-2280

The personnel who should be contacted at SGS AXYS in case of any questions on analysis of PFAS are shown in Table 4.

**Table 4. Laboratory Contact Information**

Lab / Company	Name	Phone	Email	Shipping Address
SGS AXYS	Sean Campbell	(250) 655-5834	Sean.Campbell@sgs.com	2045 Mills Rd W V8L5X2 Sidney, British Columbia, CA

### 3. Sampling Schedule

**Table 5. Anticipated Schedule for 2020 BACWA PFAS Study, Phase 1**

Date	Activity
October 20, 2020	Draft Sampling and Analysis Plan
November 2020	Final Sampling and Analysis Plan

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November 6 - December 18, 2020	<p>Sample Collection. Avoid sampling on the week of November 23 to prevent shipping issues related to the Thanksgiving holiday.</p> <p>*Sampling during precipitation events expected to cause inflow and infiltration should be avoided to limit dilution of the wastewater signal. Sampling can resume when wastewater flows return to dry weather conditions. The precipitation criteria will be different for each facility.</p>
February 2021*	<p>Expected completion of analyses</p> <p>Laboratory analytical results are expected within 6 weeks of receiving final samples from facilities.</p>
February - April 2021*	<p>SFEI will upload analytical results to GeoTracker on behalf of facilities within 60 days of receiving final analytical results. SFEI will upload a monitoring report to GeoTracker within 90 days of receiving the final laboratory analytical report.</p>
Summer 2021*	<p>SFEI will provide a technical memo describing results of Phase 1 of this study</p>

\*Dates are subject to change based on adjustments to sampling plans

## 4. Sampling Procedure

The following guidelines were adapted using guidance from California State Water Quality Control Board (California State Water Resources Control Board, 2020), Michigan Department of Environmental Quality (Michigan Department of Environmental Quality, 2018) and current literature on PFAS background contamination (Bartlett and Davis, 2018; Rodowa et al., 2020). Recent studies examining sources of PFAS contamination during sampling indicate background contamination may not be as common as previously suggested. To be consistent with published guidance, previous studies, and in an abundance of caution, several materials are best avoided if they do not compromise safety or practicality.

### 4.1 General Sampling Guidelines

At each POTW, samples should be collected in the following order. The purpose of this order is to avoid contamination of samples by collecting the cleanest sample first.

1. Effluent or ROC (if applicable)
2. Influent
3. Biosolids

#### *Site Set-Up*

The sampling site should be evaluated prior to sampling to identify potential contamination risks and to select dedicated staging and sampling areas as defined below:

1. Eating Area: The eating area is separate from the sampling and staging areas, and the only place where food and drink should be stored and consumed. Food packaging must not be in the sampling and staging areas during sampling due to the potential for PFAS cross-contamination.

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2. **Staging Area:** The staging area is where equipment is set-up and personal protective equipment is put on and taken off. PFAS-free over-boots and PPE should be put on in the staging area prior to sampling activities.
3. **Sampling Area:** Sampling areas are the areas of the field where samples are collected. When staff need to leave the site, they should move to the staging area before removing gloves, coveralls, and any other appropriate PPE, if worn.

### ***Sample Collection***

Most samples for this study will be collected via grab sampling to minimize background contamination, increase method consistency, and best ensure each facility has the capabilities to meet sampling needs. Grab samples will be collected during each facility's peak flow. Collection during peak flow is expected to be more representative of total flows entering the POTW compared to other non-peak flow times. To compare the representativeness of grab samples collected during peak flow with 24-hour composites, a few facilities (CCCSD, FSSD, and SFO-I) will also be collecting daily composite influent and effluent samples. This will aid in understanding how grab samples compare with 24-hour composites in representing influent and effluent PFAS concentrations, while informing future sampling design.

### ***Field Sampling Form***

For all sampling events, please fill out the associated (aqueous or biosolids) field sampling form shown in Appendix A. The information requested specifically relates to each sampling event including sampling equipment used, procedures followed, and daily conditions at the POTW. Field sampling forms will be sent as excel files to each facility and include sampling IDs. The form may be completed after each sampling event and once all information requested is available. Please send completed forms to [diana@sfei.org](mailto:diana@sfei.org) and [miguelm@sfei.org](mailto:miguelm@sfei.org).

### **4.2 Sample Equipment: Acceptable and Prohibited Materials**

The typical field sampling environment has many potential sources of PFAS including sampling equipment, field documentation, personal protective equipment, clothing, and personal care products. As this can lead to background contamination, common materials in the field sampling environment have been separated into three categories as defined below:

**Acceptable Materials:** These materials are known not to be sources of PFAS cross contamination and can be used during all sampling stages and in the immediate sampling environment.

**Staging area-only materials:** These materials may contain PFAS and should not come into direct contact with the sample. These materials can be used in the staging area, but should be used away from all sampling equipment. Thoroughly wash hands and use new gloves after handling any of these materials.

**Prohibited materials:** These materials are known to contain PFAS that may present a threat to sample integrity and should not be used during any stage of the sampling events.

Each facility has been provided with a PFAS field sampling kit including sample containers and shipping materials to collect and ship all requested samples. The contents of each kit shipped to participating POTWs is found in Appendix B.

### ***All Sampling Equipment***

**Prohibited:** Any and all sampling equipment that contain PFAS-based (fluoropolymer) parts that would be in direct contact with the sample or sampling environment. These fluoropolymers include, but are not limited to:

- Polytetrafluoroethylene (PTFE), including the trademark Teflon® and Hostaflon®, which can be in the ball lining of some hoses and tubing, and some objects that require the sliding action of parts.
- Polyvinylidene fluoride (PVDF), including the trademark Kynar®, which can be in tubing and films/coatings on aluminum, galvanized or aluminized steel.
- Polychlorotrifluoroethylene (PCTFE), including the trademark Neoflon®, which can be in many valves, seals, and gaskets.
- Ethylene-tetrafluoro-ethylene (ETFE), including the trademark Tefzel®, which can be in many wire and cable insulation and covers, liners in pipes, and some cable tie wraps.
- Fluorinated ethylene propylene (FEP), including the trademarks Teflon® FEP and Hostaflon® FEP, and may also include Neoflon®, which can be in wire and cable insulation and covers, pipe linings, and some labware.

**Staging area-only:** Low density polyethylene (LDPE) should be avoided if it comes into direct contact with the sample. If absolutely necessary, LDPE parts may be used if an equipment blank has confirmed it is PFAS-free. LDPE resealable storage bags (i.e., Ziploc bags) may be used for storage and shipping.

### ***Sample Containers***

**Acceptable:** High-density polyethylene (HDPE) containers of various sizes (500 mL, 250 mL, 125 mL, and/or 60 mL) provided by SGS AXYS.

### ***Pumps, Tubing and Sampling Instruments***

**Acceptable:** Supplies must be made from acceptable materials known to be PFAS free, which include HDPE, polypropylene, silicone, stainless steel, nylon (e.g., cable ties), polyvinyl chloride (PVC), acetate, and cotton. Glass may be used as long as it is known to be PFAS-free (or decontaminated; see Section 4.3) and comes into contact with the sample for a short period of time (not appropriate for storage).

To collect composite samples, automatic samplers may be used though there may be an increased potential for cross-contamination because the tubing, valves, strainers, suction lines, distribution nozzles, and other parts may be made from PFAS (fluoropolymers). It is recommended that parts on the sampler be screened prior to sampling by reviewing the safety data sheets (if available) and collection of an equipment blank to verify that the parts are PFAS-free.

### ***Field Documentation***

**Acceptable:** Ballpoint pens and Sharpie® markers (only fine or ultra-fine) for writing and labeling. Loose paper (non-waterproof, non-recycled) as well as aluminum, polypropylene, or Masonite field clipboards may be used.

**Staging area-only:** Rite in the Rain® notebooks, provided gloves are changed after note taking.

**Prohibited:** Regular and thick sized markers of any brand, sticky notes, plastic clipboards, or waterproof paper and notebooks.

### ***Personal Protective Equipment and Other Clothing***

**Acceptable:** Synthetic or 100% cotton clothing that has been well-laundered without the use of fabric softeners. Any clothing (including shoes) made of or with polyurethane, PVC, wax coated fabrics, rubber and neoprene. Powderless nitrile gloves for all sampling events.

**Staging area-only:** Non PFAS-free boots and first aid adhesive wrappers.

**Prohibited:** Latex gloves, new or unwashed clothing, any clothes recently treated with fabric softeners, fabric protectors, insect resistance and water/stain/dirt-resistant chemicals. Anything made with water/stain/dirt-resistant fabrics such as Coated Tyvek®, Gore-Tex®, Scotchgard™, and RUCO®.

Personal safety is paramount and should not be compromised to prevent cross-contamination. Therefore, if the use of PPE is necessary to ensure the health and safety of sampling personnel and no PFAS-free alternative is available, then note the use in the field sampling form. Please wash hands and change gloves after handling any PFAS containing products (including items designated only to the staging-area).

### ***Personal Care Products***

**Staging area-only:** Sunscreens and insect repellents. preferably from products known to not contain PFAS (nonexhaustive list provided from the [Michigan PFAS Sampling Quick Reference Field Guide](#)).

**Prohibited:** Application of any PCPs in the sampling area.

If possible, please try to avoid use of personal care products (hair products, make-up, perfume/cologne, moisturizers, etc.) on the day of sampling. If any are used on the day of sampling, record in the field sampling form.

### ***Food Packaging Materials***

**Prohibited:** PFAS are known to be prevalent in food packaging, including paper plates, aluminum foil, paper towels, food containers, bags, and wraps. Food and beverages should not be consumed at the sampling site. If they must be consumed during the sampling event, a dedicated eating area should be identified (see section 4.1).

## **4.3 Sample Equipment Cleaning and Decontamination Procedures**

Sample equipment that comes into contact with the sampling media (i.e., buckets, carboys, extension rods, scoops, tubing, parts of automatic samplers) should be cleaned and decontaminated (or new) prior to use where possible. Automatic samplers should be decontaminated, or the strainer replaced between each sampling event. If new tubing is used, decontamination procedures are not necessary. Sampling equipment can be scrubbed using a polyethylene or PVC brush to remove particulates.

The following procedure is recommended for cleaning and decontamination: Wash with PFAS-free soap (i.e., Alconox®), scrub (if applicable). Follow up with a methanol rinse and rinse with PFAS free water. The laboratory will provide PFAS free reagent water for a final rinse collected for an equipment blank. Please note if this, or any other cleaning method, has been used in the field sampling form.

#### **4.4 Aqueous Sampling Guidelines**

**Tables 6 and 7** detail the aqueous samples each POTW will be collecting for PFAS analysis. The contents of the table as they relate to sample guidelines are summarized below:

The following facilities will focus only on **grab** sampling for influent, effluent, and/or ROC:

- City of San Mateo Wastewater Treatment Plant (CSM)
- Dublin San Ramon Services District (DSRSD)
- East Bay Dischargers Authority (EBDA),
- East Bay Municipal Utility District Main Wastewater Treatment Plant (EBMUD)
- Novato Sanitary District (NSD)
- Oceanside Water Pollution Control Plant (OSP)
- Palo Alto Regional Water Quality Control Plant (PA)
- Southeast Water Pollution Control Plant (SEP)
- San Francisco International Airport Mel Leong Treatment Plant (SFO-S)
- Vallejo Flood & Wastewater District (VFWD)
- Valley Water (VW)

Two facilities will conduct **grab** sampling of influent and effluent with additional collection of **duplicate and/or equipment blank samples**:

- San Jose-Santa Clara Regional Wastewater Facility (SJ-SC)
- Union Sanitary District (USD)

The remaining facilities will run **both grab and composite** sampling for influent and effluent while also taking **duplicates (grab and composite), field blanks, and equipment blanks**:

- Central Contra Costa Sanitary District (CCCSD)
- Fairfield-Suisun Sewer District (FSSD)
- San Francisco International Airport Mel Leong Treatment Industrial Plant (SFO-I)

The following protocols should be followed when collecting any aqueous PFAS samples:

- Powderless nitrile gloves must be worn on hands before collecting samples, handling sample containers, or handling sampling equipment.
- The sample container must be kept sealed and only opened during sample collection. The sampling container cap or lid should never be placed on the ground or on any other surface unless it is PFAS-free. If it is necessary to set the cap down, it should be set on a clean surface (cotton sheeting, HDPE sheeting, triple rinsed cooler lid, etc.).
- Do not insert or let tubing or any materials inside the sample bottle. Dust and fibers must be kept out of sample bottles.
- Samples containers should be filled to 80-90% capacity (providing 10-20% volume headspace) to allow for expansion during freezing (all samples will be frozen upon receipt at the laboratory). Final volumes should correspond to roughly 400-450 mL (500 mL container), 100-110 mL (125 mL), and 48-55 mL (60 mL).

**Table 6. Influent Samples collected at each POTW.**

Facility	Influent Grab (125 mL)	Influent Grab Duplicate (125 mL)	Influent Grab Back-Up (125 mL)	Influent Grab + Backup (60 mL)	Influent Composite (125 mL)	Influent Composite Duplicate (125 mL)	Influent Composite Back-Up (125 mL)	Influent Field Blank (Pre-filled 500 mL)	Influent Equipment Blank (500 mL)	Total Influent Samples Collected
	PFAS Target Analysis			PFAS TOP Analysis	PFAS Target Analysis			QA/QC		
CCCSD	2	1	3	2	2	1	3	1	1	16
CSM	1	-	1	2	-	-	-	-	-	4
DSRSD	1	-	1	2	-	-	-	-	-	4
EBMUD	1	-	1	2	-	-	-	-	-	4
FSSD	2	1	3	2	2	1	3	1	2**	17
NSD	1	-	1	2	-	-	-	-	-	4
OSP	1	-	1	2	-	-	-	-	-	4
PA	1	-	1	2	-	-	-	-	-	4
SEP	1	-	1	2	-	-	-	-	-	4
SFO-S	1	-	1	2	-	-	-	-	-	4
SFO-I	2	1	3	2	2	1	3	1	1	16
SJ-SC	1	4*	1	2	-	-	-	-	-	8
USD	1	-	1	4^	-	-	-	-	1	7
VFWD	1	-	1	2	-	-	-	-	-	4

Sample container size noted in parenthesis. Sample containers should be filled with 10-20% headspace to allow for expansion upon freezing.

Facilities highlighted in blue are collecting grab and composite samples to compare sampling methods

\* Sampling includes two Matrix Spike (MS) and two Matrix Spike Duplicate (MSD)

\*\*Sampling includes an equipment blank from the composite sampler and an equipment blank from the stainless steel bucket used to collect the grab sample.

^Sampling includes a duplicate influent grab and back-up that will be used for TOP analysis.

**Table 7. Effluent Samples collected at each POTW.**

Facility	Effluent Grab (500 mL)	Effluent Grab Duplicate (500 mL)	Effluent Grab Back-Up (500 mL)	Effluent Composite (500 mL)	Effluent Composite Duplicate (500 mL)	Effluent Composite Back-up (500 mL)	Effluent Field Blank (Pre-filled 500 mL)	Effluent Equipment Blank (500 mL)	Total Effluent Samples Collected
	PFAS Target Analysis						QA/QC		
CCCS	2	1	3	2	1	3	1	1	14
CSM	1	-	1	-	-	-	-	-	2
DSRSD	2**	-	2**	-	-	-	-	-	4
EBDA	1	-	1	-	-	-	-	-	2
EBMUD	1	-	1	-	-	-	-	-	2
FSSD	2	1	3	2	1	3	1	1	14
NSD	1	-	1	-	-	-	-	-	2
OSP	1	-	1	-	-	-	-	-	2
PA	1	-	1	-	-	-	-	-	2
SEP	1	-	1	-	-	-	-	-	2
SFO-S	1	-	1	-	-	-	-	-	2
SFO-I	2	1	3	2	1	3	1	1	14
SJ-SC	1	4*	1	-	-	-	-	-	6
USD	1	-	1	-	-	-	-	1	3
VFWD	1	-	1	-	-	-	-	-	2
VW	2	1	3	-	-	-	-	-	6

Sample container size noted in parenthesis. Sample containers should be filled with 10-20% headspace to allow for expansion upon freezing.

Facilities highlighted in blue are collecting grab and composite samples to compare sampling methods

Facilities highlighted in gray are collecting reverse osmosis concentrate (ROC)

\* Sampling includes two Matrix Spike (MS) and two Matrix Spike Duplicate (MSD)

\*\* Sampling includes one effluent sample and one ROC sample

### **Grab Sampling**

Influent and effluent samples will be collected during peak flow at each facility to ensure capture of dominant flows. Wastewater influent and effluent will be directly collected in HDPE containers (500 mL, 125 mL or 60 mL) provided by SGS AXYS. If this is not possible, a beaker made of a known PFAS-free material (examples of allowable materials listed above) may be used to pour into the HDPE container. It is acceptable to collect chlorinated effluent samples and ship to the laboratory without any further treatment. Please note if samples are chlorinated in the field sampling form.

Various types of immersion sampling equipment may be used for sampling. Equipment used must be PFAS-free (see section 4.2), new or decontaminated (see section 4.3), and may include extension rods to immerse the laboratory sample bottle at the sample location, cable ties, beakers, and peristaltic pumps with tubing that extends into the wastewater. If the sampling bottle can not be used to directly sample, a sampling port or pump may be used instead. Sampling locations for all facilities are shown in process diagrams in Appendix D. Please document the use of any equipment or materials that come in direct contact with the sample and any change in sampling location in the field sampling form.

Weekend flow patterns at POTWs tend to be different from weekday flows patterns due to differences in activities from the serviced population. **Samples should be collected at peak flow on a weekday to capture representative weekday flows. Sampling should be avoided on weekends and Mondays, because Mondays may be influenced by weekend flows. If samples are collected on Thursday or Friday, please freeze samples and ship to SGS AXYS on Monday.**

### ***Influent***

Influent samples should be collected at a location and in a manner that is representative of all influent received by the facility prior to treatment. Influent samples should be collected in a well mixed location prior to primary settling, which include but are not limited to the headworks of the inlet to the grit chamber or prior to any biological treatment. If possible, samples should be collected after bar screening and grit removal but before fine screening to obtain a representative influent sample. Please note any treatment processes before the influent sampling location in the field sampling form. The sampling location is also marked in the facility diagrams in Appendix D.

Each 125 mL influent field sample will have a back-up sample collected in a separate 125 mL HDPE container. In addition, two 60 mL samples will be collected to conduct TOP analysis. This means each influent grab sampling event will consist of two 125 mL samples and two 60 mL samples (both PFAS target and TOP analysis will be conducted on influent samples). Since collecting a 60 or 125 mL grab sample directly into a container may be difficult, it is suggested that the sample can be poured off from a larger beaker. A 500 mL HDPE container (extras provided) could be used to pour into a smaller container.

### ***Effluent***

Effluent should be collected at a location and in a manner that is representative of final effluent discharged to receiving waters. Only 500 mL effluent samples will be collected (PFAS target analysis will be conducted on effluent samples). Each 500 mL effluent field sample will have a 500 mL back-up collected.

## ***ROC***

ROC samples should be collected at a location and in a manner that is representative of the final ROC discharged to receiving waters. A grab sample and field duplicate will be collected on the same day and time (to capture variation from sampling the same day and time). The second field sample will be collected on any different day of the week. This is being done to capture the day-to-day variation. Only 500 mL ROC samples will be collected (PFAS target analysis will be conducted on ROC samples). Each 500 mL ROC field sample will have a 500 mL back-up collected.

## ***Field Duplicate including Matrix Spike (MS) and Matrix Spike Duplicate (MSD)***

For grab sampling, field duplicates are replicate samples collected in the field and submitted to the laboratory as two different samples. Field duplicates can be used to evaluate both field and laboratory precision. Influent and effluent field duplicates will be collected in HDPE containers (500 mL or 125 mL) provided by SGS AXYS using the same procedures noted above.

Two 60 mL field duplicates will be collected at USD at the same time as the other 60 mL samples being used for TOP analysis (4 samples total). The MS and MSD will be collected as 500 mL duplicate samples at SJ-SC at the same time as influent and effluent sampling events.

Full instructions for duplicates for facilities conducting both grab and composite sampling (CCCSD, FSSD, SFO-I) are noted in the composite sampling section (below).

## ***Field Blank***

The field blank is collected to verify that the sampling environment does not introduce PFAS and cross-contaminate samples during the sampling event. The field blank is collected by opening a 500 mL container pre-filled with PFAS-free water (provided by SGS-AXYS) while collecting the grab sample. The field blank is treated the same throughout field and laboratory procedures as other collected field samples. A field blank will be collected at four facilities (CCCSD, FSSD, SFO-S, SFO-I) to be representative of typical POTW sampling environments.

## ***Equipment Blank***

For grab sampling, only USD will be collecting an equipment blank due to its particular sampling set-up. Equipment blanks are collected from a final rinse by passing PFAS-free reagent water (provided by SGS-AXYS) over or through field sampling equipment prior to sample collection to assess the adequacy of the decontamination process and evaluate potential contamination from the equipment used. Each equipment blank should fill up to 500 mL (450 mL if frozen) in the provided HDPE containers.

## **Composite Sampling**

Influent and effluent samples at CCCSD and FSSD will be collected as 24-hour composites using autosamplers concurrently with grab sampling (same days). HDPE containers (500 mL) can be filled directly from the autosampler or poured from a PFAS-free container where a larger composite is collected. If possible, new tubing should be used for each sampling event. Automatic samplers should be decontaminated or strainer replaced between each sampling event. Record if tubing and strainers were decontaminated prior to use or new in the field sampling form.

Manual composites will be collected at SFO-I. This will require the collection of several grab samples (at least 4) spaced evenly through a 24-hour period and composited together. The sample should be

well-mixed to ensure solids are homogenized and the sample is representative of the solids content. It is acceptable to collect chlorinated effluent samples and ship to the laboratory without any further treatment. Further details for manual compositing should be included in the field sampling form.

### ***Field Duplicate***

For composite sampling, field duplicates are replicate or split samples collected in the field and submitted to the laboratory as two or more different samples. Influent and effluent field duplicates will be collected in HDPE containers (125 or 500 mL) provided by SGS AXYS using the same procedures as in the sections above.

Facilities performing both grab and composite sampling (CCCSD, FSSD, SFO-I) will collect a total of three grabs and three composites. A field duplicate will be collected with the first field composite sample. Ideally, the field duplicate is a true replicate, where the sample is collected the same day but from different sips from the composite sampler. For example, if the normal sampling procedure is hourly sips into the composite bottle, the field replicate would be a second composite bottle; hourly sips would first go in Bottle A, then another in bottle B, repeated throughout the day. Another option is for replicates to be collected from a separate autosampler at the same location. If these options are not possible, then the field duplicate could be a split sample poured from a larger composite bottle on the same day, making sure the larger composite bottle is well-mixed and homogenized before pouring. The third sample will be collected as a separate field sample on a different day of the week to capture daily variations. The purpose of these samples are to determine how grab samples compare with 24-hour composites in representing influent and effluent PFAS concentrations. This evaluation will inform how representative wastewater samples should be collected in future PFAS POTW studies.

### ***Equipment Blank***

For automatic samplers, equipment blanks are collected from a final rinse by passing PFAS-free reagent water (provided by SGS-AXYS) over or through field sampling equipment (i.e., tubing) before the collection of samples. Each equipment blank should fill up to 500 mL (or 450 mL if the sample will be frozen) in the provided HDPE containers. If a larger composite container is used to collect samples prior to pouring into the sample container, the composite container should also be rinsed.

### **Sample Storage**

It is recommended that aqueous samples to be analyzed for PFAS be frozen (below 0°C) as soon as possible. When frozen, the hold time for wastewater influent and effluent is 90 days from collection. If samples cannot be frozen on site after collection, samples should be shipped immediately to SGS AXYS (see section 7). Samples will be frozen there when they arrive.

## **4.5 Biosolids Sampling Guidelines**

**Table 8** details the biosolids samples each POTW will be collecting for PFAS analysis. The contents of the table as related to sample guidelines are summarized below:

All facilities listed will collect **biosolid grab samples** (CCCSD, CSM, DSRSD, EBMUD, FSSD, NSD, OSP, PA, SJ-SC, SEP, SFO-S, USD, VFWD). SFO-I does not produce biosolids; instead the solids separated from the wastewater treatment process will be sampled.

**Table 8. Biosolids samples collected at each POTW.**

Facility	Biosolids (250 mL)	Biosolids Duplicate (250 mL)	Biosolids Field Blank (Pre-filled 500 mL)	Biosolids Equipment Blank (500 mL)	Total Biosolids Samples Collected
	Target and TOP Analysis		QA/QC Target Analysis		
<b>CCCSD</b>	3*	-	1	1	5
<b>CSM</b>	1	-	-	-	1
<b>DSRSD</b>	1	-	-	-	1
<b>EBMUD</b>	1	1	-	-	2
<b>FSSD</b>	1	-	1	-	2
<b>NSD</b>	1	-	-	-	1
<b>OSP</b>	1	-	-	-	1
<b>PA</b>	1	-	-	-	1
<b>SEP</b>	1	-	-	-	1
<b>SFO-S</b>	1	1	1	1	4
<b>SFO-I</b>	1**	-	-	-	1
<b>SJ-SC</b>	1	1	-	-	2
<b>USD</b>	1	1	-	-	2
<b>VFWD</b>	1	-	-	-	1

*Sample container size noted in parenthesis. Sample containers should be filled half-way (125 mL).*

*\* Facility has various biosolids waste streams*

*\*\* Facility does not produce biosolids; instead the solids seperated from the treatment process will be sampled.*

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Some facilities will also collect a **field duplicate** to assess the precision of field and laboratory activities: EBMUD, SJ-SC, SFO-S, USD. Differences between the field sample and field duplicates calculated from these facilities will be used to assess the precision for all facilities.

Three facilities will also collect **equipment blanks and/or field blanks** to assess the potential for contamination in field and laboratory activities: CCCSD, FSSD, SFO-S.

### **The following protocols should be followed when collecting any biosolids PFAS samples:**

- Powderless nitrile gloves must be worn on hands before collecting samples, handling sample containers, or handling sampling equipment.
- The sample container must be kept sealed and only opened during sample collection. The sampling container cap or lid should never be placed on the ground or on any other surface unless it is PFAS-free.

### **Biosolids Sampling**

A biosolids sample should be collected from the final step in the treatment process at each facility to represent the final product (highest solids content possible) that is produced and removed from each POTW. If liquids are present, a representative whole sample aliquot that includes both liquid and solid fractions should be collected.

Samples should be collected in a way that is representative of biosolids produced by the facility. A single grab sample is appropriate if collected from a well-mixed treatment process. The 250 mL HDPE containers should be filled half-way (125 mL) by directly pouring or scooping from a well mixed location. If biosolids piles are heterogenous, several grab samples may be collected and composited to create a representative sample of biosolids. Please note the sampling method in the field sampling form.

To fully assess the presence of PFAS in the biosolids treatment process, CCCSD will be collecting three biosolids samples. The first two samples (sludge cake and scum) will be collected before the biosolids are incinerated. A wet ash sample will also be collected after biosolids incineration.

### ***Field Duplicate***

For biosolids sampling, field duplicates are replicate samples collected in the field and submitted to the laboratory as two or more different samples. Biosolids field duplicates will be collected in HDPE containers (250 mL) provided by SGS AXYS on the same day as biosolids samples using identical procedures noted in the section above.

### ***Field Blank***

The field blank is collected by opening a 500 mL container pre-filled PFAS-free water (provided by the SGS-AXYS) while out in the field conducting grab sampling. The field blank is treated the same throughout field and laboratory procedures as collected grab samples.

### ***Equipment Blank***

For biosolids, equipment blanks are collected from a final rinse by passing PFAS-free reagent water (provided by SGS-AXYS) over or through field sampling equipment (i.e., scoops and/or containers used to grab biosolids) before the collection of samples. Each equipment blank should fill up to 500 mL (or 450 mL if the sample will be frozen) in the provided HDPE containers.

## Sample Storage

It is recommended that biosolids samples to be analyzed for PFAS be frozen (below 0°C) as soon as possible. When frozen, the hold time is extended to one year for biosolids. If samples cannot be frozen on site after collection, samples should be shipped immediately to SGS AXYS (see section 7).

## 5. Sampling Sites

See Appendix D for individual POTW processing diagrams and location of sampling sites.

## 6. Sample Labeling

The sample ID system used for the PFAS POTW analytical samples is as follows:

Facility Acronym - Matrix - Identifying Letter - Collection Method - Analysis - Number\*

Where:

Facility Acronym = CCCSD, CSM, EBDA, EBMUD, FSSD, NSD, OSP, PA, SEP, SFOS, SFOI, SJSC, USD, VFWD, VW

Matrix = Influent (INF), Effluent (EFF), Reverse Osmosis Concentrate (ROC), Biosolids (BIO)

Identifying Letter = field sample (F), field sample from different day (F2), field replicate (R), field blank (B), and equipment rinse blank (E), back-up additional samples (A), matrix spike (MS), matrix spike duplicate (MSD)

Collection Method = Grab or Composite (Comp)

Analysis = Target or TOP (Total Oxidizable Precursor); Backups clearly denoted

Number\* = wet ash (01), cake (02), scum (03); (Only for CCCSD biosolids)

Example: SJSC-INF-F-Grab-TOP

Every container will be labeled with a unique sample ID following this system. **SFEI will provide a full list of the sample labels including sample IDs and requested analytical methods from SGS AXYS that will be collected from each facility.** The sample ID will be recorded on the field sampling form.

## 7. Sample Handling and Custody

Chain of custody (COC) records will be maintained throughout the course of the sampling effort. SFEI will provide a pre-filled COC form for each facility listing the expected samples collected and indicate the requested laboratory analysis for each sample. Each participating facility will complete the COC form by filling out any missing information, include the original form with the sample shipment, and provide an electronic copy of the form to SFEI at the time of the shipment.

Samples must be chilled during storage and shipment. It is preferred for samples to be frozen (below 0°C) as soon as possible at the facility until all samples are ready for shipment. Once frozen, hold time is one

year for the biosolids and 90 days for aqueous matrices (influent, effluent, ROC). Otherwise, the samples should be shipped immediately to the analytical laboratory, where they will be frozen when they arrive. The analytical laboratory will analyze and report analytical results within 6 weeks of receiving the last sample, as specified in the contract agreement.

When preparing samples for shipment, it is recommended to double-bag samples (especially influent) using PFAS-free bags. HDPE bags are preferred, though LDPE bags may be used if they do not come into direct contact with the sample media. As much double-bagged wet ice as will fit in the cooler should be used for transporting and shipping liquid and frozen samples. Chemical or blue ice should not be used.

Samples must be shipped by FEDEX priority overnight service on Monday, Tuesday, or Wednesday to avoid any issues with weekend shipping. As this is an international shipment, a commercial invoice (CI) is needed. The CI will be partially completed by SGS AXYS and sent together with the PFAS field sampling kit, which will also include specific facility packaging and shipping instructions (also found in Appendix B). Both SFEI (diana@sfei.org; miguelm@sfei.org) and SGS AXYS (Sean.Campbell@sgs.com) should be included in any FedEx shipment notifications.

## 8. Laboratory Analytical Methods

Aqueous samples and biosolids are analyzed using target PFAS analysis and Total Oxidizable Precursors analysis (TOP; influent and biosolids only). The method information including analytical list, reporting limits, and laboratory QA/QC measures can be directly obtained from SGS AXYS. TOP analysis is used to evaluate the presence of precursors that may not be included in the target analyte list. Biosolid samples will also be analyzed for Percent Solids.

**Table 9** notes the analytes with reporting limits (RLs) included for Target PFAS analysis (MLA-110, SGS AXYS). The RLs are below the reporting requirements specified by the Water Board (State Water Resources Control Board, 2020).

TOP analysis includes the analytes with RLs in **Table 10**; SGS Axys has noted that additional PFAS analytes, including precursors, are currently being added to the analyte list. The concentrations of PFAS precursors are expected to be minimal after the oxidation procedures, and will be confirmed through the analysis of these compounds. TOP samples will be analyzed post-oxidation using base and heat activated persulfate. Laboratory analytical methods are specified in MLA-111 (SGS AXYS). Aqueous samples will be reported in units of ng/L; biosolid samples will be reported in units of ng/g dry weight, and percent solids content (%).

Analytical SOPs will be requested from the laboratory and stored at SFEI, but will not be released to external parties without prior consent of the laboratory.

**Table 9: Target PFAS analyte list (MLA-110, SGS AXYS) including reporting limits (RLs) for aqueous and biosolids samples.**

Abbreviation	Geotracker PARLABEL	PFAS Chemical Name (Acid/Conjugate Base)	Aqueous RLs (ng/L)	Biosolids RLs (ng/g dw)
PFBA	PFTBA	Perfluorobutanoic acid (Perfluorobutanoate)	1.6	0.32
PFPeA	PFPA	Perfluoropentanoic acid (Perfluoropentanoate)	0.8	0.16
PFHxA	PFHA	Perfluorohexanoic acid (Perfluorohexanoate)	0.4	0.08
PFHpA	PFHPA	Perfluoroheptanoic acid (Perfluoroheptanoate)	0.4	0.08
PFOA	PFOA	Perfluorooctanoic acid (Perfluorooctanoate)	0.4	0.08
PFNA	PFNA	Perfluorononanoic acid (Perfluorononanoate)	0.4	0.08
PFDA	PFNDCA	Perfluorodecanoic acid (Perfluorodecanoate)	0.4	0.08
PFUnA	PFUNDCA	Perfluoroundecanoic acid (Perfluoroundecanoate)	0.4	0.08
PFDoA	PFDOA	Perfluorododecanoic acid (Perfluorododecanoate)	0.4	0.08
PFTTrDA	PFTRIDA	Perfluorotridecanoic acid (Perfluorotridecanoate)	0.4	0.08
PFTeDA	PFTEDA	Perfluorotetradecanoic acid (Perfluorotetradecanoate)	0.4	0.08
PFBS	PFBSA	Perfluorobutanesulfonic acid (Perfluorobutanesulfonate)	0.4	0.08
PFPeS	PFPEs	Perfluoropentanesulfonic acid (Perfluoropentanesulfonate)	0.4	0.08
PFHxS	PFHXSA	Perfluorohexanesulfonic acid (Perfluorohexanesulfonate)	0.4	0.08
PFHpS	PFHPSA	Perfluoroheptanesulfonic acid (Perfluoroheptanesulfonate)	0.4	0.08
PFOS	PFOS	Perfluorooctanesulfonic acid (Perfluorooctanesulfonate)	0.4	0.08
PFNS	PFNS	Perfluorononanesulfonic acid (Perfluorononanesulfonate)	0.4	0.08
PFDS	PFDSA	Perfluorodecanesulfonic acid (Perfluorodecanesulfonate)	0.4	0.08
PFDoS	-	Perfluorododecanesulfonic acid (Perfluorododecanesulfonate)	0.4	0.08
4:2 FTS	4:2FTS	1H, 1H, 2H, 2H-perfluorohexane sulfonic acid (1H, 1H, 2H, 2H-perfluorohexane sulfonate)	1.6	0.32
6:2 FTS	6:2FTS	1H, 1H, 2H, 2H-perfluorooctane sulfonic acid (1H, 1H, 2H, 2H-perfluorooctane sulfonate)	1.6	0.32
8:2 FTS	8:2FTS	1H, 1H, 2H, 2H-perfluorodecane sulfonic acid (1H, 1H, 2H, 2H-perfluorodecane sulfonate)	1.6	0.32
3:3 FTCA	3:3FTCA	2H, 2H, 3H, 3H-perfluorohexanoic acid (2H, 2H, 3H, 3H-perfluorohexanoate)	1.6	0.32
5:3 FTCA	5:3FTCA	2H, 2H, 3H, 3H-perfluorooctanoic acid (2H, 2H, 3H, 3H-perfluorooctanoate)	10	2
7:3 FTCA	7:3FTCA	2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA, 2H, 2H, 3H, 3H-perfluorodecanoate)	10	2
PFOSA	PFOSA	Perfluorooctanesulfonamide	0.4	0.08
N-MeFOSA	MEFOSA	N-Methylperfluorooctanesulfonamide	0.4	0.08
N-EtFOSA	ETFOSA	N-Ethylperfluorooctanesulfonamide	0.4	0.08
N-MeFOSAA	NMEFOSAA	N-Methylperfluoro-1-octanesulfonamidoacetic acid (N-Methylperfluoro-1-octanesulfonamidoacetate)	0.4	0.08
N-EtFOSAA	NETFOSAA	N-Ethylperfluoro-1-octanesulfonamidoacetic acid (N-Ethylperfluoro-1-octanesulfonamidoacetate)	0.4	0.08
N-MeFOSE	MEFOSE	N-Methylperfluoro-1-octanesulfonamidoethanol	4	0.8
N-EtFOSE	ETFOSE	N-Ethylperfluoro-1-octanesulfonamidoethanol	4	0.8

HFPO-DA (GenX)	HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionic acid (2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionate)	1.6	0.32
ADONA	ADONA	Decafluoro-3H-4,8-dioxanonic acid (Decafluoro-3H-4,8-dioxanoate)	1.6	0.32
NFDHA	NFDHA	Perfluoro-3,6-dioxaheptanoic acid (Perfluoro-3,6-dioxaheptanoate)	0.8	0.16
PFMBA	PFMBA	Perfluoro-3-methoxypropanoic acid (Perfluoro-3-methoxypropanoate)	0.8	0.08
PFMPA	PFMPA	Perfluoro-4-methoxybutanoic acid (Perfluoro-4-methoxybutanoate)	1.6	0.16
9Cl-PF3ONS	9-Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-chlorohexadecafluoro-3-oxanonane-1-sulfonate)	1.6	0.32
11Cl-PF3OUdS	11-Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-chloroeicosafluoro-3-oxaundecane-1-sulfonate)	1.6	0.32
PFEESA	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid (Perfluoro(2-ethoxyethane)sulfonate)	0.4	0.08

**Table 10: Total Oxidizable Precursors (TOP) PFAS analyte list (MLA-111, SGS-AXYS) including reporting limits (RLs) for aqueous samples. SGS AXYS is currently updating the protocol, and the updated protocol is expected to include additional PFAS precursors, which are expected to be minimal after the oxidation procedure.**

Abbreviation	PFAS Chemical Name (Acid/Conjugate Base)	Aqueous RLs (ng/L)	
PFBA	Perfluorobutanoic acid (Perfluorobutanoate)	6-32  For Perfluorinated Carboxylates C4-C14	
PFPeA	Perfluoropentanoic acid (Perfluoropentanoate)		
PFHxA	Perfluorohexanoic acid (Perfluorohexanoate)		
PFHpA	Perfluoroheptanoic acid (Perfluoroheptanoate)		
PFOA	Perfluorooctanoic acid (Perfluorooctanoate)		
PFNA	Perfluorononanoic acid (Perfluorononanoate)		
PFDA	Perfluorodecanoic acid (Perfluorodecanoate)		
PFUnA	Perfluoroundecanoic acid (Perfluoroundecanoate)		
PFDoA	Perfluorododecanoic acid (Perfluorododecanoate)		
PFTTrDA	Perfluorotridecanoic acid (Perfluorotridecanoate)		
PFTTeDA	Perfluorotetradecanoic acid (Perfluorotetradecanoate)		
PFBS	Perfluorobutanesulfonic acid (Perfluorobutanesulfonate)		8  For perfluorinated sulfonates C4-C10, C12
PFPeS	Perfluoropentanesulfonic acid (Perfluoropentanesulfonate)		
PFHxS	Perfluorohexanesulfonic acid (Perfluorohexanesulfonate)		
PFHpS	Perfluoroheptanesulfonic acid (Perfluoroheptanesulfonate)		
PFOS	Perfluorooctanesulfonic acid (Perfluorooctanesulfonate)		
PFNS	Perfluorononanesulfonic acid (Perfluorononanesulfonate)		
PFDS	Perfluorodecanesulfonic acid (Perfluorodecanesulfonate)		
PFDoS	Perfluorododecanesulfonic acid (Perfluorododecanesulfonate)		

## 9. Quality Control Requirements

### Field Quality Control Samples

Field blanks and equipment rinse blanks for each matrix are included in the sampling plan.

The field blank is collected to verify that the sampling environment does not introduce PFAS and cross-contaminate samples during the sampling event. The field blank is collected by opening a 500 mL container pre-filled PFAS-free spring water (provided by SGS-AXYS) while collecting the grab sample.

Additionally, the equipment blank is collected to evaluate potential contamination from equipment used during sampling, including automated samplers used to collect aqueous samples and scoops used to collect biosolids. The field blank and equipment rinse blank are treated the same throughout field and laboratory procedures as other field samples.

The field blank and equipment blank will be analyzed using target PFAS analysis (MLA-110), which has lower detection limits compared to the TOP analytical method (MLA-111).

The number of field blanks and field duplicates in this study exceed the minimum outlined in the RMP QAPP, which is a minimum frequency of one per 20 samples to evaluate variability including performance of the sampling system and methodology. Field blanks and field duplicates are collected from three different facilities that are meant to represent all participating facilities.

### Laboratory Quality Control Procedures

Laboratory QC measures will comply with QA/QC criteria specified in DoD Table B-15 of Quality Systems Manual (QSM), version 5.3, which is included in Appendix C.

Total Oxidizable Precursor results will be evaluated using the RMP QAPP Quality Assurance Program Plan (QAPP) (Yee et al., 2019). At a minimum the following QC data for TOP analysis will be evaluated:

1. Method/Procedural Blanks: samples of a clean (PFAS-free reagent water) taken through the entire analytical procedure, including preservatives, reagents, and equipment used in preparation and quantitation of analytes in samples; including the field sampling procedures for field blanks.
2. Ongoing Precision and Recovery (OPR) Quality Control Sample: an OPR is prepared by adding an aliquot of native standard containing model precursor compounds and terminal acids (containing C4-C14 carboxylates and C4-C11 and C12 sulfonates) to a clean sample matrix. During the oxidation procedure the model precursors in the OPR react to form terminal perfluorinated acids. Recovery values of the perfluorinated acids spiked plus the predicted reaction products are determined to quantify recovery and used as indication of overall method performance; see the tables below for acceptance ranges. A duplicate sample will be analyzed, with batches containing 7-20 samples. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria tabulated in the laboratory's operating procedures.
3. Surrogate (or internal) Standards: analytes (often isotopes or other substituted analogues of target compounds) introduced to samples to measure and correct for losses and errors introduced during analysis, with recoveries and corrections to reported values generally reported for each sample individually.

4. Matrix Duplicates: field samples to which known amounts of target analytes are added, indicating potential analytical interferences present in field samples and errors or losses in analyses not accounted for by surrogate or internal standard correction.

## 10. Data Management

SFEI will request information about the field sampling parameters from each facility in the field sampling form. SFEI will use the information provided by the facility to fill out the appropriate CEDEN/electronic data format.

SGS AXYS will provide data to SFEI in the appropriate CEDEN/electronic data format templates (as provided by SFEI) within the timeframe stipulated in the contract (6 weeks). SGS AXYS should use the current on-line data checker to review data for vocabulary and business rule violations prior to submitting to SFEI using the SFEI Data Submittal Portal <https://rdcddataupload.sfei.org/> (contact DS@sfei.org for the current login and password). SFEI will work with the laboratory to address vocabulary and business rule issues identified from using the data checker.

SFEI will require data to be corrected and resubmitted if any of the following issues are encountered:

- Data submittal is missing target analytes listed in the contract
- Results not reported in the units and basis requested in the contract
- Field and QC samples not reported in equivalent units and basis for a given analyte.

The QA officer or designee will review the data for quality assurance and quality control and appropriate QA codes are applied to the dataset. The QAO or designee writes a report for each dataset outlining the quality of the data. This report highlights any issues that need to be addressed by the laboratory, project manager, or data management staff.

## 11. Reporting

Each participating facility will be responsible for providing SFEI the facility's GeoTracker Global ID and Field Point Names for each of the sampling sites associated with the study (influent, effluent, biosolids, and/or ROC). Each participating facility will be responsible for creating the Field Point Names in GeoTracker and for uploading a Geo\_XY file with non-surveyed latitude and longitude into GeoTracker. Each facility will be responsible for generating the Geo\_XY file. The Global IDs and Field Point Names should be included on the sampling form, or transmitted to SFEI after the sampling event.

Within 60 days of receiving the final analytical laboratory report, SFEI will upload an Electronic Data Format (EDF) of the analytical results into the Water Board's GeoTracker system on behalf of BACWA and participating POTWs. Field sampling analytical results corresponding to each facility will be uploaded. If appropriate, associated QA/QC samples, such as field blanks and field duplicates, which may have been collected from another facility, will be included. Only target PFAS analytical results for influent, effluent, and biosolids will be uploaded to GeoTracker. (TOP analytical results will not be uploaded to avoid misinterpretation of the results. ROC results will also not be uploaded to GeoTracker. A summary of TOP PFAS analytical results and ROC results will be included in the technical memo submitted to the state and regional Water Boards.)

One monitoring report will be developed from this SAP, and which will include target and TOP results from all participants of this study. Within 90 days of the receipt of the final analytical laboratory report, SFEI will upload a monitoring report via GeoTracker's ESI portal on behalf of each facility. It is expected that each facility will provide SFEI with information about the sampling locations, flow measurements, and flow measurement devices used during sampling in a timely manner. SFEI will compile all reported data (analytical results, QA/QC analyses, any deviations from the SAP reported from each facility, and sampling locations and flow measurements reported by each facility) into one monitoring report.

## 12. Data Validation and Usability

Data quality objectives for field and laboratory measurements evaluate the following:

- Field measurements – sensitivity, precision, accuracy, completeness
- Laboratory chemical analyses – sensitivity, precision, accuracy, completeness, contamination

SFEI staff will examine the data set for completeness (e.g., correct numbers of samples and analyses, appropriate QC sample data included) and accuracy (e.g., in sample IDs). The SFEI QAO or designee will examine submitted target PFAS QA data for conformance with MQOs, specified in DoD Table B-15 of Quality Systems Manual (QSM), version 5.3 (Appendix E). Data that are incomplete, inaccurate, or failing MQOs without appropriate explanation will be referred back to the laboratory for correction or clarification. The QAO will discuss data failing MQOs with laboratory staff to determine whether modifications to analytical methods can be made to improve results on reanalysis. If problems cannot be readily corrected (insufficient sample, irremovable interferences, or blank contamination based on past attempts with the lab), results outside the MQOs may be flagged to alert data users to uncertainties in quantitation. Results will not be censored.

## 13. References

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- Department of Defense (DOD), Department of Energy (DOE), 2019. DOD/DOE Consolidated Quality Systems Manual (QSM) for Environmental Laboratories (DoD Quality Systems Manual Version 5.3).
- Michigan Department of Environmental Quality, 2018. General PFAS Sampling Guidance.
- Rodowa, A.E., Christie, E., Sedlak, J., Peaslee, G.F., Bogdan, D., DiGuseppi, B., Field, J.A., 2020. Field Sampling Materials Unlikely Source of Contamination for Perfluoroalkyl and Polyfluoroalkyl Substances in Field Samples. *Environ. Sci. Technol. Lett.* 7, 156–163. <https://doi.org/10.1021/acs.estlett.0c00036>
- State Water Resources Control Board, 2020. Water Code Sections 13267 and 13383 Order for the determination of the presence of per-and polyfluoroalkyl substances at publicly owned treatment works.
- Yee, D., Franz, A., Wong, A., Ross, J., 2019. Quality Assurance Program Plan for the Regional Monitoring Program for Water Quality in San Francisco Bay (No. Contribution Number 966). San Francisco Estuary Institute, Richmond, CA.

## **Appendix A: Field Sampling Forms**

**BACWA PFAS Study - Phase 1 (2020)**  
**Wastewater Facility Field Sampling Form - Aqueous**

Instructions: Please fill out the following form for each influent, effluent, and/or ROC sampling event. Please email completed form to: [diana@sfei.org](mailto:diana@sfei.org) and [miguelm@sfei.org](mailto:miguelm@sfei.org).

Parameter	Description / examples	Response
Personnel collecting sample	Name	
Facility		
Geotracker Global ID #		
Geotracker Field point name (Influent)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (Effluent)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (ROC)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (Field Blank)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (Field Duplicate)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (Equipment Blank)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Influent flow rate for sampling date	units mgd (peak and daily average flow)	
Effluent flow rate for sampling date	units mgd (peak and daily average flow)	
Influent TSS	measurement from date closest to sampling date	
Effluent TSS	measurement from date closest to sampling date	
Aqueous Sample Storage	e.g. Are samples frozen after collection? If not, note date shipped to SGS AXYS.	
Has your facility accepted landfill leachate?	Indicate yes or no	
Industrial Dischargers	If applicable, indicate known industrial dischargers that <b>ARE</b> represented in the sample(s) collected.	
	If applicable, indicate known industrial dischargers <b>NOT</b> represented in the sample(s) collected.	
Additional Notes	Please note method of collecting duplicate composite samples (e.g. hourly sips into two separate containers, separate autosampler at the same location, or split sample poured from a larger composite container)	
<b>Influent Grab Sample IDs</b>		
Influent grab back-up Sample ID		
Influent collection date and time	e.g. 10/19/20 8:00 AM	
Influent sample collection location	e.g. after grit chamber, in settling tank (note if location is indoors or outdoors)	
Additional influent samples collected	Sample ID(s) for field blanks, field duplicates (include MS and MSD), and equipment blanks.	
Sampling collection equipment	e.g. What sampling equipment (extension rod, pump, tubing) was used that came in contact with the sample (please be specific)? Were recommended equipment cleaning and decontaminating procedures used? If not, were other cleaning procedures used? Please note any modifications.	
<b>Effluent Grab Sample IDs</b>		
Effluent grab back-up Sample ID		
Effluent collection date and time	e.g. 10/19/20 8:00 AM	
Effluent sample collection location	e.g. secondary treatment after dechlorination (note if location is indoors or outdoors and if they are chlorinated)	
Additional effluent samples collected	Sample ID(s) for field blanks, field duplicates (include MS and MSD), and equipment blanks.	
Sampling collection equipment	e.g. What sampling equipment (extension rod, pump, tubing) was used that came in contact with the sample (please be specific)? Were recommended equipment cleaning and decontaminating procedures used? If not, were other cleaning procedures used? Please note any modifications.	
<b>Influent Composite Sample IDs</b>		
Influent composite back-up Sample ID		
Influent composite type	e.g. time-weighted composite, flow-weighted composite, manual composite	
Influent collection start date and time	e.g. 10/19/20 8:00 AM	
Influent collection end date and time	e.g. 10/20/20 8:00 AM	
Influent sample collection location	e.g. after grit chamber, in settling tank (note if location is indoors or outdoors)	

Influent collection method	e.g. automated sampler into composite container, poured into sample container	
Additional influent samples collected	Sample ID(s) for field blanks, field duplicates, and equipment blanks.	
Manual Composite Sampling collection equipment	e.g. What sampling equipment (extension rod, pump, tubing) was used that came in contact with the sample (please be specific)? Were recommended equipment cleaning and decontaminating procedures used? If not, were other cleaning procedures used? Please note any modifications.	
Autosampler information	e.g. Type and brand of autosampler, type of tubing used (note if new). Were tubing and strainer replaced or decontaminated before sample collection? Were recommended equipment cleaning and decontaminating procedures used? If not, were other cleaning procedures used? Please note any modifications.	
<b>Effluent Composite Sample IDs</b>		
Effluent composite back-up Sample ID		
Effluent composite type	e.g. time-weighted composite, flow-weighted composite, manual composite	
Effluent composite start date and time	e.g. 10/19/20 8:00 AM	
Effluent composite end date and time	e.g. 10/20/20 8:00 AM	
Effluent sample collection location	e.g. secondary treatment after dechlorination (note if location is indoors or outdoors and if they are chlorinated)	
Effluent collection method	e.g. automated sampler into composite container, poured into sample container	
Additional effluent samples collected	Sample ID(s) for field blanks, field duplicates, and equipment blanks.	
Manual Composite Sampling collection equipment	e.g. What sampling equipment (extension rod, pump, tubing) was used that came in contact with the sample (please be specific)? Were recommended equipment cleaning and decontaminating procedures used? If not, were other cleaning procedures used? Please note any modifications.	
Autosampler information	e.g. Type and brand of autosampler, type of tubing used (note if new). Were tubing and strainer replaced or decontaminated before sample collection? Were recommended equipment cleaning and decontaminating procedures used? If not, were other cleaning procedures used? Please note any modifications.	
<b>Contact with PFAS containing products</b> Please note if you have handled or come into contact with any of the following in the last 8 hours:		
<b>Personal care products</b>	Hair products	
	Insect Repellants	
	Make-up	
	Perfume/Cologne	
	Moisturizers	
<b>Personal protective equipment and other clothing</b>	Sunblock	
	Brand new clothes	
	Chemically treated clothing (e.g. water/stain/dirt/insect resistance)	
	Recently laundered clothes	
	If applicable, please indicate PFAS containing PPE or other clothing used during the sampling event.	
<b>Additional Notes</b>	Indicate if any other products containing PFAS were used while sampling.	

**BACWA PFAS Study - Phase 1 (2020)**  
**Wastewater Facility Field Sampling Form - Biosolids**

**Instructions:** Please fill out the following form for each biosolids event. Please email completed form to: [diana@sfei.org](mailto:diana@sfei.org) and [miguelm@sfei.org](mailto:miguelm@sfei.org).

Parameter	Description / examples	Response
Personnel collecting sample	Name	
Facility		
Geotracker Global ID #		
Geotracker Field point name (Biosolids)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (Field Blank)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (Field Duplicate)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Geotracker Field point name (Equipment Blank)	sampling point name, geographical coordinates (latitude and longitude) Please update once points are added to geotracker.	
Biosolids Sample Storage	e.g. Are samples frozen? If not, note date shipped to SGS AXYS.	
Has your facility accepted landfill leachate?	Indicate yes or no	
Industrial Dischargers	If applicable, indicate known industrial dischargers that <b>ARE</b> represented in the sample(s) collected.	
	If applicable, indicate known industrial dischargers <b>NOT</b> represented in the sample(s) collected.	
Additional Notes		
<b>Biosolids Sample ID(s)</b>		
Sampling Method	Please note if the sample was collected as a single grab or a composite (indicate the number of samples combined).	
Biosolids collection date and time	e.g. 10/19/20 8:00 AM (indicate all times if a composite)	
Biosolids sample collection location changes	Please clarify if a sampling location is indoors or outdoors. Also note if the location is different from what was indicated in the SAP.	
Biosolids moisture content	% weight of solid per volume of sample	
Additional biosolids samples collected	Sample ID(s) for field blanks, field duplicates (include MS and MSD), and equipment blanks.	
Sampling collection equipment	e.g. What sampling equipment was used that came in contact with the sample? Were recommended equipment cleaning and decontaminating procedures used? If not, were other cleaning procedures used? Please note any modifications.	
<b>Contact with PFAS containing products</b> Please note if you have handled or come into contact with any of the following in the last 8 hours:		
Personal care products	Hair Products	
	Insect Repellants	
	Make-up	
	Perfume/Cologne	
	Moisturizers	
	Sunblock	
Personal protective equipment and other clothing	Brand new clothes	
	Chemically treated clothing (e.g. water/stain/dirt/insect resistance)	
	Recently laundered clothes	
	If applicable, please indicate PFAS containing PPE or other clothing used during the sampling event.	
Additional Notes	Indicate if any other products containing PFAS were used while sampling.	

## **Appendix B: Shipping Instructions**

## PREPARING AND SHIPPING SAMPLES TO SGS AXYS

### LABELING

- Follow procedures described in section 6 of Study of Per- and Polyfluoroalkyl Substances in Bay Area POTWs: Phase 1 Sampling and Analysis Plan.

### CHAIN OF CUSTODY (COC)

- Follow procedures described in section 7 of Study of Per- and Polyfluoroalkyl Substances in Bay Area POTWs: Phase 1 Sampling and Analysis Plan.
- Original copy is placed inside a ziptop plastic bag and placed inside the cooler.

### PACKAGING

1. Freeze all samples before packaging (if possible, also cool shipping container).
2. Place layer of bagged wet ice at bottom of cooler.
3. Place a layer of bubble wrap over the ice
4. Place each sample in a separate zip top bag lay over to of the bubble wrap layer.
5. Place a layer of bubble wrap over the sample containers.
6. Fill all remaining space with bagged wet ice.
7. Place Chain of Custody documents inside the cooler.
8. Close cooler and secure with tape.

### SHIPPING DOCUMENTS

Complete and attach all required shipping documents to the **outside** of container.

- FedEx waybill – See waybill instructions and example waybill below.
- 3 Copies of Commercial Invoice - Sign; date; add waybill number\* - 2 copies included with waybill; 3rd copy to courier

\*FedEx waybill number is the tracking number (top right-hand corner).

### Commercial Invoice

Fill the remaining sections of the commercial invoice

- Date of exportation
- Shipper/exporter information
- Waybill number (same as FedEx tracking number)
- No. of PKGS = number of coolers
- Qty. = number of samples
- Total value = QTY x \$5
- Total invoice value = total value if one cooler
- Total invoice value = total value x no of PKGS if more than one cooler being shipped

## International Air Waybill Instructions

### Section 1: "From"

Enter Shipper's information as completely as possible

### Section 2: "To"

Enter our address as follows:

Sample Receiving  
SGS AXYS Analytical Services Ltd.  
2045 Mills Road W  
Sidney B.C. V8L 5X2  
Phone: 250-655-5800

### Section 3: "Shipment Information"

Enter total number of packages, total weight, and dimensions. Don't enter any declared value.

- Enter the approximate dimensions in the dimensions section.
- Commodity description can be taken from the commercial invoice. See example for 4 effluent samples below.
- Use Harmonized Sales Code **provided on commercial invoice**.
- Country of Manufacture is the USA.
- Enter the total customs value. Use a nominal value of \$5.00 per bottle.

Note, Canada Export Declaration is not applicable. Leave this section blank.

Example:

Commodity Description / Description de la marchandise <b>DETAIL REQUIRED. PRINT IN ENGLISH. / DÉTAIL REQUIS. EN ANGLAIS SVP.</b>	Harmonized Code Code harmonisé	Country of Manufacture Pays de fabrication	Value for Customs Valeur déclarée à la douane
<b>Effluent Water Samples for Scientific Testing</b>	<b>3825.20</b>	<b>USA</b>	<b>\$20.00</b>

<b>Canada Export Declaration/B13A: / Déclaration d'exportation/B13A:</b> <input type="checkbox"/> B13A filed electronically / B13A enregistrée électroniquement <input type="checkbox"/> Manual B13A attached / B13A remplie manuellement et jointe.	<input type="checkbox"/> No B13A required. B13A non exigée. <input type="checkbox"/> B13A Summary Reporting. B13A Rapport sommaire.	Total Declared Value for Carriage Valeur totale déclarée pour le transport N° ID form/ID. aut. _____	Total Declared Value for Customs / Valeur totale déclarée à la douane (Specify Currency) (Préciser la monnaie)
--	--	--	--

**Leave this section blank**

**Section 4: “Express Package Service”**

- Select “FedEx International Priority”,

**Section 5: “Packaging”**

- Select “Other” and write “cooler”.

**Section 6: “Special Handling and Delivery options”**

- Select “Direct Signature”

**Section 7: “Payment”**

Bill transportation charges to:

- Select “Sender” and fill in your FedEx account number.

Bill Customs charges to:

- Select “Recipient”

**Section 8: “Required Signature”**

Sign your name

**SHIPPING DATE**

Please do not ship later than Wednesday afternoon to ensure that if there is delays samples are not sitting over the weekend.

**Should you have any questions/concerns regarding the completion of the shipping documentation or packaging of samples, please contact your dedicated SGS AXYS Project Manager.**

**EXAMPLE FEDEX WAYBILL**

014819486391

014819486391

014819486391

014819486391

014819486391

**International Air Waybill**  
*Lettre de transport aérien internationale*

For FedEx services worldwide. Packages up to 150 lbs. (68 kg), excluding dangerous goods. Not all services and options are available to all destinations. For all services FedEx is International. Certain duties & taxes may apply. A description of all services and options is available on the back of this form. Certains services et options ne sont pas disponibles pour toutes les destinations.

**Sender's Copy**  
*Copie expéditeur*

---

**1 From / Please print and press hard / Expéditeur** *Ecrivez en caractères d'imprimerie et appuyez fermement.*

Date: MM/DD/YY      Sender's FedEx Account Number: \_\_\_\_\_  
SENDER'S FEDEX ACCOUNT NUMBER ONLY / NUMÉRIQUE DU COMPTE FEDEX INTERNATIONAL SOUS ÉCRIT

Sender's Name / Nom de l'expéditeur: \_\_\_\_\_      Phone / Téléphone: \_\_\_\_\_

Company / Nom de la société: \_\_\_\_\_

Address / Adresse: \_\_\_\_\_

Address / Adresse: \_\_\_\_\_      Dept./Floor/Service/Etage: \_\_\_\_\_

City / Ville: \_\_\_\_\_      Province: \_\_\_\_\_      Canada      Postal Code / Code postal: \_\_\_\_\_

Email Address / Adresse courriel: \_\_\_\_\_

Internal Billing Reference / Référence pour facturation interne: \_\_\_\_\_  
FIRST 24 CHARACTERS WILL APPEAR ON INVOICE / LES 24 PREMIERS CARACTÈRES APPARAÎTRONT SUR LA FACTURE

FedEx Tracking Number / Numéro de suivi de FedEx: **8148 1948 6391**      Form ID No. / N° du formulaire: **0412**

**4 Express Package Service / Service colis express**

**NOTE: Service order has changed. Please select carefully.**  
*NOTE: L'ordre des services a changé. Veuillez sélectionner avec attention.*

FedEx International First       FedEx International Priority       FedEx International Economy

**5 Packaging / Emballage**

FedEx Envelope / Enveloppe FedEx       FedEx Pak / Pak FedEx       FedEx Box / Boîte FedEx       FedEx Tube / Tube FedEx

FedEx 10kg Box / Boîte 10kg FedEx       FedEx 25kg Box / Boîte 25kg FedEx       Other / Autre

**6 Special Handling and Delivery Signature Options / Options de manutention spéciale et de signature de livraison**

HOLD at FedEx Location / RETENIR à la succursale FedEx       SATURDAY Delivery / Livrer le SAMEDI

Direct Signature / Signature directe       Indirect Signature / Signature indirecte

Someone at recipient's address may sign for delivery / Quelqu'un à l'adresse du destinataire peut réceptionner l'envoi. / If no one is available at recipient's address, someone at a neighbouring address may sign for delivery for residential deliveries only / Si personne n'est disponible à l'adresse du destinataire, quelqu'un à une adresse voisine peut réceptionner l'envoi. Livraisons résidentielles seulement.

**7 Payment / Paiement**      Complete payment options for both transportation charges and duties and taxes. / Inscrire les options de paiement pour les frais de transport et les droits et taxes.

**Bill transportation charges to:**      Enter FedEx Acct. No. or Credit Card No. below. / Indiquer ci-dessous le n° de compte FedEx ou de la carte de crédit.

Sender / Expéditeur Acct. No. in Section 1 will be billed / Les frais seront imputés au numéro de compte figurant à la rubrique 1.       Recipient / Destinataire       Third Party / Tierce partie       Credit Card / Carte de crédit       Cash/Cheque / Argent liquide/Chèque

FedEx Acct. No. / N° de compte FedEx: \_\_\_\_\_  
 Credit Card No. / N° de carte de crédit: \_\_\_\_\_  
 Credit Card Exp. Date / Date d'expiration de la carte de crédit: \_\_\_\_\_  
 Credit Card Auth. / Autorisation de carte de crédit: \_\_\_\_\_

**Bill Customs charges to:**      Enter FedEx Acct. No. below / Indiquer le n° de compte FedEx.

Sender / Expéditeur Acct. No. in Section 1 will be billed / Les frais seront imputés au numéro de compte figurant à la rubrique 1.       Recipient / Destinataire       Third Party / Tierce partie

FedEx Acct. No./N° de compte FedEx: \_\_\_\_\_

**8 Required Signature / Signature requise**

Use of this Air Waybill constitutes your agreement to the Conditions of Contract on the back of this Air Waybill, and you represent that this shipment does not contain dangerous goods. Certain international treaties, including the Warsaw or Montreal Convention, may apply to this shipment and limit our liability for damage, loss, or delay, as described in the Conditions of Contract. En utilisant la présente lettre de transport aérien, vous acceptez les conditions du contrat indiquées au verso et vous garantissez que cet envoi ne contient pas de marchandises dangereuses. Certains traités internationaux, y compris la Convention de Varsovie ou de Montréal, peuvent s'appliquer à cet envoi et limiter notre responsabilité pour tout dommage, perte ou retard, tel que précisé dans les Conditions du contrat.

Sender's Signature / Signature de l'expéditeur: \_\_\_\_\_

---

**2 To / Destinataire**       Residential Delivery / Livraison résidentielle

Recipient's Name / Nom du destinataire: \_\_\_\_\_      Phone / Téléphone: \_\_\_\_\_

Company / Nom de la société: \_\_\_\_\_

Address / Adresse: \_\_\_\_\_

Address / Adresse: \_\_\_\_\_      Dept./Floor/Service/Etage: \_\_\_\_\_

City / Ville: \_\_\_\_\_      State / Province/Etat: \_\_\_\_\_

Country / Pays: \_\_\_\_\_      ZIP/Postal Code / Code postal: \_\_\_\_\_

Email Address / Adresse courriel: \_\_\_\_\_

Recipient's Tax ID Number Required for Customs Purposes / N° fiscal du destinataire aux fins de la douane: \_\_\_\_\_  
GST/REGISTRATION NO. OR AQ LOCALLY REQUIRED / TFS/CA/TVA/REGISTRATION NO. SELON LA LOI DU PAYS

**3 Shipment Information / Informations sur l'envoi**

Total Packages / Nombre total de colis: \_\_\_\_\_       lbs.       in.       po

Shipper's Lead and Count/SLAC / Envs. chang. et défilé par l'expéditeur/ÉVVE: \_\_\_\_\_       DIM Pkts /  VOL       L /  W /  H       cm

Commodity Description / Description de la marchandise: \_\_\_\_\_

DETAIL REQUIRED. PRINT IN ENGLISH / DÉTAIL REQUIS. EN ANGLAIS SVP.	Harmonized Code / Code harmonisé	Country of Manufacture / Pays de fabrication	Value for Customs / Valeur déclarée à la douane

Canada Export Declaration/É13A / Déclaration d'exportation/É13A: \_\_\_\_\_

B13A filed electronically / B13A enregistrée électroniquement       No B13A required. / B13A non exigée.

Manual B13A attached / B13A remplie manuellement et jointe       B13A Summary Reporting / B13A Rapport sommaire.

Total Declared Value for Customs / Valeur totale déclarée pour le transport: \_\_\_\_\_

Total Declared Value for Customs / Valeur totale déclarée à la douane (Specify Currency) / (Préciser la monnaie): \_\_\_\_\_

Auth. ID / Form ID / N° ID: \_\_\_\_\_      Form/AG: \_\_\_\_\_

---

For questions or to ship and track packages, visit [fedex.ca](http://fedex.ca). / Visitez [fedex.ca](http://fedex.ca) si vous avez des questions, désirez expédier un colis ou faire le suivi de vos envois.

Or call 1.800.GoFedEx 1.800.463.3339. Terms and conditions of service may vary from country to country. / Ou composez le 1.800.GoFedEx 1.800.463.3339. Les modalités de service peuvent varier selon les pays.

**660**

Freight / Pwt: \_\_\_\_\_      Other / Autre: \_\_\_\_\_      Total: \_\_\_\_\_

PART 15021 • Rev. Date 11/13 • ©1994-2013 FedEx • PRINTED IN U.S.A. RIDA • Non-Negotiable International Air Waybill/Lettre de transport aérien internationale non négociable

RETAIN THIS COPY FOR YOUR RECORDS / CONSERVEZ CET EXEMPLAIRE POUR VOS DOSSIERS.

157821 02/19

## **Appendix C: Supplementary Information Tables**

**Appendix C Table 1. Characteristics of POTWs included in study.**

<b>POTW</b>	<b>Subembayment</b>	<b>Estimated Population</b>	<b>Permitted ADWF (MGD)</b>	<b>2018/2019 Flow to Bay</b>	<b>Secondary Treatment Type</b>	<b>Advanced Secondary/Filtration (Yes/No)</b>	<b>Residential/ Commercial % of Total Received Flows*</b>
<b>CCCSD</b>	Suisun	482,000	53.8	38.6	AS	No	96
<b>CSM</b>	South	150,000	15.7	11.6	AS	No	100
<b>DSRSD</b>	South	146,900	20.2	2.3 (2016)	AS	No	94.3
<b>EBDA</b>	South	-	107.8	65.0	-	-	-
<b>EBMUD</b>	Central	650,000	120	58.0	High Purity Oxygen	No	94
<b>FSSD</b>	Suisun	144,000	23.7	15.4	Oxidation Towers/AS	Yes	75
<b>NSD</b>	San Pablo	60,000	7	4.8	AS	No	95
<b>OSP (SFPUC)</b>	Ocean	250,000	43	0	AS	Yes	99
<b>PA</b>	Lower South	217,000	39	21.9	TF/AS	Yes	90
<b>SEP (SFPUC )</b>	South	580,000	58	55.5	High Purity Oxygen	No	98
<b>SFO-I</b>	South	-	1.2	1.2	AS	No	0
<b>SFO-S</b>	South	-	1.2	1.2	AS	No	100
<b>SJ-SC</b>	Lower South	1,500,000	167	93.8	AS/BNR	Yes	94
<b>USD</b>	South	343,500	22	21.6 (2015)	AS	No	75
<b>VFWD</b>	San Pablo	120,000	9	10.1	TF/Solids Contact	No	97.4

*AS = Advanced Secondary, TF = Trickling Filter, BNR = Biological Nutrient Removal*

*\*Based on responses from each POTW regarding their service population*

Appendix C Table 2. POTW shipping addresses and supplies provided in each PFAS sampling kit.

Facility	Shipping Address	Effluent, Equipment Blanks (500 mL HDPE)	ROC (500 mL HDPE)	Influent (125 mL HDPE)	Influent TOP (60 mL HDPE)	Biosolids (250 mL HDPE)	Field Blank Spring Water (500 mL HDPE)	PFAS-Free Reagent Water (4 L)
CCCS	ATTN: Mary Lou Esparza, Blake Brown Central San District Offices & Treatment Plant 5019 Imhoff Place Martinez, CA 94553	16	-	17	4	6	3	1
CSM	ATTN: Xiongbing Liang City of San Mateo WWTP 2050 Detroit Dr San Mateo, CA 94404	4	-	6	4	3	-	-
DSRSD	DSRSD Laboratory Attn: Connie Sanchez 7399 Johnson Drive Pleasanton, CA 94588	4	-	4	4	3	-	-
EBDA	ATTN: Angie Berumen San Leandro WWTP 3000 Davis St. San Leandro, CA 94577	4	-	-	-	-	-	-
EBMUD	ATTN: Nick Klumpp EBMUD WWTP, Lab MS 59 2020 Wake Ave Oakland, CA 94608	4	-	6	4	4	-	-
FSSD	ATTN: Nicole Van Aken Fairfield Suisun Sewer District WWTP 1010 Chadbourne Rd Fairfield, CA 94534	16	-	17	4	3	3	1
NSD	ATTN: Liz Falejczyk Novato Sanitary District WWTP 500 Davidson Street Novato, CA 94945	4	-	6	4	3	-	-
PA	ATTN: Lab, Samantha Bialorucki City of Palo Alto RWQCP 2501 Embarcadero Way Palo Alto, CA 94303	4	-	6	4	3	-	-
OSP	ATTN: Dolson Kwan, PFAS Study Southeast Treatment Plant, Lab Sample Receiving 750 Phelps St.	4	-	6	4	3	-	-
SEP	San Francisco, CA, 94124	4	-	6	4	3	-	-
SFO-S	ATTN: Brian Kuhn SFO - Mel Leong Treatment Plant 910 Clearwater Drive	5	-	6	4	3	1	1
SFO-I	San Francisco, Ca 94128	21	-	11	4	4	2	2
SJ-SC	ATTN: Payal Sarkar San Jose-Santa Clara RWF 700 Los Esteros Road San Jose, CA 95134	10	-	10	10	-	-	-

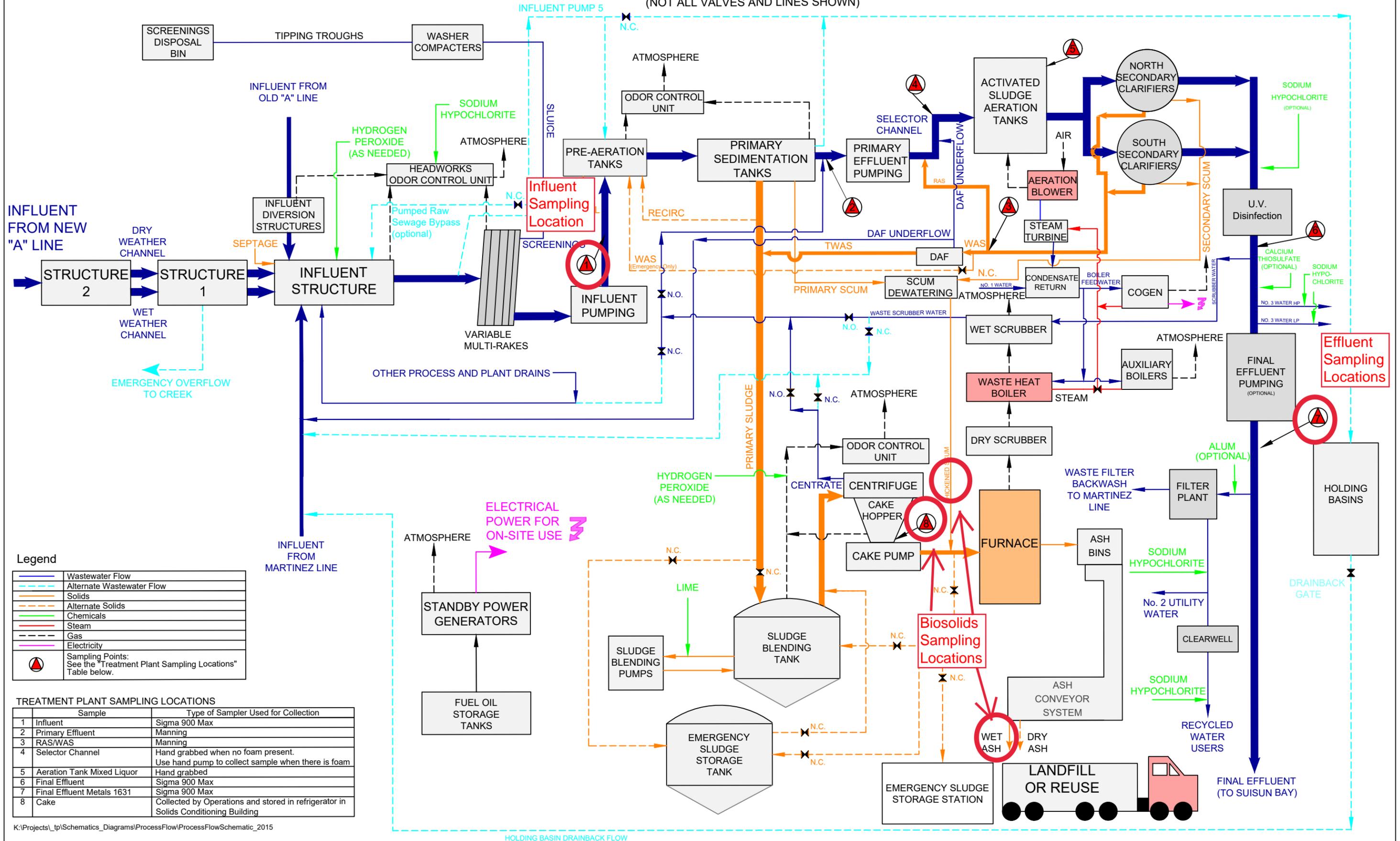
<b>USD</b>	ATTN: Dan Jackson, Laboratory Raymond A. Boege Alvarado WWTP 5072 Benson Rd. Union City, CA 94587	6	-	7	6	4	-	1
<b>VFWD</b>	ATTN: Anita Setty Vallejo Flood & Wastewater District WWTP 450 Ryder St Vallejo, CA 94590	4	-	6	4	3	-	-
<b>VW</b>	ATTN: Jason Chiar Silicon Valley Advanced Water Purification Center 4190 Zanker Road San Jose, CA 95134	-	8	-	-	-	-	-

*Extras have been provided of all sample containers.*

## **Appendix D: POTW Process Diagrams**

# WASTEWATER TREATMENT PROCESS FLOW SCHEMATIC 2020

CENTRAL CONTRA COSTA SANITARY DISTRICT  
(NOT ALL VALVES AND LINES SHOWN)



**Legend**

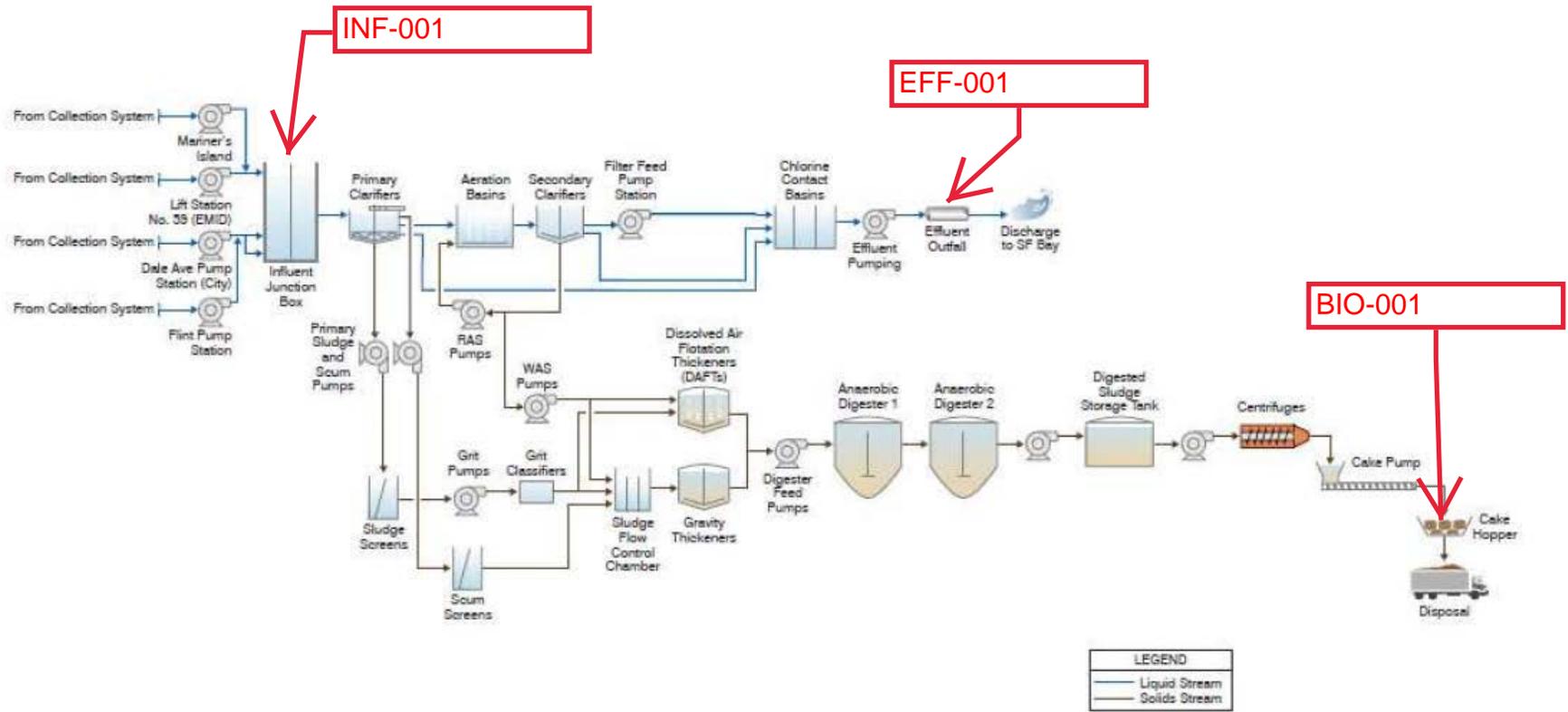
	Wastewater Flow
	Alternate Wastewater Flow
	Solids
	Alternate Solids
	Chemicals
	Steam
	Gas
	Electricity
	Sampling Points: See the "Treatment Plant Sampling Locations" Table below.

**TREATMENT PLANT SAMPLING LOCATIONS**

Sample	Type of Sampler Used for Collection
1 Influent	Sigma 900 Max
2 Primary Effluent	Manning
3 RAS/WAS	Manning
4 Selector Channel	Hand grabbed when no foam present. Use hand pump to collect sample when there is foam
5 Aeration Tank Mixed Liquor	Hand grabbed
6 Final Effluent	Sigma 900 Max
7 Final Effluent Metals 1631	Sigma 900 Max
8 Cake	Collected by Operations and stored in refrigerator in Solids Conditioning Building

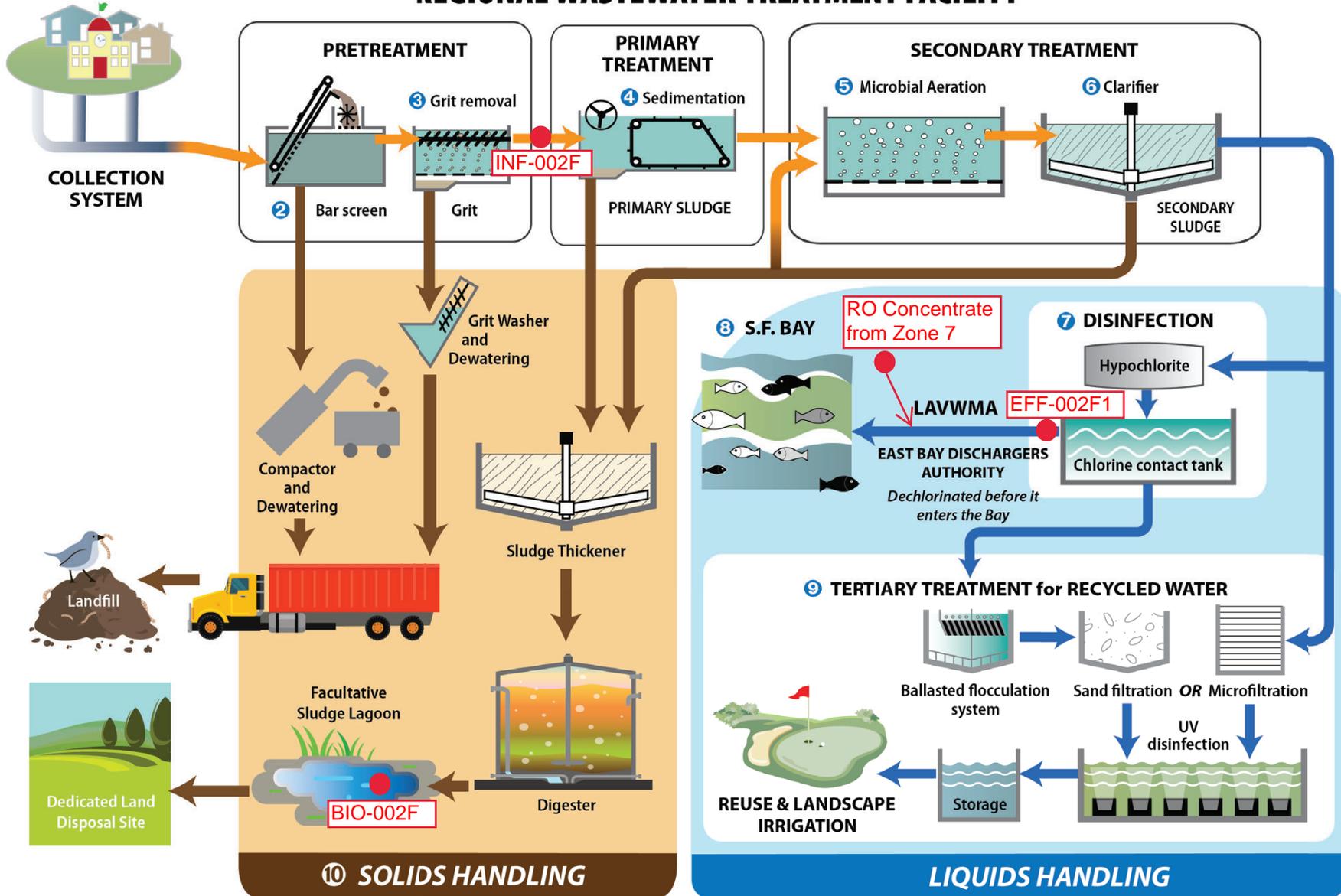
K:\Projects\tp\Schematics\_Diagrams\ProcessFlow\ProcessFlowSchematic\_2015

# City of San Mateo WWTP



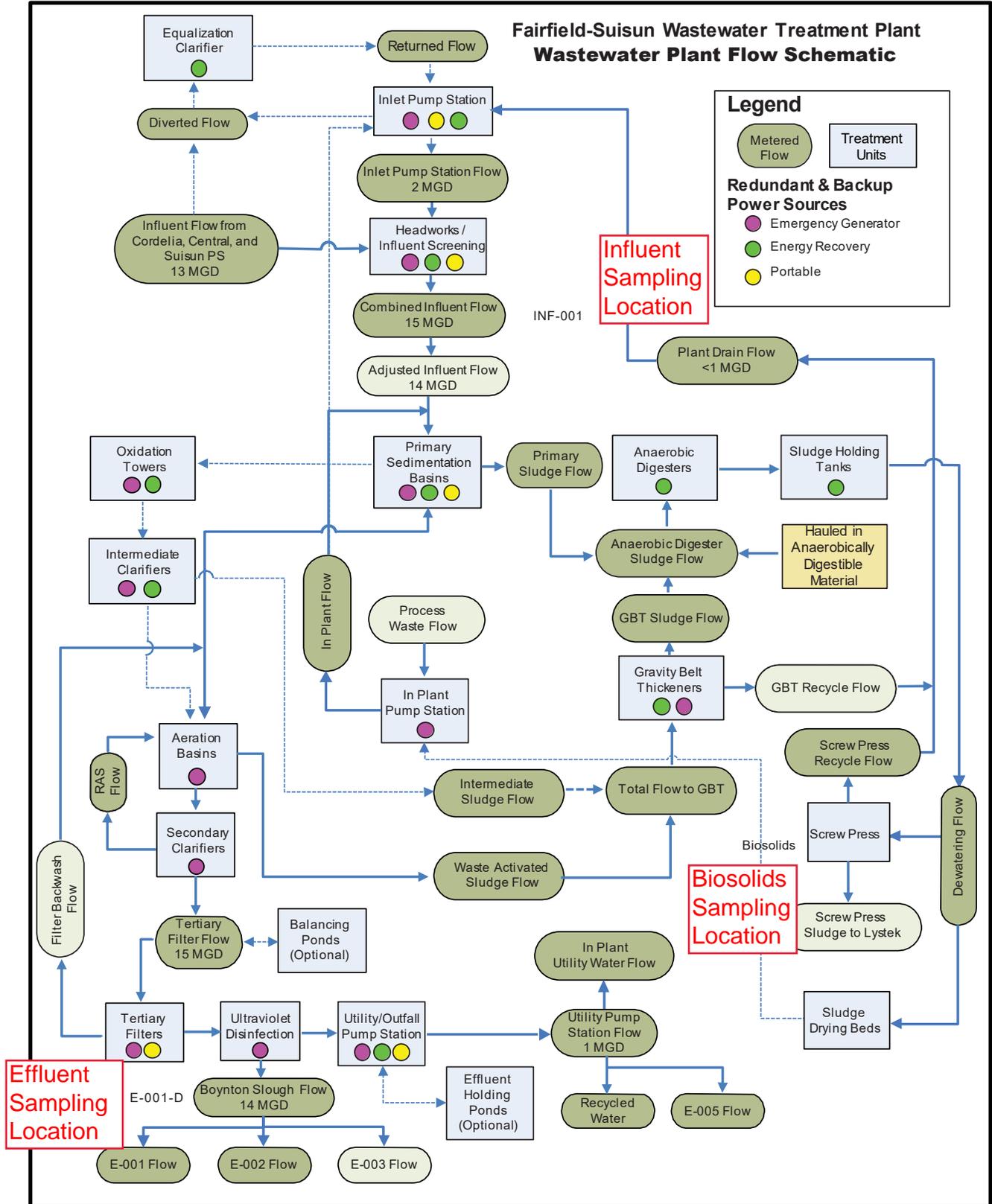
# REGIONAL WASTEWATER TREATMENT FACILITY

Homes and Businesses

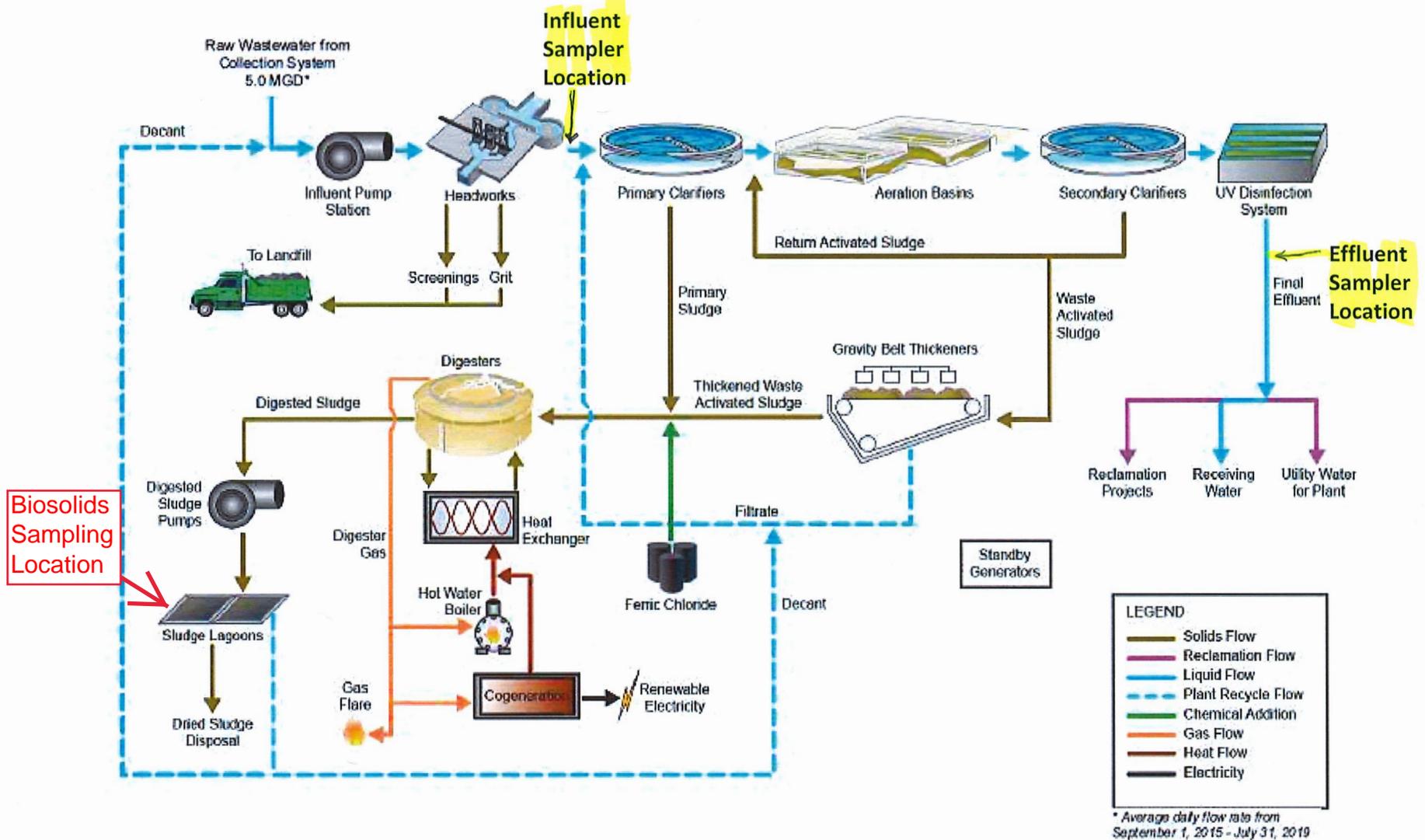




# Fairfield-Suisun Wastewater Treatment Plant Wastewater Plant Flow Schematic

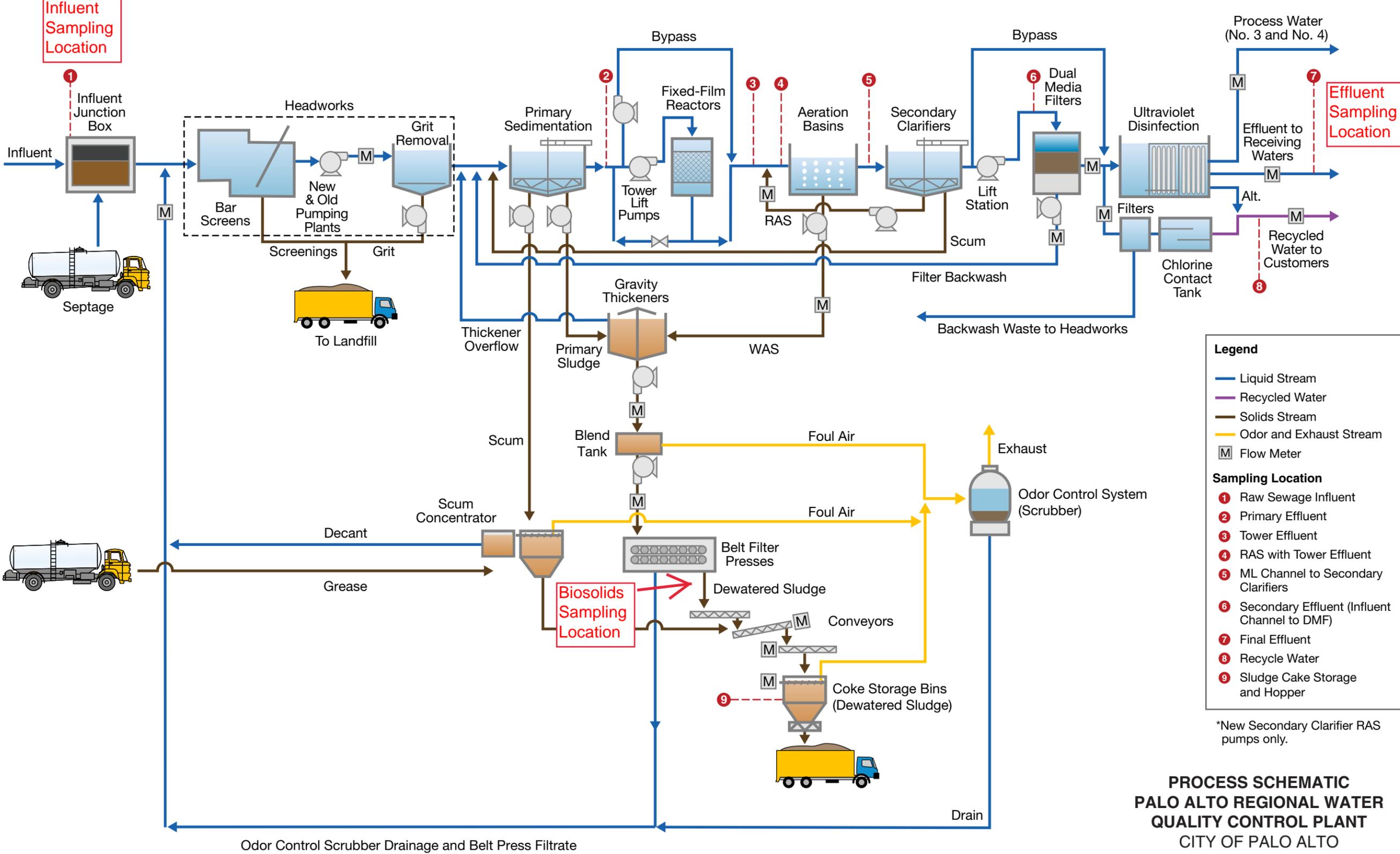


**ATTACHMENT C – PROCESS FLOW DIAGRAM**



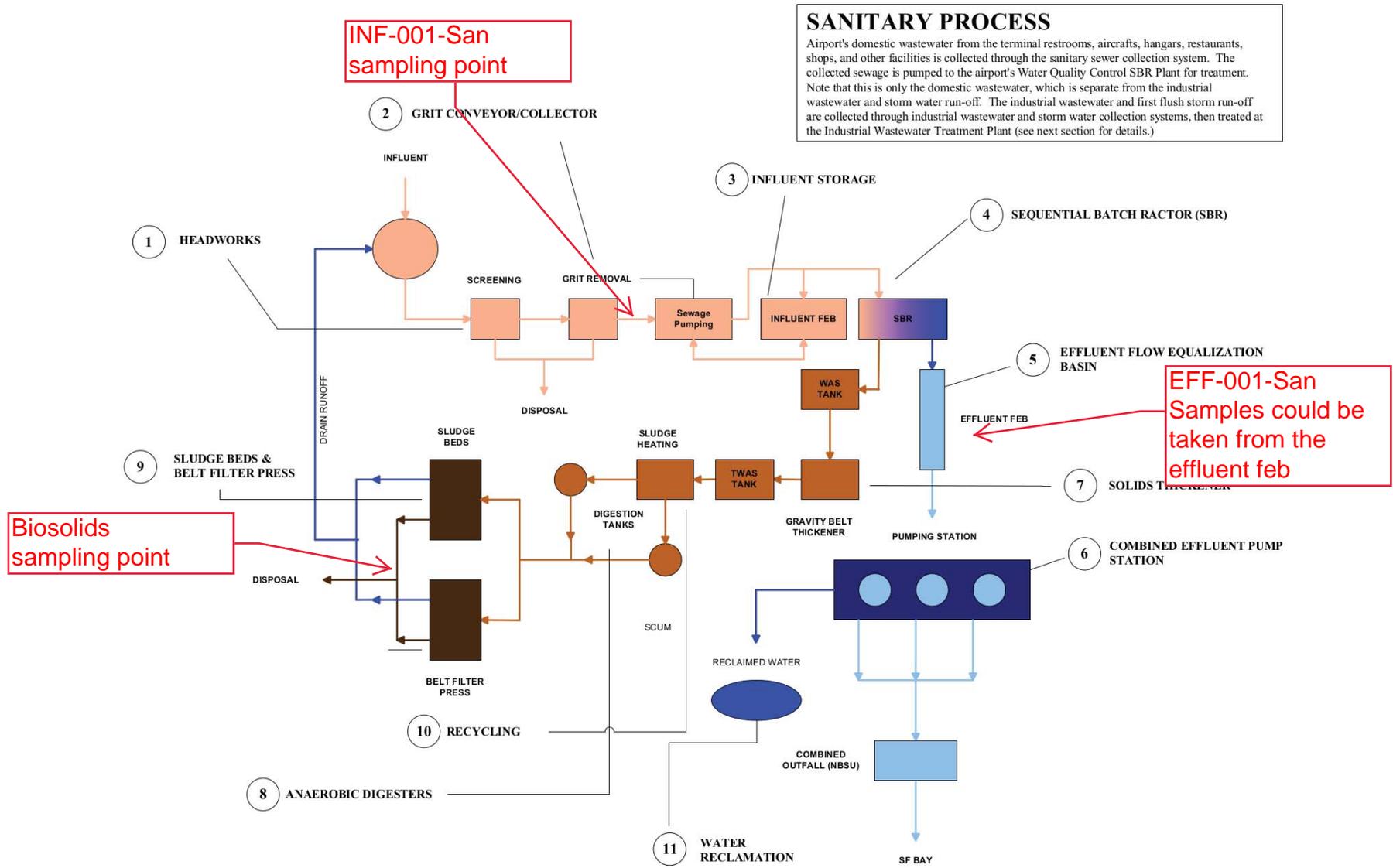


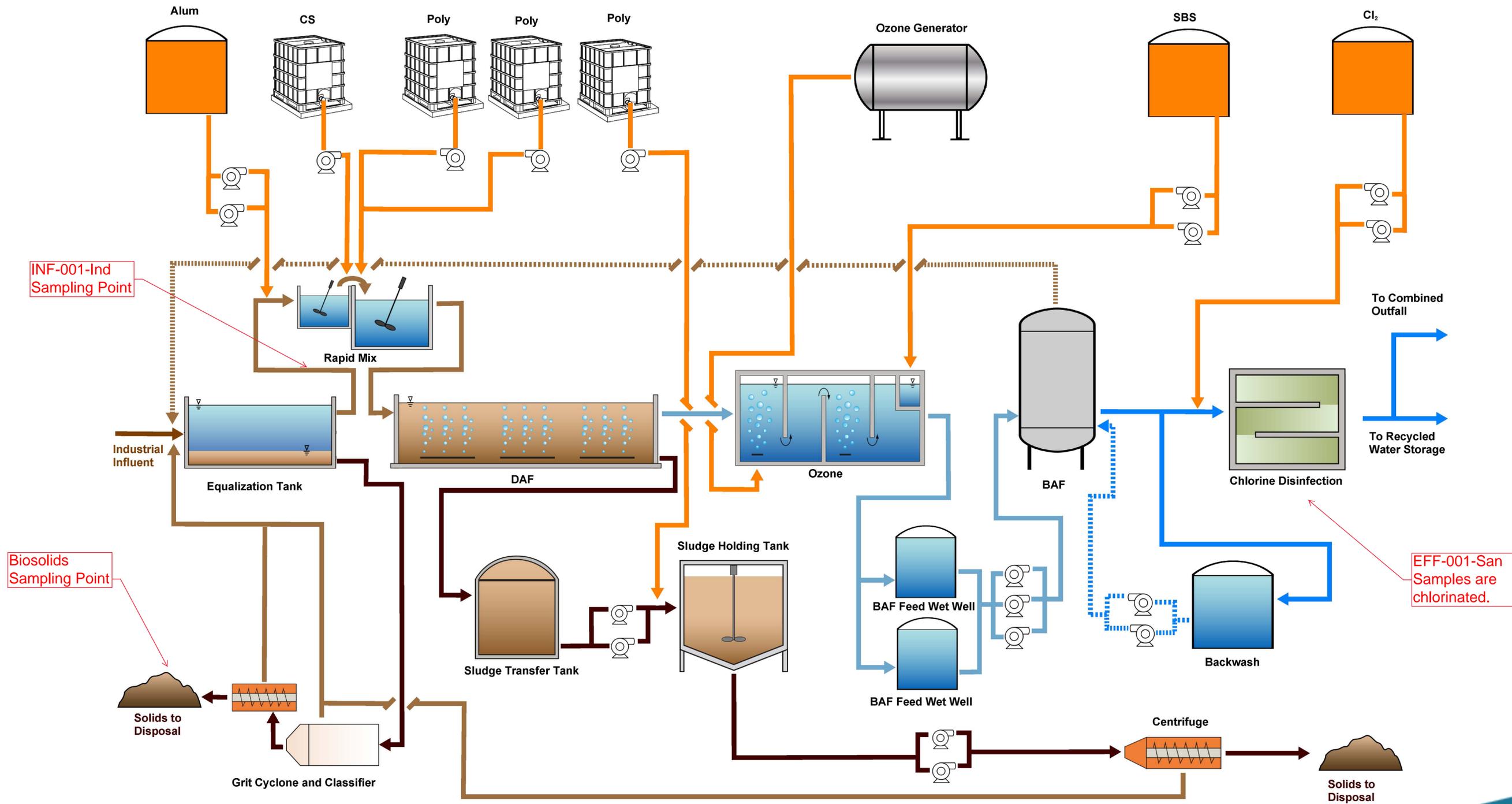
# Palo Alto Regional Water Quality Control Plant Schematic



# SFO Sanitary WWTP

## SANITARY PROCESS FLOW DIAGRAM





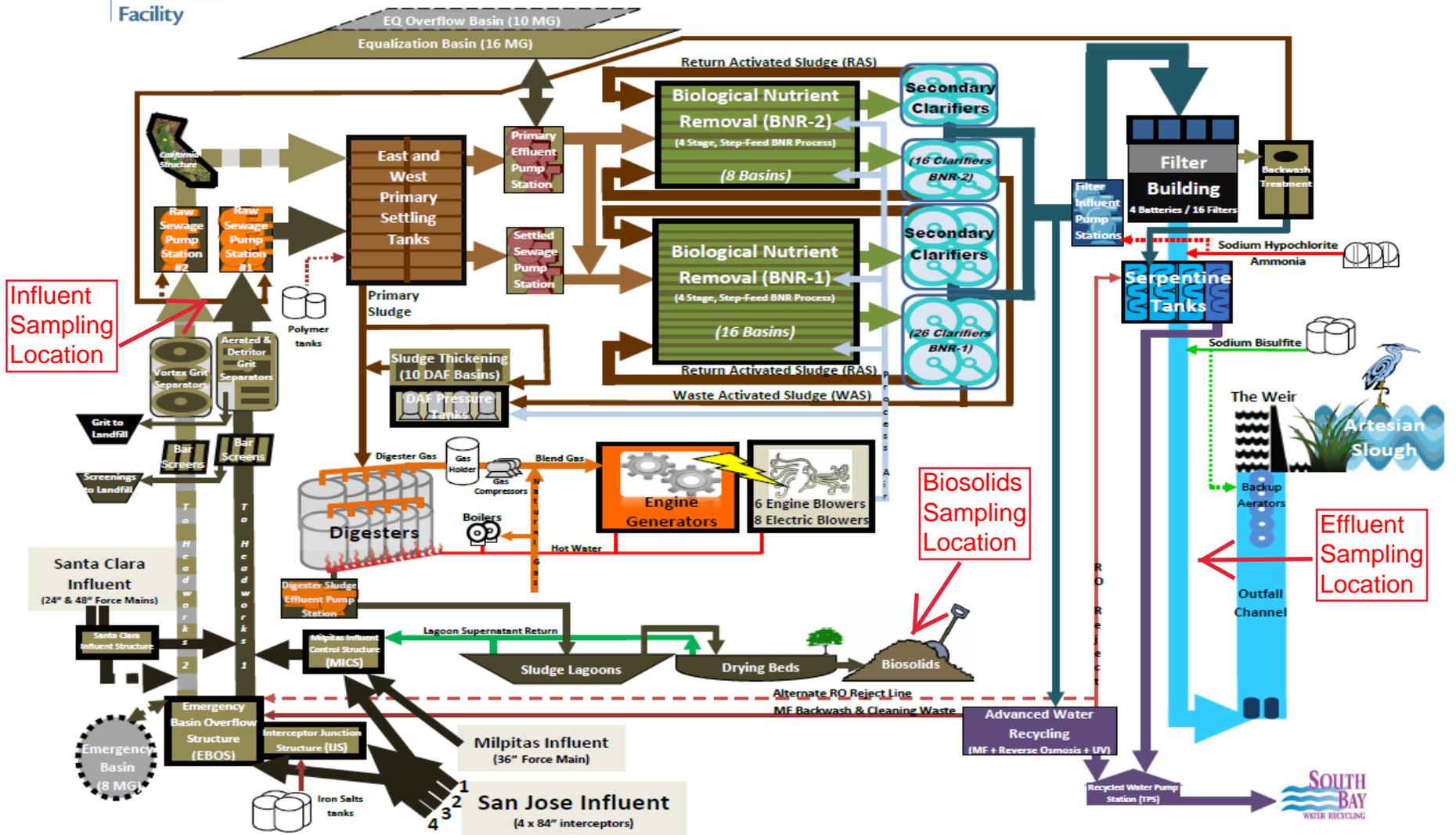
# Mel Leong Industrial Water Treatment Plant at San Francisco International Airport

C.

Revised: 9/2019

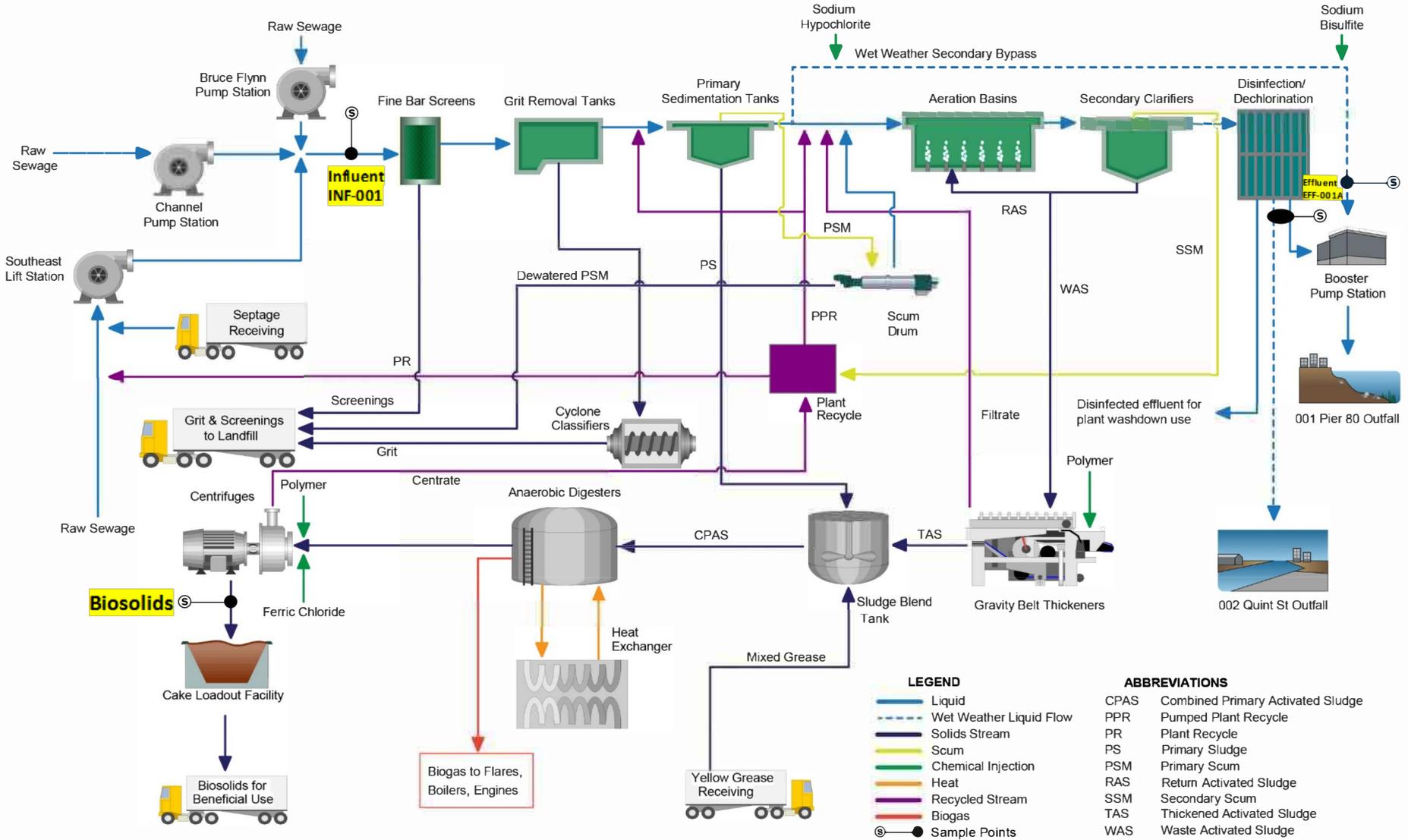


# Process Schematic



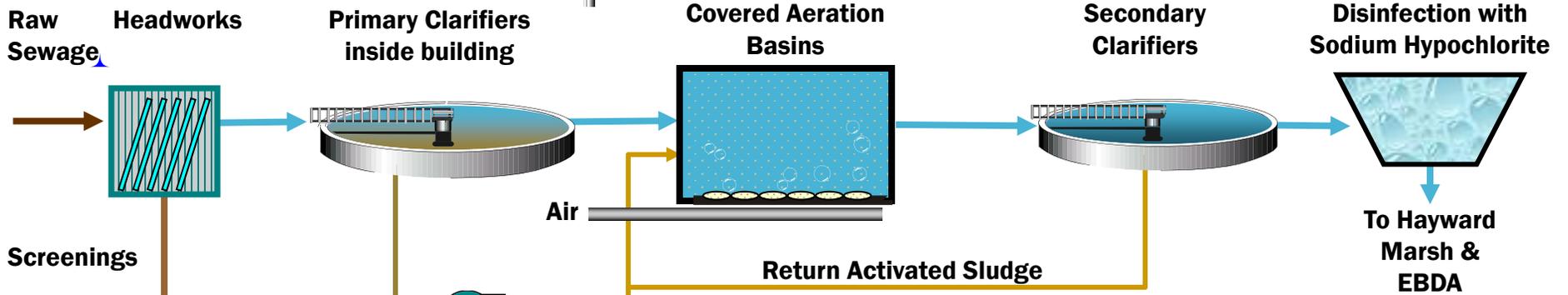
# Plant Process Schematic

## Southeast Water Pollution Control Plant



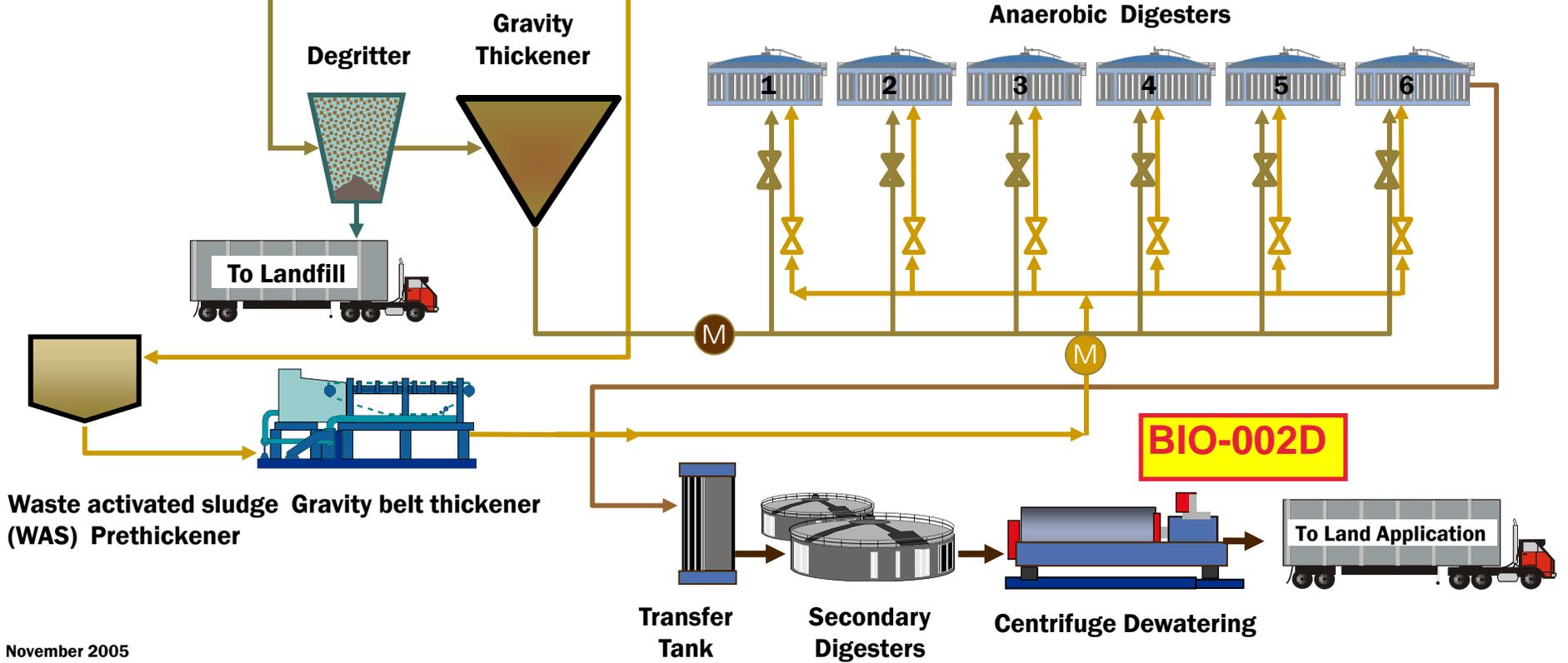
**INF-002D**

# Union Sanitary District



**EFF-002D**

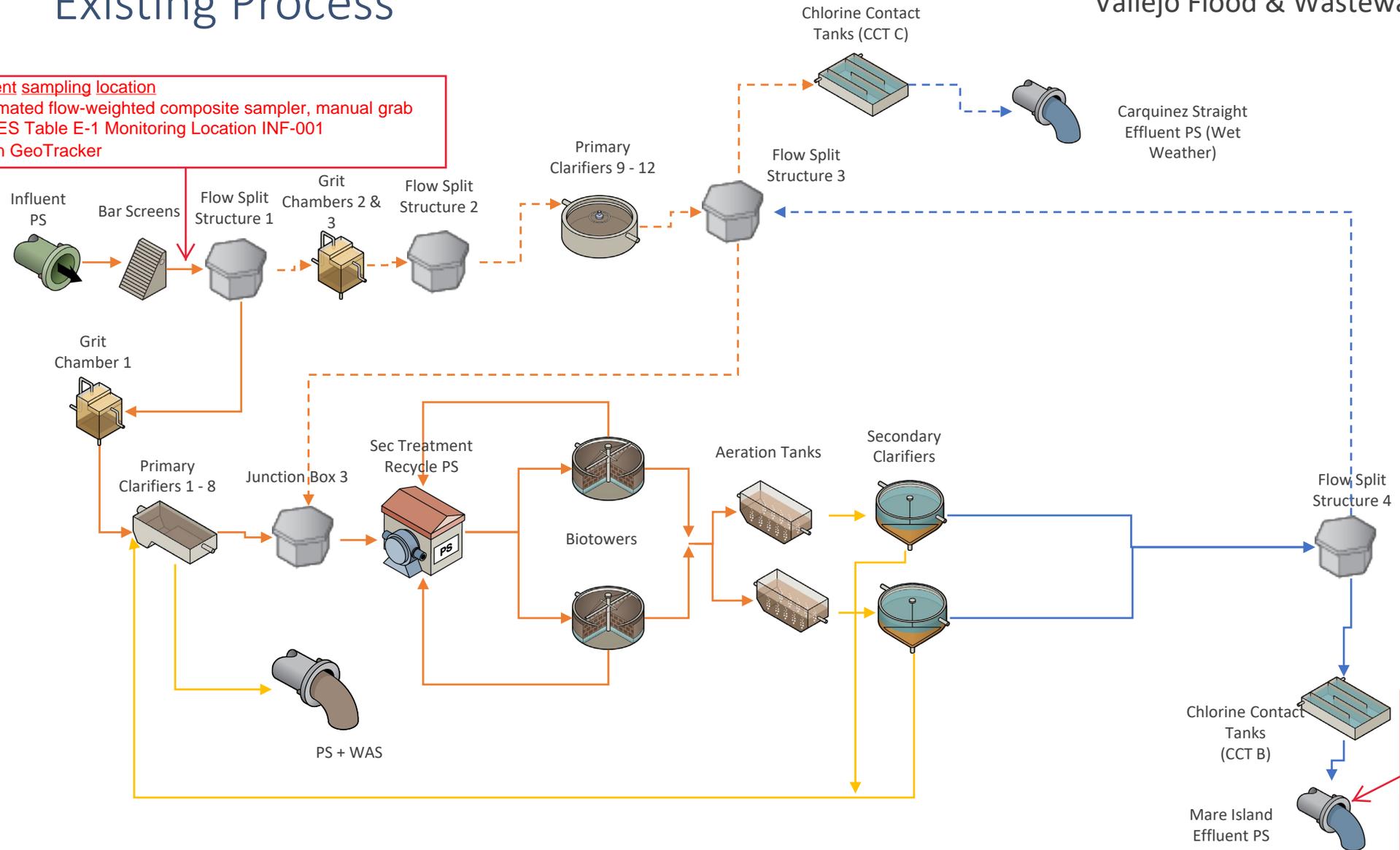
## Union Sanitary District Alvarado Wastewater Treatment Facility Digester Sludge Feeding Detail



# Existing Process

## Vallejo Flood & Wastewater District

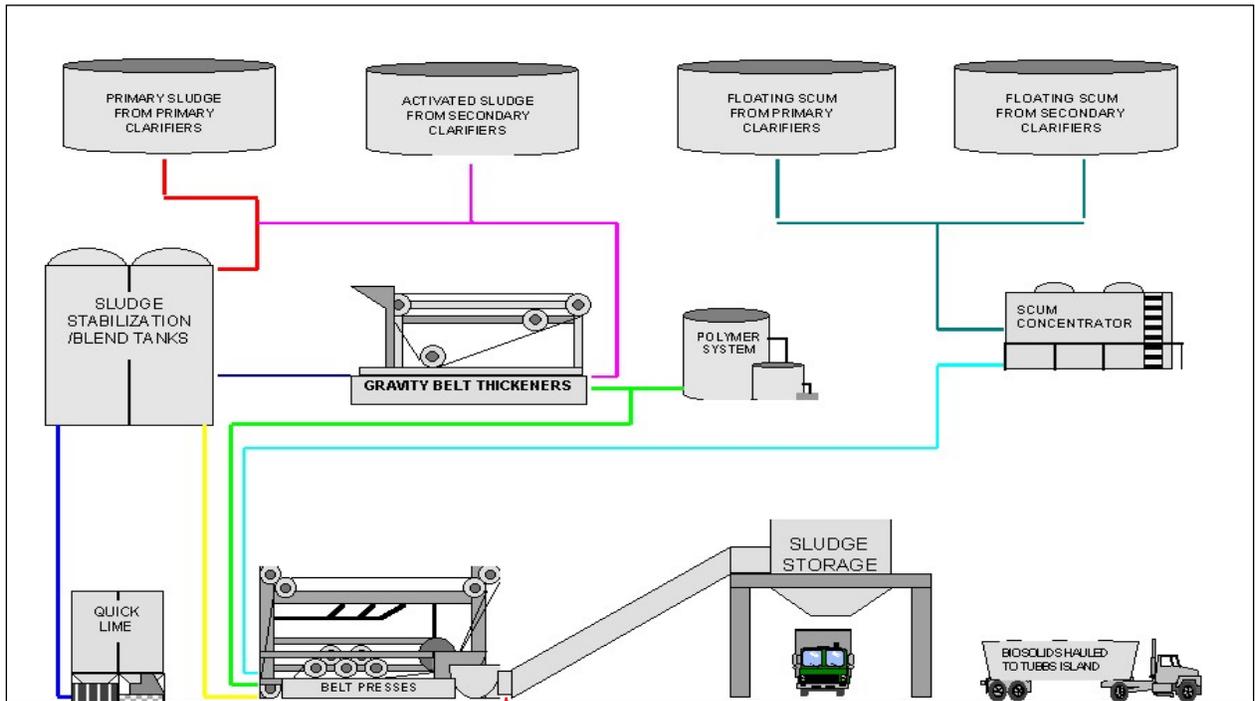
**Influent sampling location**  
Automated flow-weighted composite sampler, manual grab  
NPDES Table E-1 Monitoring Location INF-001  
Not in GeoTracker



**Effluent sampling location**  
(post-dechlorination)  
Automated flow-weighted composite sampler, manual grab  
NPDES Table E-2 Monitoring Location EFF-002  
Not in GeoTracker

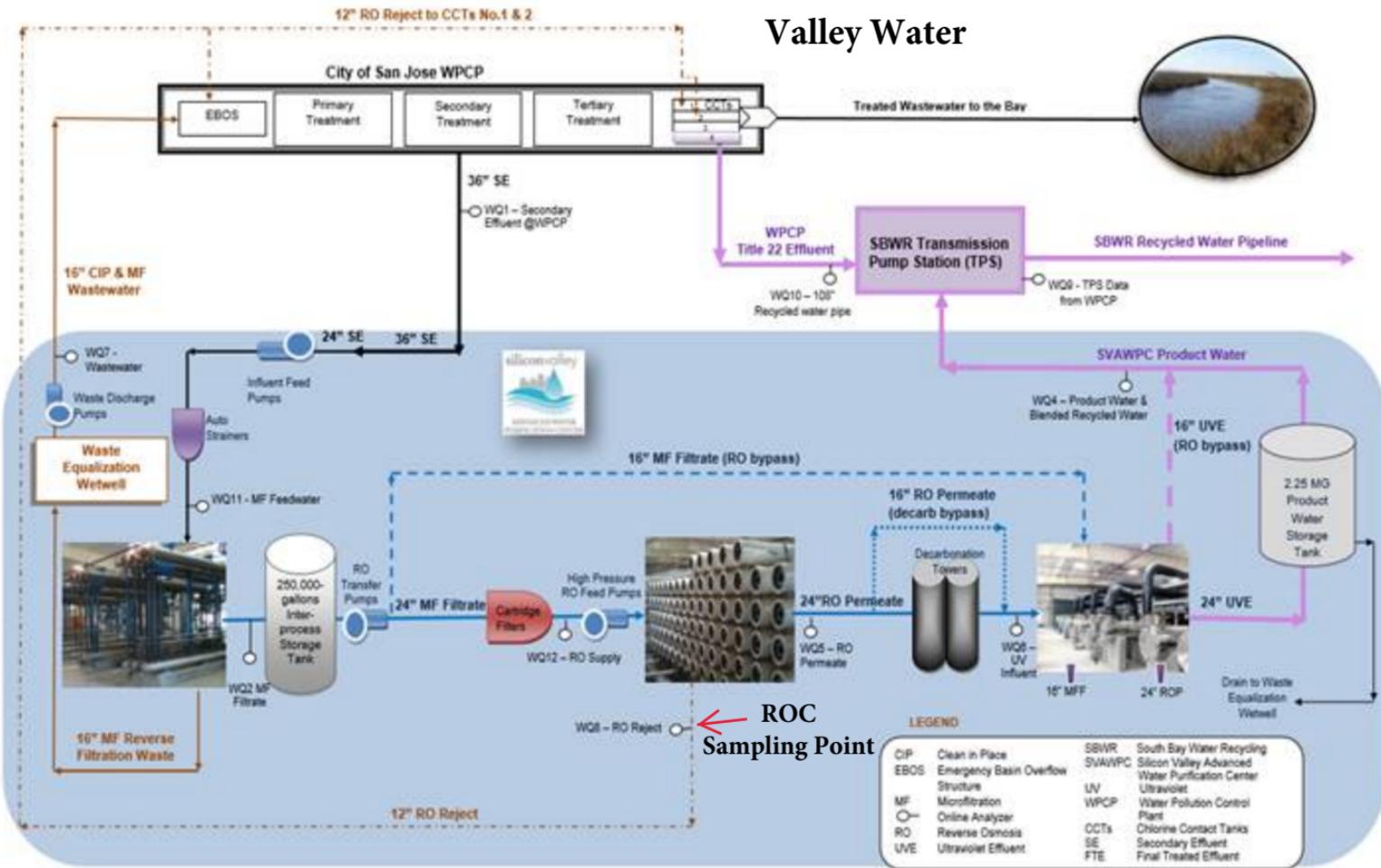
# Vallejo Flood & Wastewater District

**Figure 1**  
**Biosolids Process Flowchart**



Biosolids sampling location  
Grab, manually composited  
NPDES Table E-1 Monitoring Location BIO-001  
Not in GeoTracker

# Valley Water



ROC  
Sampling Point

**LEGEND**

CIP	Clean in Place	SBWR	South Bay Water Recycling
EBOS	Emergency Basin Overflow Structure	SWAWPC	Silicon Valley Advanced Water Purification Center
MF	Microfiltration	UV	Ultraviolet
RO	Reverse Osmosis	WPCP	Water Pollution Control Plant
UVE	Ultraviolet Effluent	CCTs	Chlorine Contact Tanks
		SE	Secondary Effluent
		FTE	Final Treated Effluent

## **Appendix E: DOD QSM, Version 5.3**

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Aqueous Sample Preparation</b>	Each sample and associated batch QC samples.	<p>Solid Phase Extraction (SPE) must be used unless samples are known to contain high PFAS concentrations (e.g., Aqueous Film Forming Foam (AFFF) formulations). Inline SPE is acceptable.</p> <p>Entire sample plus bottle rinsate must be extracted using SPE.</p> <p>Known high PFAS concentration samples require serial dilution be performed in duplicate.</p> <p>Documented project approval is needed for samples prepared by serial dilution as opposed to SPE.</p>	NA.	NA.	<p>Samples with &gt; 1% solids may require centrifugation prior to SPE extraction.</p> <p>Pre-screening of separate aliquots of aqueous samples is recommended.</p>
<b>Solid Sample Preparation</b>	Each sample and associated batch QC samples.	Entire sample received by the laboratory must be homogenized prior to subsampling.	NA.	NA.	NA.
<b>Biota Sample Preparation</b>	Each sample and associated batch QC samples.	Sample prepared as defined by the project (e.g., whole fish versus filleted fish).	NA.	NA.	NA.

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>AFFF and AFFF Mixture Samples Preparation</b>	Each sample and associated batch QC samples.	Each field sample must be prepared in duplicate (equivalent to matrix duplicate).  Serial dilutions must be performed to achieve the lowest LOQ possible for each analyte.	NA.	NA.	Adsorption onto bottle is negligible compared to sample concentration so subsampling is allowed.  Multiple dilutions will most likely have to be reported in order to achieve the lowest LOQ possible for each analyte.
<b>Sample Cleanup Procedure</b>	Each sample and associated batch QC samples.  Not applicable to AFFF and AFFF Mixture Samples.	ENVI-Carb™ or equivalent must be used on each sample and batch QC sample.	NA.	Flagging is not appropriate.	Cleanup should reduce bias from matrix interferences.
<b>Mass Calibration</b>	Instrument must have a valid mass calibration prior to any sample analysis.  Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).	Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer.  Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run.  Mass calibration must be verified to be $\pm 0.5$ amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.	Flagging is not appropriate.	Problem must be corrected. No samples may be analyzed under a failing mass calibration.  The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses fall outside of the $\pm 0.5$ amu of the true value, major instrument maintenance is performed, or the instrument is moved).

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Mass Spectral Acquisition Rate</b>	Each analyte, Extracted Internal Standard (EIS) Analyte.	A minimum of 10 spectra scans are acquired across each chromatographic peak.	NA.	Flagging is not appropriate.	NA.
<b>Calibration, Calibration Verification, and Spiking Standards</b>	All analytes.	<p>Standards containing both branched and linear isomers must be used when commercially available.</p> <p>PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes.</p> <p>For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard.</p>	NA.	Flagging is not appropriate.	<p>Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte.</p> <p>Technical grade standards cannot be used for quantitative analysis.</p>

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<p><b>Sample PFAS Identification</b></p>	<p>All analytes detected in a sample.</p>	<p>The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFBA and PFPeA).</p> <p>Documentation of the primary and confirmation transitions and the ion ratio is required.</p> <p>In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50-150%.</p> <p>Signal to Noise Ratio (S/N) must be ≥ 10 for all ions used for quantification and must be ≥ 3 for all ions used for confirmation.</p> <p>Quant ion and confirmation ion must be present and must maximize simultaneously (±2 seconds).</p>	<p>NA.</p>	<p>PFAS identified with Ion ratios that fail acceptance criteria must be flagged.</p> <p>Any quantitation ion peak that does not meet the maximization criteria shall be included in the summed integration and the resulting data flagged as “estimated, biased high”.</p>	<p>For example: Ion Ratio = (quant ion abundance/ confirm ion abundance)</p> <p>Calculate the average ratio (A) and standard deviation (SD) using the ICAL standards. An acceptance range of ratio could be within A ±3SD for confirmation of detection.</p>

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Ion Transitions (Precursor-&gt; Product)</b>	Every field sample, standard, blank, and QC sample.	<p>In order to avoid biasing results high due to known interferences for some transitions, the following transitions must be used for the quantification of the following analytes:</p> <p>PFOA: 413 → 369                      PFOS: 499 → 80                      PFHxS: 399 → 80                      PFBS: 299 → 80                      4:2 FTS: 327 → 307                      6:2 FTS: 427 → 407                      8:2 FTS: 527 → 507                      NEtFOSAA: 584 → 419                      NMeFOSAA: 570 → 419</p> <p>If these transitions are not used, the reason must be technically justified and documented (e.g., alternate transition was used due to observed interferences).</p>	NA.	Flagging is not appropriate	NA.

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<p><b>Initial Calibration (ICAL)</b></p>	<p>At instrument set-up and after ICV or CCV failure, prior to sample analysis.</p>	<p>The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation).</p> <p>Commercial PFAS standards available as salts are acceptable providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number.</p> <p>If a labeled analog is not commercially available, the Extracted Internal Standard Analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation)</p> <p>Analytes must be within 70-130% of their true value for each calibration standard.</p> <p><i>(continued next page)</i></p>	<p>Correct problem, then repeat ICAL.</p>	<p>Flagging is not appropriate.</p>	<p>No samples shall be analyzed until ICAL has passed.</p> <p>External Calibration is not allowed for any analyte.</p> <p>Calibration can be linear (minimum of 5 standards) or quadratic (minimum of 6 standards); weighting is allowed.</p>

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Initial Calibration (ICAL)</b> <i>(Continued)</i>		ICAL must meet one of the two options below:  Option 1: The RSD of the RFs for all analytes must be $\leq 20\%$ .  Option 2: Linear or non-linear calibrations must have $r^2 \geq 0.99$ for each analyte.			
<b>Retention Time window position establishment</b>	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed.  On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and EIS.
<b>Retention Time (RT) window width</b>	Every field sample, standard, blank, and QC sample.	RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or, on days when ICAL is performed, from the midpoint standard of the ICAL.  Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs.	Correct problem and reanalyze samples.	NA.	Calculated for each analyte and EIS.

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Instrument Sensitivity Check (ISC)</b>	Prior to analysis and at least once every 12 hours.	Analyte concentrations must be at LOQ; concentrations must be within $\pm 30\%$ of their true values.	Correct problem, rerun ISC. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ISC has met acceptance criteria.  ISC can serve as the initial daily CCV.
<b>Initial Calibration Verification (ICV)</b>	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations must be within $\pm 30\%$ of their true value.	Correct problem, rerun ICV. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified.
<b>Continuing Calibration Verification (CCV)</b>	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence.	Concentration of analytes must range from the LOQ to the mid-level calibration concentration.  Analyte concentrations must be within $\pm 30\%$ of their true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV.  Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.  Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs.  Instrument Sensitivity Check (ISC) can serve as a bracketing CCV.

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Instrument Blanks</b>	Immediately following the highest standard analyzed and daily prior to sample analysis.	Concentration of each analyte must be $\leq \frac{1}{2}$ the LOQ.  Instrument Blank must contain EIS to enable quantitation of contamination.	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met.  If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria ( $>1/2$ LOQ), they must be reanalyzed.	Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left.	No samples shall be analyzed until instrument blank has met acceptance criteria.  Note: Successful analysis following the highest standard analyzed determines the highest concentration that carryover does not occur.  When the highest standard analyzed is not part of the calibration curve, it cannot be used to extend out the calibration range, it is used only to document a higher concentration at which carryover still does not occur.

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Extracted Internal Standard (EIS) Analytes</b>	Every field sample, standard, blank, and QC sample.	<p>Added to solid sample prior to extraction. Added to aqueous samples, into the original container, prior to extraction.</p> <p>For aqueous samples prepared by serial dilution instead of SPE, added to final dilution of samples prior to analysis.</p> <p>Extracted Internal Standard Analyte recoveries must be within 50% to 150% of ICAL midpoint standard area or area measured in the initial CCV on days when an ICAL is not performed.</p>	<p>Correct problem. If required, re-extract and reanalyze associated field and QC samples.</p> <p>If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-extracted and analyzed (greater dilution may be needed).</p> <p>Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.</p>	Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in exactly the same manner.	<p>Failing analytes shall be thoroughly documented in the Case Narrative.</p> <p>EIS should be 96% (or greater) purity. When the impurity consists of the unlabeled analyte, the EIS can result in a background artifact in every sample, standard and blank, if the EIS is fortified at excessive concentrations.</p>
<b>Method Blank (MB)</b>	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ LOQ or $> \frac{1}{10^{\text{th}}}$ the amount measured in any sample or $\frac{1}{10^{\text{th}}}$ the regulatory limit, whichever is greater.	<p>Correct problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank.</p> <p>Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.</p> <p>Examine the project-specific requirements. Contact the client as to additional measures to be taken.</p>	<p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.</p>	<p>Results may not be reported without a valid MB.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Laboratory Control Sample (LCS)</b>	One per preparatory batch.	<p>Blank spiked with all analytes at a concentration <math>\geq</math> LOQ and <math>\leq</math> the mid-level calibration concentration.</p> <p>A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p>	<p>Correct problem, then re-extract and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.</p> <p>Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.</p> <p>Examine the project-specific requirements. Contact the client as to additional measures to be taken.</p>	<p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.</p>	<p>Results may not be reported without a valid LCS.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>
<b>Matrix Spike (MS)</b>	One per preparatory batch. Not required for aqueous samples prepared by serial dilution instead of SPE.	<p>Sample spiked with all analytes at a concentration <math>\geq</math> LOQ and <math>\leq</math> the mid-level calibration concentration.</p> <p>A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p>	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

**Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
<b>Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)</b>	<p>For MSD: One per preparatory batch.</p> <p>For MD: Each aqueous sample prepared by serial dilution instead of SPE.</p>	<p>For MSD: Sample spiked with all analytes at a concentration <math>\geq</math> LOQ and <math>\leq</math> the mid-level calibration concentration.</p> <p>A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> <p>RPD <math>\leq</math> 30% (between MS and MSD or sample and MD).</p>	<p>Examine the project-specific requirements. Contact the client as to additional measures to be taken.</p>	<p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.</p>	<p>The data shall be evaluated to determine the source of difference.</p> <p>For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is <math>\geq</math> LOQ.</p> <p>The MD is a second aliquot of the field sample that has been prepared by serial dilution.</p>
<b>Post Spike Sample</b>	<p>Only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of <math>&lt;</math> LOQ for analyte(s).</p>	<p>Spike all analytes reported as <math>&lt;</math> LOQ into the dilution that the result for that analyte is reported from. The spike must be at the LOQ concentration to be reported for this sample as <math>&lt;</math> LOQ.</p> <p>When analyte concentrations are calculated as <math>&lt;</math> LOQ, the post spike for that analyte must recover within 70-130% of its true value.</p>	<p>When analyte concentrations are calculated as <math>&lt;</math> LOQ, and the spike recovery does not meet the acceptance criteria, the sample, sample duplicate, and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.</p>	<p>Flagging is not appropriate.</p>	<p>When analyte concentrations are calculated as <math>&lt;</math> LOQ, results may not be reported without acceptable post spike recoveries.</p>

**Table C-44. Method PFAS by LCMSMS Compliant with QSM Table B-15 Aqueous Matrix**

	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2991-50-6	2-(N-Ethylperfluorooctanesulfonamido) acetic acid	1210	97.9	12.2	61	135
2355-31-9	2-(N-Methylperfluorooctanesulfonamido) acetic acid	1219	100.9	11.7	65	136
757124-72-4	Fluorotelomer sulphonic acid 4:2	789	103.2	13.2	63	143
27619-97-2	Fluorotelomer sulphonic acid 6:2	1673	102.4	12.5	64	140
39108-34-4	Fluorotelomer sulphonic acid 8:2	1657	102.3	11.6	67	138
31506-32-8	N-methylperfluorooctanesulfonamide	404	104.1	12.0	68	141
375-73-5	Perfluorobutanesulfonic acid	1832	100.8	9.4	72	130
375-22-4	Perfluorobutanoic acid	1270	100.6	9.2	73	129
335-77-3	Perfluorodecanesulfonic acid	1361	97.7	14.8	53	142
335-76-2	Perfluorodecanoic acid	1722	100.4	9.5	71	129
307-55-1	Perfluorododecanoic acid	1714	102.8	10.2	72	134
375-92-8	Perfluoroheptanesulfonic acid	1552	101.9	10.7	69	134
375-85-9	Perfluoroheptanoic acid	1837	101.4	9.5	72	130
355-46-4	Perfluorohexanesulfonic acid	1849	99.7	10.3	68	131
307-24-4	Perfluorohexanoic acid	1797	100.4	9.4	72	129
68259-12-1	Perfluorononanesulfonic acid	780	97.7	9.5	69	127
375-95-1	Perfluorononanoic acid	1846	99.9	10.0	69	130
754-91-6	Perfluorooctanesulfonamide	1453	101.9	11.4	67	137
1763-23-1	Perfluorooctanesulfonic acid	1744	102.7	12.4	65	140
335-67-1	Perfluorooctanoic acid	1962	102.2	10.1	71	133
2706-91-4	Perfluoropentanesulfonic acid	812	99.0	9.2	71	127
2706-90-3	Perfluoropentanoic acid	1695	100.7	9.3	72	129
376-06-7	Perfluorotetradecanoic acid	1714	101.8	10.0	71	132
72629-94-8	Perfluorotridecanoic acid	1696	104.8	13.0	65	144
2058-94-8	Perfluoroundecanoic acid	1746	100.8	10.6	69	133

**Table C-45. Method PFAS by LCMSMS Compliant with QSM Table B-15 Solid Matrix**

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
2991-50-6	2-(N-Ethylperfluorooctanesulfonamido) acetic acid	249	99.7	12.9	61	139
2355-31-9	2-(N-Methylperfluorooctanesulfonamido) acetic acid	254	103.9	13.3	63	144
757124-72-4	Fluorotelomer sulphonic acid 4:2	266	103.1	13.7	62	145
27619-97-2	Fluorotelomer sulphonic acid 6:2	575	101.9	12.4	64	140
39108-34-4	Fluorotelomer sulphonic acid 8:2	544	100.8	11.8	65	137
375-73-5	Perfluorobutanesulfonic acid	624	100.5	9.2	72	128
375-22-4	Perfluorobutanoic acid	333	102.9	10.5	71	135
335-77-3	Perfluorodecanesulfonic acid	336	96.2	12.4	59	134
335-76-2	Perfluorodecanoic acid	569	101.0	10.4	69	133
307-55-1	Perfluorododecanoic acid	565	101.7	10.8	69	135
375-92-8	Perfluoroheptanesulfonic acid	511	101.0	10.3	70	132
375-85-9	Perfluoroheptanoic acid	652	101.2	10.0	71	131
355-46-4	Perfluorohexanesulfonic acid	639	98.5	10.5	67	130
307-24-4	Perfluorohexanoic acid	614	100.8	10.2	70	132
68259-12-1	Perfluorononanesulfonic acid	338	96.8	9.1	69	125
375-95-1	Perfluorononanoic acid	650	100.3	9.3	72	129
754-91-6	Perfluorooctanesulfonamide	377	102.2	11.5	67	137
1763-23-1	Perfluorooctanesulfonic acid	518	101.9	11.3	68	136
335-67-1	Perfluorooctanoic acid	663	101.1	10.4	69	133
2706-91-4	Perfluoropentanesulfonic acid	335	97.9	8.1	73	123
2706-90-3	Perfluoropentanoic acid	588	100.2	10.3	69	132
376-06-7	Perfluorotetradecanoic acid	551	101.3	10.5	69	133
72629-94-8	Perfluorotridecanoic acid	548	102.3	12.1	66	139
2058-94-8	Perfluoroundecanoic acid	587	99.9	12.0	64	136