

# Appendix D: Analytical Methods

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## APPENDIX D - LABORATORY METHODS

A summary of analytical methods are presented in this section. The King County Environmental Laboratory (KCEL) reports both the reporting detection limit (RDL) and the method detection limit (MDL) for each sample and parameter, where applicable. PCB congeners were analyzed using high resolution isotopic dilution based methods, therefore MDL and RDL terms are less applicable because limits of quantitation are derived from calibration capabilities and ubiquitous, but typically low level equipment and laboratory blank contamination. As a result, PCB and dioxin/furan congener data are reported to lowest method calibration limits (LMCLs) and flagged as estimated down to the sample specific detection limit (SDL). In most cases the SDL is below the LMCL. The following sections provide a summary of the laboratory methods; greater detail can be found in the project QAPP (King County 2015).

### D.1 KCEL Analysis

KCEL analyzed all parameters except PCBs. Standard Method SM5310B was used for TOC and DOC analysis, while Standard Method SM2540D was used for TSS analysis. Nutrients were analyzed using SM4500-P-F (orthophosphate phosphorus), SM4500-P-B, F (total phosphorus), SM4500-N-C (total nitrogen), SM4500-NO3-F (nitrate-nitrite nitrogen), and Kerouel & Aminot (1997; ammonia nitrogen).

Total and dissolved metals were analyzed and reported by EPA Method 200.8 (Inductively Coupled Plasma-Mass Spectrometry [ICP-MS]), KCEL SOP 624. The specific metals analyzed included: cadmium, copper, lead, and zinc. PAHs samples were prepared by liquid-liquid extraction in general agreement with EPA method 3520C. Samples were analyzed by a modified EPA Method 8270 Gas Chromatography/Mass Spectrometry – Selected Ion Monitoring method (GC/MS-SIM; KCEL SOP 731v5). The specific PAHs included: 2-methylnaphthalene, acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(g,h,i)perylene, benzo(a)pyrene, benzo(b,j,k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluorene, fluoranthene, indeno (1,2,3-cd)perylene, naphthalene, phenanthrene, and pyrene.

The QAPP specified bacteria would be analyzed as fecal coliforms using Standard Method SM9222D; however, there was interference in the cultures. After the first set of samples with interference, the laboratory began side-by-side analysis with *Escherichia coli* (*E. coli*) to test comparability. Starting in October 2016, fecal coliform analysis was dropped in favor of *E. coli* analysis for all remaining samples. *E. coli* was analyzed using Standard Method SM9213D/3B.

Toxicity tests were conducted for four sets of samples collected at one of the bioretention planter boxes (BPB1). For each event two toxicity tests were conducted: (1) a 48-hour acute test with *Daphnia pulex* following KCEL SOP #412v2 and EPA Test Method 2021.0 (acute *Daphnia pulex*), and (2) a 7-day chronic test with *Ceriodaphnia dubia* following KCEL SOP #408v3 and EPA Method 1002.0 (chronic *Ceriodaphnia dubia*).

## D.2 PCB Analysis

PCBs are found in up to 209 different chemical forms, called congeners, and were measured using high resolution PCB congener analysis. Analysis followed EPA Method 1668 Revision C (EPA 2010), which is a high-resolution gas chromatography/high-resolution mass spectroscopy (HRGC/HRMS) method using an isotope dilution internal standard quantification. This method provides reliable analyte identification and very low detection limits. An extensive suite of labeled surrogate standards are added before samples are extracted. Data are “recovery-corrected” for losses in extraction and cleanup, and analytes are quantified against their most similar labeled analogues. The analysis included all 209 PCB congeners. Pacific Rim Laboratories (PRL) performed the PCB congener analysis according to their SOP LAB02. The samples were extracted followed by standard method clean-up, which includes an acid wash followed by Acid Silica, Carbon, and Alumina column chromatography. Analysis is performed with an SGE HT-8 column.

## D.3 Deviations from the QAPP

- The QAPP specified that DOC and TOC would only be analyzed for effluent samples at the DTS; however, the project manager requested that DOC analysis be added to the BPB and Filterra samples starting in 2017, because of surprisingly low dissolved copper concentrations observed in the BPB effluent during an initial review of the data. The BPB samples collected on 12/8/2015 also included TOC and DOC analysis, although this was an unplanned deviation. This does not affect data quality, but provides additional information to the project.

## Appendix D References

EPA. 2010. Method 1668C, Chlorinated biphenyl congeners in water, soil, sediment, biosolids, and tissue by HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Office Science and Technology, Washington, D.C. EPA-820-R-10-005.

King County. 2015. Quality Assurance Project Plan for Monitoring Stormwater Retrofit in the Echo Lake Drainage Basin – RSMP Effectiveness Study. Prepared by Carly Greyell, Water and Land Resources Division. Seattle, Washington.