SPOKANE COUNTY
WATER RECLAMATION

Spokane County Regional Water Reclamation Facility
NPDES Permit WA-0093317

FINAL

Quality Assurance Project Plan

Receiving Water Study: Toxic Parameters

October 1, 2012

Quality Assurance Project Plan

Receiving Water Study: Toxic Parameters

Approvals:

Approved by: N Bruce Rawls 10-02-2012

N. Bruce Rawls, Utilities Director, Spokane County Utilities

Date

Approved by: 10/1/12

Dave Moss, Water Reclamation Manager, Spokane County Utilities

Date

Approved by: Rob Lindsay 10/1/12

Rob Lindsay, Water Resources Manager, Spokane County Utilities

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Approved by: _____

Jim Bellatty, Water Quality Section Manager, ERO, WDOE

Date

Approved by: _____

Diana Washington, Permit Unit Supervisor, ERO, WDOE

Date

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Distribution:
Spokane County Water Reclamation
Spokane County Water Resources
Department of Ecology
Brown and Caldwell

1 INTRODUCTION

Spokane County (County) owns the Spokane County Regional Water Reclamation Facility (Facility), which provides treatment for wastewater before discharging to the Spokane River. The Facility is operated by CH2M Hill under contract to the County. A consultant team led by Brown and Caldwell (Consultant), under contract to Spokane County, will provide services for all activities related to sampling and analysis of toxic parameters associated with the Facility, collection system, and industrial pretreatment program.

The Washington State Department of Ecology (Ecology) issued the County a new National Pollutant Discharge Elimination System (NPDES) permit WA-0093317, effective December 1, 2011, for the Facility, which includes a Receiving Water Study (Section S9). The Receiving Water Study (study) comprises three elements: (1) Temperature Monitoring, (2) Conventional Parameters, and (3) Toxic Parameters. This Quality Assurance Project Plan (QAPP) addresses the Toxic Parameters element. The toxic parameters are polychlorinated biphenyls (PCBs), 2,3,7,8-Tetra-Chlorodibenzo-P-Dioxin (2,3,7,8-TCDD), and polybrominated diphenyl ethers (PBDE) and will collectively be identified as toxics in this QAPP. Section S9.C of the permit details the requirements relevant to this QAPP:

> *NPDES PERMIT WA-0093317 Section S9.C*
>
> *For toxic parameters listed in S2, the Permittee must: Conduct analyses of the wastewater facility's influent and effluent samples for PCBs, 2,3,7,8 TCDDs and PBDE at the locations and at the minimum frequencies listed in the schedule in and collected in accordance with protocols, monitoring requirements and QA/QC procedures specified in the Ecology approve quality assurance plan (QAPP). The QAPP shall include the uses of estimated values for source identification and prioritization. The QAPP shall be submitted for Ecology approval* ***by March 15, 2012.***
>
> *A report of the results with attached laboratory data sheets shall be submitted to Ecology (The Annual Toxics Management Report, see S12)*

The purpose of this element of the study is twofold: (1) source identification and prioritization, and (2) monitoring of toxics that are present in the Facility effluent that discharges to the Spokane River. Source identification and prioritization will focus on the collection system that delivers wastewater to the Facility, and will be conducted in steps. The first step is to collect samples from the North Valley Interceptor (NVI) and South Valley Interceptor (SVI). Data collected from the NVI and SVI will guide the selection of sample locations farther upstream in the collection system.

This QAPP addresses sampling and analysis of the influent to the Facility at the SVI and NVI and sampling and analysis of the Facility effluent. Analysis of data from the influent sampling will guide the identification of sample locations farther upstream in the

collection system. Sample locations upstream of the NVI and SVI will be identified in Attachment B to this QAPP when those locations are determined.

The County submitted a draft version of this QAPP on March 15, 2012. Ecology reviewed and commented on the draft version. This QAPP has been revised based on Ecology's comments dated April 2, 2012, participation in the regional PCB Workshop at Gonzaga Law Center on June 5 and 6, 2012, correspondence from Arianne Fernandez of Ecology dated June 21, 2012 regarding establishing regional consistency of sampling, analysis and reporting of PCBs, and a meeting with Ms. Fernandez, Diana Washington, and Lucy Peterschmidt of Ecology on August 7, 2012.

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2 PROJECT ORGANIZATION AND SCHEDULE

Project Organization

Table 1 lists all of the names, addresses, and phone numbers of parties involved in this monitoring program at the time of this QAPP preparation. This QAPP specifies all schedules, methods, procedures, and practices using approved methods. The project staff will use all quality assurance/quality control (QA/QC) policies required by those approved methods.

Table 1
Project Personnel and Areas of Responsibility

Title	Name	Responsibility	Contact Information
County Project Manager	Rob Lindsay, Spokane County	Project Management	509-477-7576 rlindsay@spokanecounty.org
Monitoring Project Manager	Michael Milne, Brown and Caldwell	Consultant team management	206-749-2284 mmilne@brwncald.com
Field Sampling Leader	Adam Klein, Brown and Caldwell	Supervise field monitoring team	206-749-2263 aklein@brwncald.com
Sampler	Jon Rudders, GeoEngineers	Perform sampling	509-363-3125 jrudders@geoengineers.com
Project Chemist	Dr. Khalil Abusaba, Brown and Caldwell	QAPP amendment Data quality review Chemical fingerprinting	925-210-2569 kabusaba@brwncald.com
Support Chemist	Dr. Jaime Sayre, Brown and Caldwell	Assist with QAPP amendment Assist with data quality review	213-271-2300 jsayre@brwncald.com
Contract Laboratory	Georgina Brooks, AXYS Analytical Services Ltd.	Chemical analyses	250-655-5801 gbrooks@axys.com

Project Schedule

Table 2 shows the anticipated schedule for the study. Project staff will record and document schedule changes along with the data collected.

Table 2
Project Schedule

Milestone/Activity	Date/Timeline
1. Submit draft QAPP to Ecology	March 15, 2012
2. Receive initial comments from Ecology	April 2, 2012
3. County to finalize contract with Consultant (BC)	May 10, 2012
4. Attend Toxics Workshop in Spokane	June 5 & 6, 2012
5. Update draft QAPP per initial comments and Workshop	Week of July 2, 2012
6. Meet with Ecology to discuss steps to finalize QAPP	August 7, 2012
7. Finalize influent and effluent sampling locations and document in Attachment B	August 23, 2012
8. Finalize QAPP and submit to Ecology for final approval	August 23, 2012
9. Begin influent/effluent sampling & lab analysis (Per QAPP Sections 5 through 7, and Attachment B)	Within 60 days after QAPP approval
10. Lab data review/validation by Consultant (Per QAPP Sections 7 through 10)	After each round of sampling
11. Evaluate first 3 rounds of influent sampling results	1 month after receipt of analytical results for round 3
12. Update Attachment B to incorporate source tracking locations	1 month after Milestone 11
13. Prepare and Submit Annual Toxics Management Report, including recommendations for QAPP and/or SOP revisions	April 15, 2013 April 15, 2014 April 15, 2015 April 15, 2016 April 15, 2017

3 QUALITY OBJECTIVES

Quality objectives are established for this project to control the degree of total error in data results. These objectives are established to achieve an acceptable level of confidence in decisions made from the collected data. The established objectives include the following:

- Implement procedures for field sampling, sample custody, equipment operation and calibration, laboratory sample analysis, data reduction, and data reporting that will ensure the consistency and thoroughness of data generation
- Assess the quality of data generated to ensure that collected data are scientifically valid, of known and documented quality, and legally defensible, where appropriate
- Ensure that the QAPP and associated project plans are properly implemented
- Document field conditions, sampling, and other activities using appropriate field reports to sufficiently re-create each sampling, analytical, testing, and monitoring event

Analytical precision and bias will be evaluated and controlled by use of laboratory check standards and duplicates.

Precision is a measure of the ability to consistently reproduce results. Precision will be evaluated by analysis of check standards, duplicates, and spikes. Results of laboratory duplicate (split) analyses will be used to estimate laboratory precision.

Bias is the systematic error due to contamination, sample preparation, calibration, or the analytical process. Most sources of bias are minimized by adherence to established protocols for the collection, preservation, transportation, storage, and analysis of samples. Check standards (also known as laboratory control standards) contain a known amount of an analyte and indicate bias due to sample preparation or calibration. Pre-cleaned bottles are "proofed" by the analytical laboratory to quantify background contaminant levels. Rinsate blanks quantify the potential bias that could be introduced as a result of sample collection techniques.

The contract laboratory is expected to meet all QC requirements of the analytical methods being used for this project. Measurement quality objectives (MQOs) are shown in Table 3.

Table 3
Measurement Quality Objectives

Analysis	Laboratory Method	Check Stds./Lab Control Samples (% recovery)	Duplicate Samples (RPD)	Method Detection Limits
PCB congeners	EPA 1668	50–150	≤50	10 pg/L
2,3,7,8-TCDD	EPA 1613B	50–150	≤50	5 pg/L
PBDE	EPA 1614	50–150	≤50	5 pg/L

Comparability

Section S.13 of the permit instructs the County to participate in a cooperative effort to create and participate in the functions of the Spokane Regional Toxics Task Force (SRTTF).

The County recognizes that regional comparability of data among the various permittees will be critical to the success of the regional monitoring effort. We will ensure comparability of study results by using regionally developed Standard Operating Procedures (SOPs) for toxics monitoring. We anticipate that the SOPs will describe the recommended procedures for sample collection, chemical analyses, and data quality review. These SOPs will be a stand-alone document, developed in cooperation with regional SRTTF partners, and will be incorporated into the QAPP by reference. The SOPs will also be included as an attachment in the Annual Toxics Management Report.

The Delaware River Basin Commission (DRBC)[1] protocols have been suggested by Ecology staff as a template for monitoring SOPs by SRTTF members in order to ensure comparability of results. The County proposes to use those protocols as an initial step.

[1] Available at http://www.state.nj.us/drbc/quality/toxics/pcbs/monitoring.html

The laboratory selected to support the County's toxics monitoring program, AXYS Analytical Services, has extensive experience working with the DRBC protocols. Recent discussions with AXYS and Ecology staff have identified several potential refinements to the DRBC protocols to reflect project-specific needs:

- 1-L samples should be adequate for influent and effluent analysis at an initial screening level. Higher volumes can be extracted if initial analyses are below detection limits.
- For upstream source identification, lower cost analyses (e.g., U.S. Environmental Protection Agency [EPA] Method 8082 for PCBs or Method 8270 for PBDEs) may be used for screening purposes.
- The DRBC protocols specify that "Water and sample bottles used in the collection of rinsate blanks shall be supplied by the laboratory which will be performing the analysis. The laboratories shall certify that the bottles and water are PCB free." In this study, AXYS will not be asked to certify that bottles and water are "PCB free"; rather, AXYS will "proof" bottles and blank water by certifying that background PCB levels present were quantified down to the congener-specific detection limits.

As noted above, the County, the Consultant, and AXYS will review the results of the first three rounds of influent sampling and two rounds of effluent sampling in order to (1) identify potential refinements in influent/effluent sampling or analytical procedures, and (2) refine procedures for "trackdown" sampling in the County's collection system, including potential analytical methods for "screening" purposes. We will then revise this QAPP accordingly.

Representativeness

The sampling design was developed to obtain representative data on toxics being discharged to the Spokane River and within the collection system for the Facility. We will ensure representativeness by using appropriate sampling and sample handling procedures, using composite samples, and choosing sample locations and times that best represent the dynamic influent and effluent characteristics.

Completeness

Completeness is defined as the need to collect enough valid data to allow decisions to be made for which the study was designed. The goal for completeness is to collect and analyze 100 percent of the samples described in QAPP.

4 SAMPLING PROCESS DESIGN

Sampling will be conducted as required in the NPDES permit and will include the following constituents:

- Total PCBs
- 2,3,7,8-TCDD
- PBDEs

The purpose of the sampling effort is twofold: (1) source identification and prioritization, and (2) monitoring of toxics that are present in the Facility effluent that discharges to the Spokane River. Sampling locations for source identification and prioritization will be identified and adjusted based on the results of previous sampling events. Sampling locations will be documented in Attachment B to this QAPP and in the Annual Toxics Management Report.

Sampling frequency and locations and the sample type are specified in Section S.2 of the permit. Table 4 presents this information for the wastewater influent, and Table 5 presents the same for the final wastewater effluent.

Table 4
Wastewater Influent Sampling

Constituent	Location Description	Minimum Sampling Frequency	Sample Type
Total PCBs	Each influent trunk line	Bimonthly (6/year)	24-hour time-weighted composite
2,3,7,8-TCDD	Each influent trunk line	Bimonthly (6/year)	24-hour time-weighted composite
PBDE	Each influent trunk line	1 per quarter	24-hour time-weighted composite

Table 5
Wastewater Effluent Sampling

Constituent	Location Description	Minimum Sampling Frequency	Sample Type
Total PCBs	After last treatment process	1 per quarter	24-hour time-weighted composite
2,3,7,8-TCDD	After last treatment process	1 per quarter	24-hour time-weighted composite
PBDE	After last treatment process	1 per quarter	24-hour time-weighted composite

The Facility has two influent trunk lines, the NVI and SVI. Influent trunk line sampling will be conducted at the NVI pump station and the SVI pump station. Effluent sampling will be conducted at an access point to the effluent discharge line located after the last treatment process and prior to exiting the Facility grounds. Figure 1 shows the approximate sampling locations. Attachment B contains more detailed information regarding the influent and effluent sampling locations.

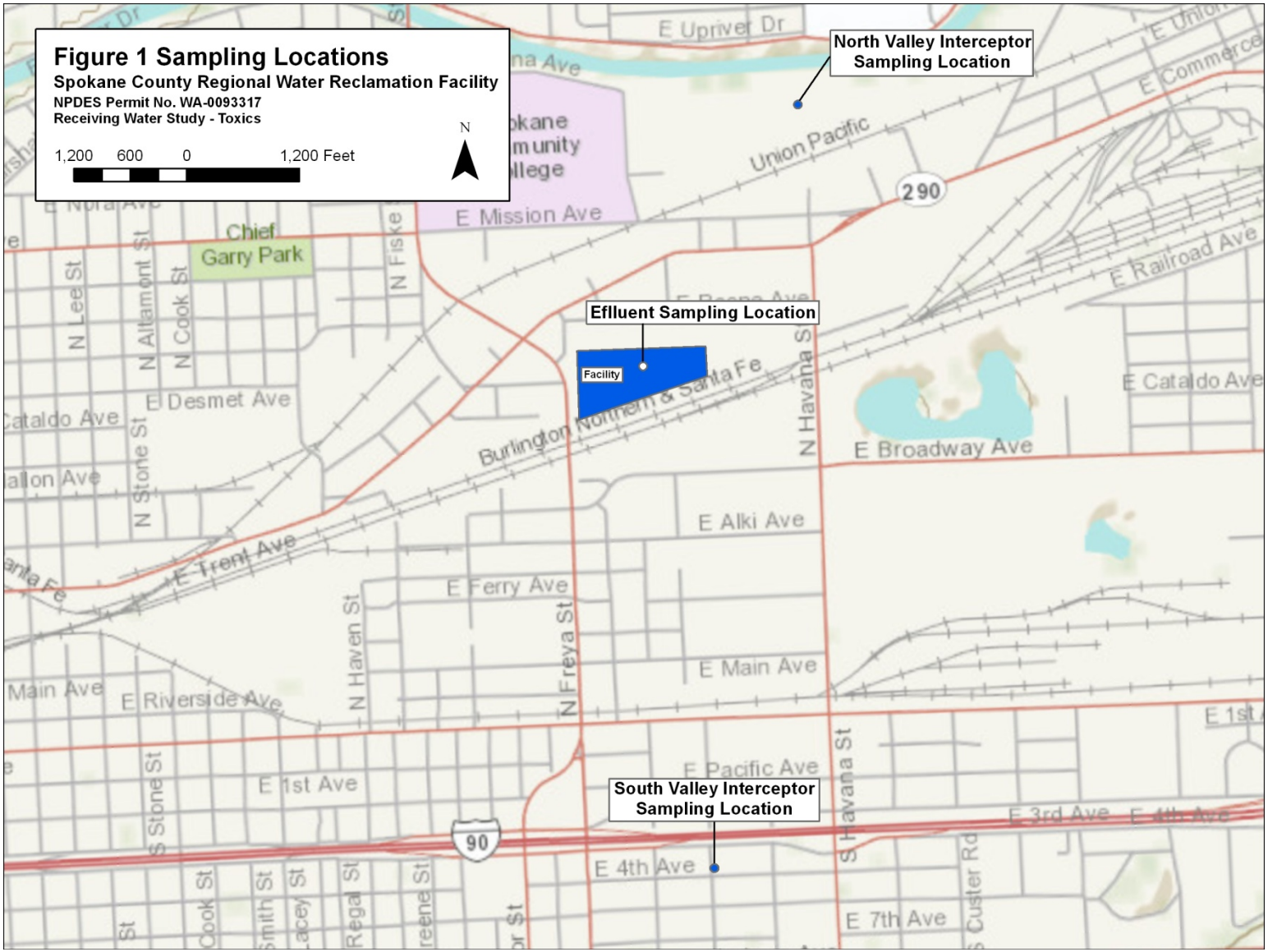
Figure 1 Sampling Locations
Spokane County Regional Water Reclamation Facility
NPDES Permit No. WA-0093317
Receiving Water Study - Toxics
1,200 600 0 1,200 Feet
N
North Valley Interceptor Sampling Location
Efflluent Sampling Location
Facility
South Valley Interceptor Sampling Location
E Upriver Dr
Union Pacific
290
E Mission Ave
Chief Garry Park
E Railroad Ave
E Cataldo Ave
E Broadway Ave
E Desmet Ave
E Alki Ave
E Trent Ave
E Ferry Ave
E Main Ave
E Riverside Ave
E 1st Ave
E Pacific Ave
E 3rd Ave
E 4th Ave
E 7th Ave
90
N Lee St
N Altamont St
N Cook St
N Fiske St
N Stone St
N Haven St
N Freya St
N Havana St
S Stone St
S Havana St
S Custer Rd

5 SAMPLING PROCEDURES

This section provides an overview of the anticipated sampling procedures based on our current understanding. As noted in Section 3 above, the regional SOPs to be developed by the County and SRTTF will provide additional details regarding sampling methods.

Influent Sampling

The Permit requires bimonthly sampling of the NVI and SVI, which are the two influent trunk lines to the Facility. ISCO model 3700 automated samplers will be used to collect 24-hour time-weighted composite samples from the NVI and SVI pump stations.

Composite samples will be collected in one 2.5 gallon (9.5L) glass bottle. From the 2.5 gallon composite sample, the following aliquots will be made and sent to AXYS for analysis: One liter for analysis of PCB congeners by Method 1668

- One liter for analysis of 2,3,7,8-TCDD by Method 1613
- One liter for analysis of PBDEs by Method 1614
- Two to three liters for backup in case the lab needs additional sample water for one or more of the above-listed analyses, or for use as a field duplicate

Effluent Sampling

An automated sampler will be used to collect 24-hour time-weighted composite samples from the final effluent of the Facility. A 2.5 gallon (9.5 L) composite sample will be taken at the sampling location. The following aliquots will be made from the composite sample and sent to AXYS for analysis:

- One liter for analysis of PCB congeners by Method 1668
- One liter for analysis of 2,3,7,8-TCDD by Method 1613
- One liter for analysis of PBDEs by Method 1614
- Two to three liters for backup in case the lab needs additional sample water for one or more of the above-listed analyses, or if a re-analysis is triggered by method blank or rinsate blank evaluation rules, or for use as a field duplicate. The fourth backup sample will not be used until the method blank evaluation rule has been applied.

Collection System Sampling

The County wastewater collection system upstream of the NVI and SVI pump stations will be sampled to help track and prioritize toxics sources. The initial locations and procedures for collection system sampling will be identified based on the results of the first three rounds of influent sampling. Additional sampling locations will be identified based on the collection system sampling results. Sampling locations will be documented in Attachment B to this QAPP and in the Annual Toxics Management Report. Collection system sampling and analyses will be performed in accordance with the regional SOPs

(see Section 3). The County may recommend revisions to the regional SOPs if appropriate based on the influent and/or collection system sampling results.

Equipment Decontamination

Sampling equipment will be cleaned prior to each sampling event. The equipment decontamination procedure is described in Attachment C.

Sample Handling and Custody

Sample Containers

The influent and effluent samples will be collected in pre-cleaned, proofed, 2.5-gallon glass bottles. The composite sample will be transferred to 1-liter amber glass bottles provided by the contract laboratory, AXYS Analytical Services Ltd, and pre-cleaned and proofed. Sample bottles will be stored in clean areas to prevent exposure to contaminants prior to transport to the laboratory for analyses.

Sample Handling and Custody

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analysis and storage, data generation and reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in field and laboratory records.

Field staff shall maintain chain-of-custody records for all field and field QC samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in his/her possession, (2) it is in his/her view, after being in his/her possession, (3) it was in his/her possession and was subsequently locked, or (4) it is in a designated secure area.

The following information concerning the sample shall be documented on the contract laboratory chain of custody (COC) form:

- Sample identification
- Date and time of sample collection
- Source of sample (including name, location, and sample type)
- Preservative used (if any)
- Analyses required
- Name of sample collector(s)
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories

All samples shall be uniquely identified, labeled, and documented in the field at the time of collection. Samples collected in the field shall be transported to the laboratory or field-testing site as expeditiously as possible. When a 4°C requirement for preserving the sample is indicated, the samples shall be packed in ice or chemical refrigerant to keep them cool during collection and transportation. If the temperature of the samples upon receipt exceeds the temperature requirements, the exceedance shall be documented in

laboratory records and communicated to project personnel. The decision regarding the potentially affected samples shall also be documented.

6 MEASUREMENT PROCEDURES

Analytical Methods

Analytical methods are either specified or recommended for the constituents included in the NPDES permit monitoring requirements. The permit provisions related to the analytical portion of this monitoring effort are summarized below.

Requirements

Section S2.A(7)(15): For PCBs use U.S. Environmental Protection Agency (EPA) Method 1668 with a reporting limit or quantitation limit of 10 picograms per liter (pg/L) per congener. For influent monitoring and source tracking a higher limit can be proposed to Ecology in the QAPP if the higher reporting limit still provides adequate source tracking and identification.

Section S2.A(7)(17): For PBDEs use draft EPA Method 1614 with a reporting limit or quantitation limit of 5 pg/L per congener. For influent monitoring and source tracking a higher limit can be proposed to Ecology in the QAPP if the higher reporting limit still provides adequate source tracking and identification.

Recommendations

Appendix A: For 2,3,7,8-Tetra-Chlorodibenzo-P-Dioxin (CAS No. 176-40-16), the recommended analytical protocol is EPA Method 1613B, with a detection limit of 1.3 pg/L and a quantitation level of 5 pg/L.

Table 6 lists the methods to be used to analyze the first three rounds of samples collected from the SCRWRF influent and effluent.

Table 6
Analytical Methods

Constituent	Analytical Protocol	Detection Limit (DL)
PCB congeners	EPA 1668	10 pg/L
PBDEs	EPA 1614	5 pg/L
2,3,7,8-TCDD	EPA 1613	10 pg/L

.

As noted in Section 1, the County and the Consultant will review the results for the first three sampling events to (1) evaluate the need for adjustments in analytical and/or sampling procedures to meet the MQOs for influent and effluent, and (2) develop sampling and analytical procedures for source tracking in the County's wastewater

collection system. We will revise this QAPP and recommend revisions to the regional SOPs as appropriate.

7 QUALITY CONTROL

Field QC Samples

Each sample delivery group will include at least one rinsate blank (a.k.a. "equipment blank") and a travel blank. To create a rinsate blank, ultrapure water provided by the lab will be pumped through a sampling device after it has been cleaned but before it is used for sampling. The rinsate blank will indicate the extent to which contaminants are introduced through the sampling procedure, equipment, or exposure to ambient air during the sample collection. Three rinsate blanks will be collected in the initial sampling event; after the initial sampling event, a rinsate blank will be collected once for each sampling event, rotating to a different location each event.

The travel blank will consist of two 1-liter bottles of ultrapure water for each sampling event that are used to fill a single 1-liter bottle in the field. The travel blank helps distinguish between potential bias introduced by contamination of sample water during collection, shipping, and handling as opposed to contamination from sampling equipment. The fourth, fifth and sixth bottle collected at each location will be used for field duplicates, and / or retained as backup samples in case reanalysis is required. Three field duplicates will be provided for each parameter analyzed over the course of a 12 month sampling period.

Laboratory QC Samples

AXYS will provide analytical services for this sampling effort. AXYS is accredited for the analyses it will perform. Attachment A includes QC requirements for the analyses specified in this QAPP. A Laboratory Quality Assurance Plan is available from AXYS on request.

The data package from AXYS will include a case narrative discussing any problems with the analyses, corrective actions taken, changes to the referenced method, and an explanation of data qualifiers. The data package will also include all associated QC results. This information is needed to evaluate the accuracy of the data and to determine whether the MQOs were met.

8 DATA MANAGEMENT

The Consultant will provide field data and analytical results to Spokane County. Field books will be stored by year in file cabinets located in the Water Resources Section of the Spokane County Public Works Building. Appropriate field data will be entered into an Excel spreadsheet. All entries will be independently verified for accuracy by another individual on the project team.

The laboratory will provide analytical reports in both Portable Document Format (PDF) and as an electronic data deliverable (EDD) in an Excel spreadsheet compatible format. Both the PDF and Excel spreadsheets will be stored on the Spokane County network drives, which are backed up and archived on a regular basis.

After PDF files and Excel spreadsheets are transmitted to Spokane County for archiving, the Consultant will check entries in the Excel spreadsheets against the PDF files to ensure accurate data transposition. Any errors or discrepancies will be noted and corrected. For archiving purposes, original files sent will be appended with the name "- as transmitted" and corrected files will be appended with the name "- as corrected."

Data provided by the laboratory on proofed contaminant concentrations in bottles and blank water will be compiled in a separate Excel workbook, titled "Blank data," along with analytical data on rinsate blanks and travel blanks analyzed. Data provided by the laboratory on spike recoveries, surrogates, and reference materials will be compiled in a separate Excel workbook titled "Accuracy data." Data provided by the laboratory on sample results will be compiled in a separate Excel workbook titled "Sample data." The file name of each of the above three compilation workbooks will include the date the workbook was last updated. Updated workbooks will be provided to Spokane County for archiving within 2 weeks of validating the updates by the Consultant.

9 AUDITS AND REPORTS

Reports

An Annual Toxics Management Report will be completed and submitted on an annual basis. The first report will be submitted by April 15, 2013. The report will include at a minimum the following information:

- Field activities
- Monitoring results and associated laboratory data documentation
- QA/QC procedures
- Detection limits
- PCB fingerprinting results
- Potential sources suggested by data analysis
- Future source identification activities including revised locations and frequency of the influent sampling in the collection system

Assessments and Response Actions

Assessment of Laboratories
AXYS is accredited by Ecology, whose Environmental Laboratory Accreditation Program evaluates a laboratory's quality system, staff, facilities and equipment, test methods, records, and reports. The accreditation establishes the laboratory's capability to provide accurate, defensible data. Results of onsite assessment and proficiency testing studies are available on request. If a laboratory loses its accreditation for any of the analyses required by this sampling effort another lab will be selected.

Assessment of Project Activities
Field audits will include examination of field sampling records; field screening results; field instrument operating records; sample collection, handling, and packaging in compliance with the established procedures; maintenance of QA procedures; chain-of-custody; etc. Follow-up audits will be conducted to correct deficiencies and to verify that QA procedures are maintained throughout the project. The audits will involve review of field measurement records, instrumentation calibration records, and sample documentation. This will occur once during the year.

Reports to Management
Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out-of-limit QC performance that can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. All corrective action proposed and implemented should be documented in the QA sections of project reports. For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. Any nonconformance with the established QC procedures in the QAPP will be identified and corrected in accordance

with the QAPP. The nonconformance and corrective action will be documented in project reports.

10 DATA VALIDATION AND USABILITY

Data Review, Verification, and Validation

AXYS will conduct a review of all laboratory data and provide case narratives. It will verify that methods and protocols specified in this QAPP were followed; that all calibrations, checks on QC, and intermediate calculations were performed for all samples; and that the data are consistent, correct, and complete, with no errors or omissions. Evaluation criteria will include the acceptability of holding times, instrument calibration, laboratory procedural blanks, spike sample analyses, precision data, laboratory control sample analyses, and appropriateness of data qualifiers assigned. A case summary will meet the requirements for a data verification report.

To determine if project MQOs have been met, results for check standards/laboratory control samples, duplicate samples, and labeled compounds will be compared to QC limits. Detection limits reported by the lab will be compared to target detection limits in Section 6 above. Data qualifier flags and the convention for reporting coeluting congeners will follow the regional SOPs, which we expect will prescribe the DRBC template.

The Consultant will evaluate method blank and rinsate blank contamination based on decision rules established by the DRBC. We will and adjust these rules as needed to be consistent with Ecology's toxic source tracking protocols. If the rules trigger a duplicate analysis, the sample will be re-analyzed if a sufficient quantity of duplicate sample is available; otherwise, the sample result will be qualified.

The Consultant will examine the rinsate blank results to quantify the average and standard deviation of analytes in the blanks. To glean as much useful information as possible, the Consultant may "blank-correct" the sample results using the running average and standard deviation of each toxic (by individual congener) in rinsate blanks collected during all sampling events in a given reporting year. Analytical results will be reported both with and without blank correction to help understand the effect of PCBs in the blank on data interpretation.

In trace analysis, blank correction can affect the overall detection limit attained. Blank correction for single analytes and the resulting method detection limit will be calculated based on the procedures outlined in Code of Federal Regulations (CFR) Title 40 Part 136 Appendix B. The water quality criterion for PCBs presents a special challenge, because the criterion is expressed as the sum of 209 congeners. The procedures for estimating method detection limits for blanks outlined in 40 CFR Part 136 Appendix B are specific to single analyte methods. There is no current official EPA guidance on estimating detection limits for blanks or low level samples that are the sum of many analytes. The Consultant proposes to use an approach recently reported in a Low Detection Limit study

conducted for stakeholders in the Marina del Rey watershed, in Los Angeles, California (Los Angeles County Flood Control District, 2011). Details of the procedure are described below.

The average and the standard deviation of each congener are calculated for the blanks. The average is added to two times the standard deviation; then the average plus two standard deviations is subtracted from each congener measurement (uncorrected PCB measurement) in each sample; negative congener results are replaced with zero. The blank-corrected congener measurements in a sample are summed to determine the sum of PCBs. In this approach, the estimated method detection limit (EMDL) is defined as the propagated standard deviations of individual congener measurements (i.e., the square root of the sum of squared standard deviations for congener measurements in replicate blanks). Consistent with the approach in the Marina del Rey study, analytical results will be reported both with and without blank correction, to help understand the effect of PCBs in the blank on data interpretation.

The Consultant will review the laboratory data packages and determine if QC criteria were met, and in cases where they were not met appropriate data qualifiers will be used. The data quality review will be consistent with the regional SOPs. Based on these reviews, the data will be either accepted, accepted with appropriate qualifications, or rejected and re-analysis considered.

Data Usability

Once the data have been reviewed, verified, and validated project staff will determine if the data can be used for project goals such as assessment of toxics concentrations in the effluent that discharges to the Spokane River, and source tracking and identification within the collection system that delivers wastewater to the Facility.

After three rounds of influent samples have been collected and analyzed, the County and the Consultant will evaluate the results to determine whether sampling or analytical procedures should be adjusted to meet the MQOs for influent and effluent sampling. For example, if reporting limits for effluent samples are elevated due to blank contamination or matrix interferences, the County may consider increasing the sample size (e.g., collecting 2- to 4-liter samples rather than 1-liter samples), or using a high-volume sampler to pre-concentrate in the field (see Figure 3). High-volume samplers can be configured to allow for particulate and dissolved fractions to be analyzed separately

Figure 3. Infiltrex 300 sampler uses high-capacity filters and XAD-2 resin columns to extract PCBs, PBDE, and dioxins/furans from large volumes of sample water.

If the initial results indicate that the influent or effluent sampling or analytical procedures need to be adjusted, the County and the Consultant will revise the QAPP accordingly. We may also recommend revisions to the regional SOPs if appropriate. We will submit the revised QAPP for Ecology review and approval prior to adjusting the influent or effluent monitoring procedures.

The analytical results for the first three rounds of influent samples will be reviewed to discern potential differences between the SVI and NVI, as well as variability over time. In addition, PCB congener and homolog patterns in the influent samples will be evaluated to identify potential source materials. These initial results will be used, together with information on sewershed attributes, to identify potential sampling locations to help track sources affecting the County's wastewater collection system.

The initial results may also be used to refine the analytical procedures to improve the efficiency of toxic trackdown efforts. For example, lower cost analyses (e.g., U.S. Environmental Protection Agency [EPA] Method 8082 or for PCBs or Method 8270 for PBDEs) could be found appropriate for screening purposes.

The constituents of concern for the toxics study (PCBs, 2,3,7,8-TCDD, and PBDEs) have very low solubilities in water and strong affinities for organic carbon. Because these constituents are often associated with organic sediments, source tracking could include sampling of bottom sediments and/or suspended solids in the wastewater collection system. Method 8082 may be more appropriate than Method 1668 for analysis of PCBs in bottom sediments or suspended solids prior to implementation of source control measures. Based on review of the initial three rounds of monitoring data, the County and the Consultant will revise this QAPP to specify the locations and procedures for tracking sources of PCBs, 2,3,7,8-TCDD, and PBDEs found in the

wastewater collection system. We may also recommend revisions to regional SOPs if appropriate. We will proceed with the source tracking sampling after Ecology has approved the revised QAPP.

11 REFERENCES

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Attachment A

EPA Method 1613 Quality Control

QC Acceptance Criteria for PCDD/F in CAL/VER, IPR, OPR and Test Samples

	Test Conc. (ng/mL)	IPR		OPR (%)	I-CAL (%)	CAL/VER (%)	Labeled Compound (% Rec. in Sample)		
		SD (%) *	$\bar{X}$ (%)				Warning Limits	Control Limits	EPS 1/RM/19 Limits
Native Compound									
2,3,7,8-TCDD	10	28	83-129	70-130	20	78-125	-	-	-
2,3,7,8-TCDF	10	20	87-137	75-130	20	84-120	-	-	-
1,2,3,7,8-PeCDD	50	15	76-132	70-130	20	78-125	-	-	-
1,2,3,7,8-PeCDF	50	15	86-124	80-130	20	82-120	-	-	-
2,3,4,7,8-PeCDF	50	17.2	72-150	70-130	20	82-122	-	-	-
1,2,3,4,7,8-HxCDD	50	18.8	78-152	70-130	20	78-125	-	-	-
1,2,3,6,7,8-HxCDD	50	15.4	84-124	76-130	20	78-125	-	-	-
1,2,3,7,8,9-HXCDD	50	22.2	74-142	70-130	35	82-122	-	-	-
1,2,3,4,7,8-HxCDF	50	17.4	82-118	72-130	20	90-112	-	-	-
1,2,3,6,7,8-HxCDF	50	13.4	92-120	84-130	20	88-114	-	-	-
1,2,3,7,8,9-HxCDF	50	12.8	84-122	78-130	20	90-112	-	-	-
2,3,4,6,7,8-HxCDF	50	14.8	74-148	70-130	20	88-114	-	-	-
1,2,3,4,6,7,8-HpCDD	50	15.4	76-130	70-130	20	86-116	-	-	-
1,2,3,4,6,7,8-HpCDF	50	12.6	90-112	82-122	20	90-110	-	-	-
1,2,3,4,7,8,9-HpCDF	50	16.2	86-126	78-130	20	86-116	-	-	-
OCDD	100	19	89-127	78-130	20	79-125	-	-	-
OCDF	100	27	74-146	70-130	35	75-125	-	-	-
Surrogate Standards									
$^{13}C_{12}$-2,3,7,8-TCDD	100	37	28-134	25-130	35	82-121	40-120	25-130	40-130
$^{13}C_{12}$-2,3,7,8-TCDF	100	35	31-113	25-130	35	71-130	40-120	24-130	40-130
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	39	27-184	25-150	35	70-130	40-120	25-130	30-130
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	34	27-156	25-130	35	76-130	40-120	24-130	30-130
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	38	16-279	25-130	35	77-130	40-120	21-130	
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	41	29-147	25-130	35	85-117	40-120	32-130	
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	38	34-122	25-130	35	85-118	40-120	28-130	30-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	43	27-152	25-130	35	76-130	40-120	26-130	30-130
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	35	30-122	25-130	35	70-130	40-120	26-123	
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	40	24-157	25-130	35	74-130	40-120	29-130	
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	37	29-136	25-130	35	73-130	40-120	28-130	
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	35	34-129	26-130	35	72-130	40-120	23-130	30-130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	41	32-110	25-130	35	78-129	40-120	28-130	30-130
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	40	28-141	25-130	35	77-129	40-120	26-130	
$^{13}C_{12}$-OCDD	200	47.5	20.5-138	25-130	35	70-130	25-120	17-130	30-130
Cleanup Standard									
$^{37}Cl_4$-2,3,7,8-TCDD	10	36	39-154	31-130	35	79-127	40-120	35-130	

* For comparability with EPA 1613B the precision specification for IPR is stated as %SD (=standard deviation relative to the fortification level)

QC Specifications for QC Samples, Instrumental Analysis, and Analyte Quantification

QC Parameter	Specification
Analysis Duplicate	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL)[1]
Procedural Blank	**Blood/serum/plasma:** TCDD/F <0.2 pg/sample, PeCDD/F <0.5 pg/sample, HxCDD/F and HpCDD/F <1.0 pg/ sample, OCDD/F<5 pg/sample. **Other matrices:** TCDD/F <0.5 pg/sample, PeCDD/F, HxCDD/F, HpCDD/F <1.0 pg/sample, OCDD/F <5 pg/sample. Higher levels acceptable where all sample concentrations are > 10X the blank concentrations.
Detection Limit	SDL Requirements **Blood/serum/plasma:** Tetra-penta-CDD/F 0.2 pg/sample Hexa-octa-CDD/F 0.5 pg/sample **Other matrices**: 0.5 pg/sample
Instrument Carryover and Background: Toluene Blank **Samples**	A. 1st toluene blank following Cal Ver must have <0.6 pg TCDD and <25 pg OCDD[2]. B. 2nd toluene blank following Cal Ver must have <0.2 pg TCDD/F, <0.8 pg Pe-HpCDD/F, and <5.0 pg OCDD[2]. Blood/serum/plasma extract analysis: As many toluene blanks as necessary are run to achieve an instrument blank level of <0.1 pg TCDD/F, <0.3 pg PeCDD/F, <0.5 pg HxCDD/F, <0.5 pg HpCDD/F and <3.5 pg OCDD. <10% contribution from preceding sample (based on observed instrument carryover rate).
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Data may be taken from more than one chromatogram to get the responses in the calibrated range.
Ion Ratios	Must be within ±15% of theoretical. For 1613B applications only an alternate acceptance criteria of within ±10% of the ratio in the midpoint calibration (CS3) or calibration verification (Cal Ver), whichever is most recent., may be applied.
Sensitivity	S:N ≥10:1 for all compounds for 0.1 pg/µL (CS-0.2) plus, for blood/serum/plasma, S:N ≥3:1 for 0.025 pg/µL 2,3,7,8-TCDD.

[1] Duplicate criterion is a guideline; final assessment depends upon sample characteristics, overall batch QC and on-going lab performance.

[2] Instrument background specifications are calculated from spiking labeled standard into the toluene blank and expressed as pg in a 20 µL extract.

EPA 1614 Quality Control

QC Acceptance Criteria for Calibration, OPR and Sample Recovery

Compound Name	BDE No.	Initial Calibration	Calibration Verification		OPR Test	Samples		
		% RSD of RRFs	% Actual Concentration		% Actual Concentration	% Recovery		
			Warning Limit for Opening CAL-VER	Acceptance Limit for Opening and Closing CAL-VER		Warning Limit	Acceptance Limit	EPA 1614 Limits
NATIVE COMPOUND								
2,4,4'-TrBDE	28	<20%	-	70-130	50-150	-	-	
2,2',4,4'-TeBDE	47	<20%	-	70-130	50-150	-	-	
2,2',4,4',5-PeBDE	99	<20%	-	70-130	50-150	-	-	
2,2',4,4',6-PeBDE	100	<20%	-	70-130	50-150	-	-	
2,2',4,4',5,5'-HxBDE	153	<20%	-	70-130	50-150	-	-	
2,2',4,4',5,6'-HxBDE	154	<20%	-	70-130	50-150	-	-	
2,2',3,4,4',5',6-HpBDE	183	<20%	-	70-130	50-150	-	-	
2,2',3,3',4,4',5,5',6,6'-DeBDE	209	<20%	-	70-130	50-150	-	-	
SURROGATE STANDARDS								
$^{13}C_{12}$-4,4'-DiBDE	15L	<35%	70-130	50-150	30-200	40-150	25-200	-
$^{13}C_{12}$-2,4,4'-TrBDE	28L	<35%	70-130	50-150	30-200	40-150	25-200	25-150
$^{13}C_{12}$-2,2',4,4'-TeBDE	47L	<35%	70-130	50-150	30-200	40-150	25-200	25-150
$^{13}C_{12}$-3,3',4,4'-TeBDE	77L	<35%	70-130	50-150	30-200	40-150	25-200	
$^{13}C_{12}$-2,2',4,4',5-PeBDE	99L	<35%	70-130	50-150	30-200	40-150	25-200	25-150
$^{13}C_{12}$-2,2',4,4',6-PeBDE	100L	<35%	70-130	50-150	30-200	40-150	25-200	25-150
$^{13}C_{12}$-3,3',4,4',5-PeBDE	126L	<35%	70-130	50-150	30-200	40-150	25-200	
$^{13}C_{12}$-2,2',4,4',5,5'-HxBDE	153L	<35%	70-130	50-150	30-200	40-150	25-200	25-150
$^{13}C_{12}$-2,,2',4,4',5,6'-HxBDE	154L	<35%	70-130	50-150	30-200	40-150	25-200	25-150
$^{13}C_{12}$-2,2',3,4,4',5',6-HpBDE	183L	<35%	70-130	50-150	30-200	40-150	25-200	25-150
$^{13}C_{12}$-2,2',3,3',4,4',6,6'-OcBDE	197L	<35%	70-130	50-150	30-200	40-150	25-200	
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeBDE	209L	<100%	NQ[1]	NQ[1]	10-200	20-200	10-400	20-200
CLEANUP STANDARD								
$^{13}C_{12}$-2,2',3,4,4',6-HxBDE	139L	<35%	70-130	50-150	30-200	40-150	25-200	30-135
FIELD STANDARD								
$^{13}C_{12}$-2,2',3,4,4',5'-HxBDE	138L	<35%	70-130	50-150	50-150	-	50-150	-

[1] NQ = Not Quantified. Refer to Table 5B for in-house GC/MS specification for BDE 209L.

Table 5B. Additional QC Criteria

QC Parameter	Specification
Closing CAL VER (internal specification only, applies to all target compound in CAL)	Within ±20% of Opening CAL- VER for all natives compounds except BDE 203, 206, 207, 208 Within ±35% of Opening CAL-VER for BDE 203, 206, 207, 208 Within ±35% of the Opening CAL-VER for ^{13}C-surrogates except ^{13}C-BDE 209 Within ±70% of Opening CAL-VER for ^{13}C-BDE 209
Analysis Duplicate	Must agree to within ±20% of the mean (applicable to concentrations >10 times the DL) [a]
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Ion Ratios	Ion ratios must fall within ±15% of the theoretical values for positive identification of all targets in the calibration standards and samples.
Sensitivity	Minimum S:N ratio 10:1 for CS1. Minimum absolute response of BDE 209L in CAL-VER is 5 x 10^6 (Quant. + confirm. ions)
CAL-VER	Specification for BDE 209L is 25%-200% of actual concentration
Carryover	1st Toluene blank: >90% target compounds < 10 pg/20 µL, BDE 209 <200 pg/20 µL 2nd Toluene blank: < 5 pg/20 µL except BDE 209 < 100 pg/20 µL
Chromatogram Quality	BDE 49 and 71 must be uniquely resolved, valley height <40% of the shorter peak. Peak tailing ratio of $^{13}C_{12}$-BDE 99 and $^{13}C_{12}$-BDE 77 peaks (baseline peak width back half: front half) < 3:1. RT of BDE 209 must be > 48 min. RT of labeled surrogates in CAL-VER must be ±15 sec of those in ICAL.

[a] Duplicate criterion is a guideline; final assessment depends upon sample characteristics, overall batch QC and on-going lab performance.

EPA Method 1668 Quality Control

QC Acceptance Criteria according to EPA method 1668C for Chlorinated Biphenyls in CAL/VER, IPR, OPR, and Samples

Congener	Cong. No.[2]	Test conc. ng/mL	CAL/VER (%)		IPR[1] (%)		OPR[1] (%)		Labelled compound[1] % recovery in samples	
			Warning limits	Acceptance limits	RSD	$\bar{X}$	Warning limits	Acceptance limits	Warning limits	Acceptance limits
2-MoCB	1	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4-MoCB	3	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'-DiCB	4	50	75-125	75-125	25	70-130	70-130	60-135	-	-
4,4'-DiCB	15	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6-TrCB	19	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4'-TrCB	37	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2'6,6'TeCB	54	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4'-TeCB	77	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,4,4',5-TeCB	81	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,6,6'-PeCB	104	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4'-PeCB	105	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,4,4',5-PeCB	114	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5-PeCB	118	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2',3,4,4',5-PeCB	123	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5-PeCB	126	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',4,4',6,6'-HxCB	155	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5-HxCB[3]	156	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5'-HxCB[3]	157	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3',4,4',5,5'-HxCB	167	50	75-125	75-125	25	70-130	70-130	60-135	-	-
3,3',4,4',5,5'-HxCB	169	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,4',5,6,6'-HpCB	188	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5'-HpCB	189	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',5,5',6,6'-OcCB	202	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,3,3',4,4',5,5',6-OcCB	205	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3',4,4',5,5',6-NoCB	206	50	75-125	75-125	25	70-130	70-130	60-135	-	-
2,2',3,3,'4,5,5',6,6'-NoCB	208	50	75-125	75-125	25	70-130	70-130	60-135	-	-
DeCB	209	50	75-125	75-125	25	70-130	70-130	60-135	-	-

Attachment B

Influent and Effluent Sampling Locations

North Valley Interceptor (Influent)

Figure 1 shows the sampling location at the North Valley Interceptor (NVI). The sample will be taken from an access hatch located in the courtyard within the pump station gates. The sampler will sit in the open near the hatch. The strainer will be connected to the suction tubing and placed in the access hatch, as shown in Figure 1. Care will be taken to ensure the strainer does not contact the walls of the channel as it is lowered into the flow stream. The suction tubing will be threaded through a small keyhole in the hatch (see Figure 1). After threading the tubing through the keyhole, the suction line will be connected to the sampler. The wet well is cleaned weekly, and the County will coordinate sampling to occur the day after a cleaning, to avoid sample contamination with foam, grease, and other floating debris.

South Valley Interceptor (Influent)

Figure 2 presents the South Valley Interceptor (SVI) sampling location. The sampling from the SVI location will be similar to the NVI sampling setup. The only difference is that the SVI has two channels. Sampling will occur from whichever channel is currently in service. As with the NVI, prior to sampling, the County will coordinate sampling to occur the day after a cleaning, to avoid sample contamination with foam, grease, and other floating debris.

Effluent

The Spokane County Regional Water Reclamation Facility effluent (plant effluent) sampling location is presented in Figure 3. This manhole is downstream of the final treatment process. The manhole cover will be removed, and a piece of plywood will be placed over the manhole. A hole will be drilled into the plywood to allow passage of the suction tubing. The manhole is located in the roadway, so the site will need to be cordoned off with fencing/cones for traffic control. The sampler will be placed in the grass near the manhole. The suction tubing will be covered and protected to avoid pinching.

Additional sampling locations will be determined following the sampling from the three locations presented in Figures 1 through 3.

Figure 1. North Valley Interceptor Pump Station Sampling Location

Figure 2. South Valley Interceptor Pump Station Sampling Location

Figure 3. Effluent Sampling Location

Attachment C

Automated Sampling and Decontamination

The equipment decontamination and sampling procedures are described in this attachment. These procedures may be revised based on the rinsate blanks testing results and/or the Spokane River Regional Toxics Task Force (SRRTTF) SOP (when available).

Prior to the first sampling event, all sampling equipment that comes in contact with the sample water will be sent to AXYS Analytical Services Ltd to be cleaned using their standard procedures for ultra-low level organic analytes. The following equipment will be cleaned by AXYS:

- Suction tubing
- Pump tubing
- 2.5 gallon glass composite sample container and lid
- One liter amber glass sample bottles and lids
- Strainer

For subsequent sampling rounds, the sampling equipment will either be shipped to AXYS for cleaning or cleaned in the field by the sampling team according to the procedure detailed below.

Equipment Cleaning Procedure

1. Thoroughly clean the equipment using warm soapy water (Liquinox).
2. Rinse the equipment with tap water and then deionized water.
3. For the pump and suction tubing, place one end in the soapy water and manually pump the cleaning solution through the tubing.
4. Rinse three times with de-ionized water and let dry.
5. Rinse the composite sampling container and one liter sampling bottles with pesticide-grade acetone.
6. Package all cleaned equipment and label “precleaned.”

Staff performing the field sampling will follow the procedure outlined below to minimize sample contamination.

Sampling Protocol

Sampler Calibration:

1. Place the sampler in a secure location.
2. Check power sources and confirm the sampler is operable.
3. Using extra Teflon tubing (cleaned or uncleaned), test the calibration of the pump by pumping deionized water into a graduated cylinder. Note pump calibration in field notebook.
4. Test air purge and rinsing timing to ensure adequate timing during sampling event.
5. Record proper rinsing timing in field notebook.
6. Remove tubing and graduated cylinder.

Rinsate Blank Sample:

1. Put on powder-free nitrile gloves.
2. Remove precleaned sampling equipment from packaging.
3. Place precleaned sampling container in the sampler base.
4. Remove lid from composite sampling container and cover any exposed opening with aluminum foil. Leave an opening on the container for the tubing. Place the lid in foil, dull-side in.
5. Place precleaned pump tubing in the sampler's pump. Place the tubing in the container opening.
6. Do not allow exposed pump tubing ends to contact hands or any other surfaces.
7. Connect the precleaned suction tubing to the precleaned pump tubing.
8. Attach the precleaned strainer to the suction tubing and place strainer in ultrapure water container provided by AXYS.
9. Check to ensure pump tubing is secured in place.
1. Put the sampler in "sampling mode." Program the sampling frequency and timing based on calibration and rising timing previously recorded in the field notebook during the "Sample Calibration" to take one sample for the rinsate blank.
10. To create a rinsate blank, pump ultrapure water provided from AXYS through the sampling device and collect this water in the composite sampler.
11. Pour the water from the composite sampler into one of the precleaned one liter sample containers and label rinsate blank.
12. If pouring becomes difficult, decontaminate a stainless steel or glass funnel per the "Equipment Cleaning Procedure" above.

Sample Collection:

2. Add ice in sealed plastic bags (double bagged) or ice packs around the composite sample container. Ensure ice/ice packs will not contaminate sample.
3. Program the sampling frequency and timing based on calibration and rising timing previously recorded in the field notebook during the "Sample Calibration." Record the sample volume and frequency in the field notebook.
4. Program the sampler to rinse and then air purge between samplings.
5. Place the strainer in sampling location. Minimize the exposure to surfaces other than the sampling liquid.
6. Check sample tubing to ensure it is secured in place.
7. Start the sampler and watch one sample being collected.
8. Take photos and document all activities in the field notebook.

Sample Container Replacement:

1. There will always be one spare composite sampling container. If you think a sampler container has been contaminated, put the sampler into pause mode if in the middle of a sampling program.
2. Wear clean powder free gloves and practice clean sampling techniques.
3. Remove the sampler base. Do not allow the exposed pump tubing end to contact hands or any other surfaces.
4. Place the lid on the sampling container and replace container with precleaned composite bottle.
5. Continue sampler program and verify that the automatic sampler is in sampling mode.
6. Secure sampler at site.

7. Document composite bottle replacement activities in the field notebook.

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Sample Retrieval:

1. Upon arrival, inspect all components of the automatic sampling system to make sure sample was properly collected. If any unwarranted conditions are found, note conditions in field notebook.
2. Check battery levels and/or power source.
3. Visually inspect the components and tubing for damage and/or clogging.
4. Download field data from the automatic sampling system.
5. Wear clean powder free gloves and practice clean sampling techniques.
6. Place tubing and strainer in separate container and place lid on composite sample container.
7. Take sampler to Facility laboratory.
8. At laboratory, remove the composite sample bottle from the base and gently shake.
9. Remove composite sample bottle lid and pour directly into the precleaned one liter amber glass sample bottles provided by AXYS. Place the lids on the one liter sample bottles.
10. If pouring becomes difficult, decontaminate a stainless steel or glass funnel per the "Equipment Cleaning Procedure" above.
11. Label each sample bottle (if not prelabeled) with appropriate location, date and sample identification.
12. Wrap the sample bottles with bubble wrap to prevent breakage of glass and place sample bottles inside a sealed Ziploc bag.
13. Place sample bottles in cooler for transport.
14. Wrap with bubble wrap and place travel blank into cooler.
15. Add ice around the sample bottles to keep them cool during transport (between 0ºC and 6ºC).
16. Fill out chain of custody.
17. Rinse suction tubing, pump tubing, strainer, 2.5-gallon glass composite sampling container with soapy water.
18. Ship samples overnight to AXYS. Keep samples in the dark and cool during transport.
19. Ship sampling equipment via ground to AXYS to be precleaned prior to the next sampling event.

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