COMMONWEALTH OF VIRGINIA
DEPARTMENT OF ENVIRONMENTAL QUALITY
WATER DIVISION
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P.O.BOX 1105 **Richmond, VA 23218**

Subject: TMDL Guidance Memo No. 09-2001. Guidance for monitoring of point sources for TMDL development using low-level PCB method 1668.

To: Regional Directors

From: Ellen Gilinsky Ellen Gilinsky

Date: March 6, 2009

Copies: James Golden, Rick Weeks, Regional Water Permit Managers and Watershed Staff

Summary:

This guidance establishes procedures for implementing point source monitoring of polychlorinated biphenyls (PCBs) in support of Total Maximum Daily Loads (TMDL) development. This monitoring applies to the following categories of point sources discharging to PCB impaired waters: municipal (major and minor) and industrial wastewater facilities; and industrial storm water discharges, whether operating under an individual or general storm water permit. PCB monitoring entails sample collection and low-level analysis using EPA Method 1668 to determine individual PCB congener concentrations. This action replaces the current method that is unable to detect low-level PCBs against the Virginia water quality criterion for total PCBs.

Electronic Copy:

An electronic copy of this guidance in PDF format is available for staff internally on DEQNET, and for the general public on DEQ's website at: http://www.deq.virginia.gov.

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Disclaimer:

Guidance documents are developed as guidance and, as such, set forth presumptive operating procedures for the Agency. Guidance documents do not establish or affect legal rights or obligations, do not establish a binding norm, and are not determinative of the issues addressed. However, this document does not mandate any particular method nor does it prohibit any particular method for the analysis of data, establishment of a wasteload allocation, or establishment of a permit limit. If alternative proposals are made, such proposals should be reviewed and accepted or denied based on their technical adequacy and compliance with appropriate laws and regulations.

Monitoring of Point Sources for TMDL Development Using Low-Level PCB Method 1668

I. Introduction

The purpose of this guidance is to establish procedures for implementing point source monitoring of polychlorinated biphenyls (PCBs) in support of Total Maximum Daily Loads (TMDLs) development. This monitoring may apply to the following categories of point source discharges: 1) municipal (major and minor) and industrial wastewater facilities, and 2) industrial storm water discharges, whether operating under an individual or general storm water permit. Facilities will be notified by the DEQ regional office if low-level PCB monitoring is requested. The selection of facilities to conduct monitoring is determined by regional permitting and TMDL staff and is based upon the criteria identified herein.

PCB monitoring entails sample collection and low-level analysis using the most recent version of Environmental Protection Agency's (EPA) Method 1668. Currently, Revision B (1668B) (EPA 2008) is used to determine individual PCB congener concentrations. Implementation of this procedure will result in the collection of low-level data for source-specific PCB effluent concentrations to meet the required elements of the TMDL submittal by identifying sources and to quantify the existing loads for TMDL development. While the low-level PCB Method 1668 has not yet been promulgated by EPA, they recommend its use for data generated in support of TMDL development. Therefore, data generated under this guidance should not be used for compliance purposes.

This guidance was developed with assistance of a PCB Point Source Monitoring Technical Advisory Committee (TAC). The TAC consisted of representatives from both the regulated community and environmental groups throughout the state. The PCB point source monitoring approach being adopted is similar to those used in New York (Panero *et al*., 2005) (http://www.nyas.org/programs/harbor.asp), Delaware and New Jersey (DRBC 1998) (http://www.state.nj.us/drbc/regs/pcb-new.pdf) and employed in the Tidal Potomac PCB TMDL developed by EPA in cooperation with District of Columbia, Maryland, and Virginia (Haywood and Buchanan 2007).

II. Background

In 2004, the Virginia Department of Health lowered the trigger value for fish consumption advisories for PCBs from 600 ppb to 50 ppb. The following year, the Virginia Department of Environmental Quality (DEQ) published the "PCB Strategy for the Commonwealth of Virginia" (www.deq.virginia.gov/fishtissue/pcbstrategy.html). This document describes the regulatory framework and state initiatives to address PCB impaired waterbodies due primarily to high PCB levels in fish tissue.

III. Authority

Development of a PCB TMDL requires consideration of the Virginia water quality criterion for Total PCBs (9 VAC 25-260-140) to protect the "fishable" designated use (9VAC 25-260-10). The current PCB compliance Method 608 (40 CFR Part 136) is incapable of meeting these regulatory requirements as the method detection level is well above the water quality criterion. In order to characterize PCB loadings for TMDL development, DEQ is implementing low-level PCB monitoring as recommended by EPA (Appendix A). The monitoring will be coordinated through the Virginia Pollutant Discharge Elimination System (VPDES) permit and TMDL programs.

IV. Definitions

"Composite Sample" as used in this guidance is a combination of individual samples of water or wastewater taken in proportion to flow or time which ensures that a representative sample is collected. Composites most often represent samples collected over 24 hours.

"Congener" is a chemical compound in the polychlorinated biphenyls (PCB) category and is a derivative or a compound in the same group. In this case, there are 209 congeners of PCBs.

"Industrial storm water" as referenced by the VPDES Permit Regulation (9VAC25-151-10) means storm water runoff associated with the definition of "storm water discharge associated with industrial activity."

"Manual composite" as used in this guidance is a "composite sample" which consists of individual grab samples taken during a time sequence and final compositing of the individual grabs occurring within the laboratory and not at the facility where the samples were collected.

"Method 1668" is an analytical method developed by the EPA Office of Water's Office of Science and Technology for use in Clean Water Act programs. This method is used to determine chlorinated biphenyl congeners in environmental samples by isotope dilution and internal standard high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). This analytical method has been recommended by EPA for data generation related to TMDL development.

"No exposure" as referenced by VPDES Permit Regulation (9VAC25-151) means all industrial materials or activities are protected by a storm-resistant shelter to prevent exposure to rain, snow, snowmelt, and/or runoff.

"PCB" and PCBs as referenced in EPA 40 CFR 761 means any chemical substance that is limited to the biphenyl molecule that has been chlorinated to varying degrees or any combination of substances which contains such substance. They are a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl and a general chemical formula of $C_{12}H_{10}$-xClx (where x = 1-10). For purposes of this Guidance, total PCBs is the summation of all detected PCB congeners (refer to Appendix D - Attachment 3 for use of qualifiers).

"pg/L" refers to picograms per liter (pg/L) and corresponds to parts per quadrillion (ppq) (1.0 E-12) (EPA 1999a, 2008).

"Publicly owned treatment works" or "POTW" as defined by the VPDES Permit Regulation (9VAC25-31-10) means a treatment works as defined by §212 of the Clean Water Act, which is owned by a state or municipality (as defined by §502(4) of the Act). This definition includes any devices and systems used in the storage, treatment, recycling and reclamation of municipal sewage or industrial wastes of a liquid nature. It also includes sewers, pipes and other conveyances only if they convey wastewater to a POTW treatment plant. The term also means the municipality as defined in §502(4) of the Act, which has jurisdiction over the indirect discharges to and the discharges from such a treatment works.

"Process wastewater" referenced in VPDES Permit Regulation (9VAC25-31-10) means any water which, during manufacturing or processing, comes into direct contact with or results from the production or use of any raw material, intermediate product, finished product, byproduct, or waste product.

“SIC” means the Standard Industrial Classification Code or Industrial Grouping from the U.S. Office of Management and Budget Standard Industrial Classification Manual, 1987 Edition. It is used to describe the specific industrial activities occurring at a facility to determine whether or not the facility is required to be permitted under the regulations.

“Storm event” as used by the general permit monitoring instructions (9VAC25-151-70-A.2.b) refers to an event that is greater than 0.1 inch in magnitude (defined as a “measurable” event), providing the interval from the preceding measurable storm is at least 72 hours. In this guidance, high flow events or wet conditions are storm events. To be consistent with monitoring requirements at 9 VAC 25-151-70-A.1.a(2), snow melt samples are not to be considered.

“Storm water” referenced in VPDES Permit Regulation (9VAC25-31-10) means storm water runoff, snow melt runoff, and surface runoff and drainage.

V. Procedure

The objective of this guidance is to facilitate the generation of low-level PCB data for TMDL development as well as identify and quantify sources of PCBs as required (EPA 1992, 1999b). Monitoring and data analysis is performed using EPA approved procedures for total PCBs on a congener basis (EPA 1999a, 2008).

A. Facilities Identified for Monitoring

VPDES permitted facilities discharging into PCB impaired waters and subject to this guidance include:

- all major municipals (POTWs) (1.0 MGD design capacity and above, including combined sewer overflows or CSOs),
- minor municipals (less than 1.0 MGD),
- industrial wastewater facilities, and
- industrial storm water discharges under individual or general permits.

Certain exceptions may be considered:

- For minor municipal facilities, the permittee shall provide adequate documentation that the facility is not a potential source of PCBs. This is based on a certified report by the owner and may include site inspection by DEQ staff.
- Storm water discharge is to a POTW or through a CSO, or if the facility meets the definition of “no exposure” under 9 VAC 25-151-70.
- A representative outfall may be sampled by the permittee, if a facility has two or more outfalls that discharge substantially identical effluents as specified in 9 VAC 25-151-70.
- An industrial facility may ask for a waiver from monitoring if the owner can certify that PCBs were never present on the site. However, if the written certification letter is not

supported by adequate low-level PCB data obtained in accordance with method described in Appendix C and D, DEQ may still request monitoring to obtain the data. The decision will be case by case based on the needs of the TMDL.

- This guidance does not apply to municipal separate storm water systems permitted by DCR.

Specific types of industrial or commercial operations are more likely than others to have a discharge that includes PCBs. Therefore, industrial activities with primary or secondary Standard Industrial Classification ("SIC") codes identified in Table 1 are subject to this monitoring guidance. Other industrial facilities may be identified for monitoring based on additional information or recommendations of DEQ technical staff.

Table 1. Industrial facilities by SIC code subject to PCB monitoring guidance.

SIC Code	Code Name Facility	SIC Code	Code Name Facility
26 & 27	Paper and Allied Products	5093	Scrap recycling
30	Rubber and Misc. Plastics	1221 & 1222	Bituminous Coal
33	Primary Metal Industries	3612	Transformers
34	Fabricated Metal Products	3731 & 3732	Ship/Boat Building/Repair
37	Transportation Equipment	4011	Railroad Transportation
49	Electrical, Gas and Sanitary Services	5015	Automobile Salvage Yards

Once a PCB impaired segment appears on the TMDL development schedule, the regional TMDL coordinator is responsible for notifying the facility of data needs. If data for TMDL source characterization are not available through this effort, DEQ may request data by letter or amend a VPDES permit with special conditions (Appendix B).

B. Monitoring Frequency

The minimum monitoring frequency for facilities described in Section A is outlined in Table 2. For load characterization, both base flow (dry) and storm or high flow event (wet) sampling are recommended as described in Appendix C unless stated otherwise.

Table 2. Type of facility and minimum monitoring frequency recommended.

VPDES Facility				
Municipals		Industrials		
Major ≥ 1 MGD	Minor < 1 MGD	Process wastewater only	Process wastewater with storm water	Storm water only
2 wet + 2 dry	1 wet + 1 dry	2 samples (storm event sampling not required)	1 dry + 1 wet	2 wet

Dischargers subject to the monitoring guidance shall begin monitoring within one year of receipt of DEQ notification and conclude the monitoring within two years of receipt of the notification from DEQ. Samples previously collected and analyzed, may be used in satisfying the total number of samples required provided monitoring and analysis are conducted in accordance with Sections C and D of this guidance.

C. Sample Collection and Analytical Requirements

The analytical approach used with this guidance is EPA Method 1668B (EPA 2008), capable of detecting low-level concentrations for all 209 PCB congeners. Method 1668B has replaced 1668A (EPA 2008) (http://www.epa.gov/waterscience/methods/). Individual congeners are summed to form total PCB. Based on the sensitivity of this high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS), it is necessary to follow ultra-clean sample collection and handling techniques. Specific guidance for sample collection is provided in Appendix C. The application of these procedures ensures consistency between sampling events and among participants collecting samples.

Some facilities have expressed an interest in monitoring their water supply intake in order to demonstrate that the wastewater treatment plant (WWTP) is not an actual source of PCBs. Influent and effluent data from WWTPs in Washington State indicate that the treatment plants effectively remove greater than 95% of the PCBs in the influent to the plant. A WWTP therefore cannot demonstrate that they are not a source of PCBs by showing that the PCBs in the intake and in the effluent are of the same order of magnitude. Any effort to make such a demonstration must include intake, WWTP influent and effluent analysis along with a comparison of the individual congeners in each. Sample collection and analysis at all three locations should be consistent with this guidance and procedures with particular attention to this section and PCB Reporting Requirements (Section E) below.

Method 1668 is performance based, which allows analytical laboratories to improve upon the method capabilities (EPA 2008). Appendix D contains the specified laboratory requirements. Included are the congener-specific Estimated Method Detection Levels (EMDLs) and the Minimum Level (ML) or concentration(s) at which the congeners are to be reported. Analytical consistency along with the ability to meet quality control requirements is essential among participating laboratories.

D. Analytical Laboratories

While the agency cannot recommend any testing laboratory, a list of laboratories that conduct testing and employ equipment capable of performing EPA Method 1668 will be on file and posted on the DEQ web site. However, the laboratory must be capable of meeting the EMDLs and MLs specified in Appendix D (http://www.deq.state.va.us/tmdl/pcb.html).

E. PCB Reporting Requirements

Data should be delivered to DEQ in two electronic data formats. Format and files are described in Appendix E and are available for download from the DEQ TMDL website (http://www.deq.state.va.us/tmdl/pcb.html).

F. References

Delaware River Basin Commission (DRBC). 1998. Study of the loadings of polychlorinated biphenyls from tributaries and point sources discharging to the tidal Delaware River.

Estuary Toxics Management Program, DRBC West Trenton, New Jersey. June.

Environmental Protection Agency (EPA). 1999a. Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment and Tissue by HRGC/HRMS, EPA-821-R-00-002, December 1999. *(with corrections and changes through August 20, 2003)*

Environmental Protection Agency (EPA). 1999b. Draft Guidance for Water Quality-based Decisions: The TMDL Process (Second Edition). EPA 841-D-99-001.

Environmental Protection Agency (EPA). 1992. Reviewing TMDLs Under Existing Regulations Issues in 1992. http://www.epa.gov/owow/tmdl/guidance/final52002.html

Environmental Protection Agency (EPA). 2008. Method 1668B, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS. EPA-821-R-08-020, November 2008.

Haywood, H. C. and C. Buchanan. 2007. Total maximum daily loads of polychlorinated biphenyls (PCBs) for tidal portions of the Potomac and Anacostia rivers in the District of Columbia, Maryland, and Virginia. Interstate Commission on the Potomac River Basin. ICPRB Report 07-7. Rockville, MD. October 2007.

Panero, M, S Boehme and G Munoz. 2005. Pollution Prevention and Management Strategies for Polychlorinated Biphenyls in the New York/New Jersey Harbor. A Report from the Harbor Consortium of the New York Academy of Sciences, NYAS, February, 2005.

VI. Appendices

A. NPDES Permitting Authorities have some discretion in specifying methods more sensitive than 40 CFR Part 136**.** Draft memo from Brian P. Trulear, NPDES Program Manager, EPA Region 3. June 8, 2007.
B. Example Notification Letters
C. Sample Collection Methods for Effluent and Storm Water
D. Virginia PCB TMDL Analytical Quality Control Requirements
E. Reporting Requirements for Analytical (PCB) Data Generated Using EPA Method 1668

Appendix A

Draft memo from Brian P. Trulear, NPDES Program Manager (Office of Permits and Enforcement, EPA Region III Water Protection Division) to Charles Martin (DEQ Watersheds Manager) dated June 8, 2007.

NPDES Permitting Authorities have some discretion in specifying methods more sensitive than 40 CFR Part 136 as follows:

EPA regulations require that samples and measurements taken for purposes of monitoring shall be representative of the monitored activity. [40 CFR 122.41(j)(1) & 122.48(b)] EPA has approved test methods under 40 CFR Part 136 for use as compliance monitoring requirements in an NPDES permit. [40 CFR 122.41(j)(4)] Where an authorized State wants to include in an NPDES permit requirements to monitor for informational purposes with methods more sensitive than the measurement capabilities of methods approved in Part 136, the State may specify the suitable method. Under these circumstances, the State is not bound to require the use of a Part 136 method because no such method exists to provide data at required levels. The NPDES permitting authority is responsible for ensuring that the specified test method will yield results at concentrations of concern that are reliable enough to meet the needs for permit monitoring under the Clean Water Act. In addition, if an appropriate non-136 method is required for the use in the permit, the NPDES permitting authority should specify in the permit Fact Sheet / Statement of Basis not only the selected method but also state the rationale for specifying the selected method.

Basis for draft option above:

1. EPA regulations at 40 CFR 122.41(j) and 122.48 can be read together to allow flexibility for the NPDES Issuing Authority to use non-136 methods that give representative results when there is no 136-approved method with sensitivity in the range of concern (and therefore cannot give representative results).

2. The EPA Part 136 method for monitoring PCBs is Method 608. This is the method currently to use for compliance purposes. Method 608 can measure only a limited number of PCB congeners with a Method Detection Limit (MDL) of 0.065 micrograms per liter. VA has a water quality standard of 0.0017 micrograms per liter for total PCBs. Method 1668A, which measures total PCBs on a congener-specific basis, has an MDL of 5.0 picograms (0.000005 micrograms) per liter and would provide more representative data for use in TMDL development. 1668A has been proposed for adoption into Part 136. The method is based on validation in a single laboratory. The basic revision of Method 1668 was validated in two laboratories. (See EPA publication No. EPA-821-R-00-002 which can be found at http://www.epa.gov/Region3/1668a.pdf) Adopting new methods into Part 136 or approving alternative test procedures is a time consuming, expensive and cumbersome process. Therefore, EPA recognizes that there is potentially a long lag time between the development of new reliable methods and the Agency's adoption into Part 136, and that delay should not be an absolute barrier to the valid exercise of an NPDES permitting authority to establish representative monitoring requirements in the permit.

3. EPA Region 3 is proposing the use of 1668A for monitoring purposes in an EPA NPDES permit for a power plant in DC. "Samples for compliance purposes are to be tested using

> EPA approved Method 608. (The permittee may test split samples using Method 1668A, for informational purposes only. Method 1668A sampling results shall not be used for compliance purposes.)" The permittee has agreed to this testing.

Applicable regulations:

The applicable regulations regarding the use of Part 136 approved methods are 40 CFR 122.41(j)(1) & (4); 122.44(i)(1)(iv); and 122.48.

40 CFR 122.41(j)(1) requires that "samples and measurements taken for purposes of monitoring shall be representative of the monitored activity."

122.41(j)(4) require that "monitoring results must be conducted according to test procedures approved under 40 CFR Part 136... unless other test procedures have been specified in the permit."

122.44(i)(1)(iv) requires permits to specify monitoring requirements "(a)ccording to the test procedures approved under 40 CFR part 136 for the analysis of pollutants having approved methods under that part, and according to a test procedure specified in the permit for pollutants with no approved method."

40 CFR 122.48 requires that "[a]ll permits must specify require monitoring including type, intervals, and frequency sufficient to yield data which are representative of the monitored activity…."

Previous EPA statements: This is consistent with the approach in "Strategy for the Regulation of Discharges of PHDDs and PHDFs from Pulp and Paper Mills to Waters of the United States" dated 05/21/1990, as excerpted below:

Analytical Methods -

EPA regulations found at 40 CFR 122.41(j)(4) require that monitoring results must be conducted according to test procedures approved under 40 CFR Part 136... unless other test procedures have been specified in the permit." The analytical method currently specified in 40 CFR Part 136 for dioxin is EPA method 613. Method 613 is a low resolution method incapable of detecting dioxin in the range of many of the recently developed water quality-based limitations. Therefore, it would be inappropriate to include method 613 in current pulp and paper mill permits. Instead, EPA recommends U.S. EPA method 1613 as the analytical method which should be specified in permits in conjunction with numerical permit limitations for 2,3,7,8-TCDD and limitations and/or monitoring requirements for other PHDDs and PHDFs. Thus, method 1613 should be specified on a permit- specific basis citing the authority of 40 CFR 122.41(j)(4) and 122.44(i)(l)(iv). This high resolution method was not available at the time of the March 15, 1989 guidance. Although this method has not yet been formally promulgated and published in 40 CFR Part 136, its use is recommended. EPA method 1613 can also be used to determine other dioxin/furan congeners in effluents.

Appendix B
Example Notification Letters

Date

Name
Title
Facility Name
Street Address
City, State Zip

RE: VPDES Permit No.
Facility Name

Dear :

The Department of Environmental Quality is beginning the process of developing a Total Maximum Daily Load (TMDL) for PCBs in the watershed. In order to identify all possible sources of PCBs and to develop existing loads for those sources, low-level PCB monitoring of VPDES permitted that discharge into impaired waters must be performed.

Sampling and laboratory analysis of low-levels of PCBs requires the use of specialized techniques which are outlined in the attached guidance memorandum. While the agency cannot recommend a specific testing laboratory, a list of qualified laboratories using performance based EPA Method 1668 (current version) is also attached. Low-level PCB monitoring is expensive. However, substantial cost savings can be achieved if facilities work together. Generally, analytic costs decrease as the number of samples increase. Therefore, facilities can collect their own samples and submit them as a group (batch) to an approved laboratory for subsequent analysis. Failure to generate these data using the prescribed approach shall result in the derivation of estimated PCB loads.

An informational meeting is schedule for on in DEQ's Regional Office. This meeting is to facilitate possible coordination by the watershed permit holders and to answer any questions you have regarding low-level PCB analysis and the TMDL process. We hope that you will be able to attend.

If you have any questions, please contact at .

Sincerely,

Name
Title

Date

Name
Title
Facility Name
Street Address
City, State Zip

RE: VPDES Permit No.
Facility Name

Dear :

The Department of Environmental Quality has initiated the development of a Total Maximum Daily Load (TMDL) for Polychlorinated Biphenyls (PCBs) in the watershed. In order to identify all possible sources of PCBs and to develop appropriate source characterization for applicable point sources, low-level PCB monitoring of VPDES permitted discharges to impaired waters has been requested using specialized low-level techniques.

An informational meeting was held at DEQ's Regional Office on , 200x. The purpose of the meeting was to allow VPDES permit holders the opportunity to gain a more comprehensive understanding of the TMDL process as well as answer questions regarding low-level PCB sample collection and analysis. A secondary purpose was to facilitate possible coordination amongst the dischargers to provide the opportunity for substantial cost savings by pooling samples for PCB analysis at a laboratory.

In order to address the PCB impairment, a TMDL development includes assigning WLA to all point sources that discharge to the impaired waterbody. This letter is being provided to alert you to DEQ's need for PCB data for development of existing loads from your facility. In accordance with Section 9 VAC 25-31-190.H of the Board's VPDES Permit Regulation and Part II.D of your VPDES permit, you are requested to provide the results of sampling events (dry weather and wet weather) to this office by . Sampling and laboratory analysis for low-level PCBs requires the use of specialized techniques which are outlined in the attached guidance memorandum. While the agency cannot recommend a specific testing laboratory, a list of qualified laboratories using performance based EPA Method 1668 is also attached. Failure to generate these data using the prescribed approach shall result in the derivation of estimated PCB loads.

If you have any questions, please contact at .

Sincerely,

Name
Title

VPDES Permit Special Conditions

This special condition shall be used only for data generation as part of TMDL development or implementation. Non-numeric water quality based effluent limits shall be used to ensure that the permits are consistent with the WLA provisions of the TMDL. For new permits or those scheduled for reissuance, the following language is offered. The sample number will depend on the type of facility as described in Section B of the Guidance:

> The permittee shall monitor the effluent at Outfall ___ for Polychlorinated Biphenyls (PCBs). DEQ will use these data for development (*or implementation*) of a PCB TMDL for the ______name of waterbody___ . The permittee shall conduct the sampling and analysis in accordance with the requirements specified below. At a minimum:
>
> a. Monitoring and analysis shall be conducted in accordance with the most current version of EPA Method 1668, congener specific results as specified in the PCB Point Source Monitoring Guidance. It is the responsibility of the permittee to ensure that proper QA/QC protocols are followed during the sample gathering and analytical procedures.
>
> b. The permittee shall collect a minimum of 2 wet weather samples and 2 dry weather samples according to the PCB Point Source Guidance, Appendix C (Sample Collection Methods for Effluent and Storm Water). These samples shall be taken at Outfall ___during the term of the permit. Samples previously collected and analyzed with Method 1668, may be used in satisfying the total number of samples required even if the collection occurred prior to the current permit term.
>
> c. The sampling protocol shall be submitted to DEQ-__Regional Office for review and approval prior to the first sample collection.
>
> d. The data shall be submitted to DEQ-___Regional Office by the 10th day of the month following receipt of the results according to the PCB Point Source Guidance, Appendix E (Reporting Requirements for Analytical (PCB) Data Generated Using EPA Method 1668). The submittal shall include the unadjusted and appropriately quantified individual PCB congener analytical results. Additionally, laboratory and field QA/QC documentation and results should be reported. Total PCBs are to be computed as the summation of the reported, quantified congeners.
>
> e. If the results of this monitoring indicate actual or potential exceedance of the water quality criterion or the Waste Load Allocation specified in the approved TMDL, the permittee shall submit to DEQ- ____Regional Office for review and approval a Pollutant Minimization Plan (PMP) designed to locate and reduce sources of PCBs in the collection system. A component of the plan may include an evaluation of the PCB congener distribution in the initial source intake water to determine the net contributions of PCBs introduced to the treatment works.

Appendix C
Sample Collection Methods for Effluent and Storm Water

(Edited 1/26/09)

1.0 Background

To assist in the generation of low-level PCB data to better characterize PCB loadings from point sources and to aid with TMDL development and implementation, a scientifically sound approach is necessary for collection and analysis of PCB samples from VPDES wastewater sources. Previously, PCBs have not been detected in effluents with the use of the current compliance method, EPA Method 608. The method has elevated detection levels (0.5 – 50 ug/L) and is selective toward PCB Aroclor analysis. Recently, EPA has recommended the use of Method 1668 for TMDL development since it supports a much lower detection. It uses clean sampling techniques and a congener-specific, high resolution/low detection analytical method in the pg/L range. Subsequent data generation during tidal Potomac River and the Roanoke River PCB TMDL development showed effluent discharged by municipal and industrial point sources may contribute to the overall loading of PCBs to these impaired waters.

Whether the effluent is from a continuous flowing discharge or is storm water based, the sample collection methods contained in this appendix were developed to: 1) ensure PCB samples are collected on a consistent basis using clean techniques, 2) provide flexibility to account for variation in sampling points at different facilities, and 3) contain costs where possible.

2.0 Effluent/Industrial Storm water Samples

There are several factors that may determine what sampling approach is utilized by a discharger. Consideration for choosing an approach could depend on whether the wastewater is discharged on a continual or intermittent basis, the impact of storm water, and the physical location or accessibility to the final discharge point. The two available options for collecting point source wastewater include: 1) twenty-four hour composite samples or 2) instantaneous grab samples. The latter may consist of a single sample, or two or more samples collected at a pre-determined frequency and then composited at the analytical laboratory. For continuous dischargers, the approach used to collect a sample can be determined by the permit holder but should be in accordance with the guidelines specified within this protocol. Dischargers of intermittent industrial storm water are limited to the use of grab samples. Either sampling method is considered acceptable for TMDL development with recognition that there are advantages and disadvantages to each approach.

The <u>required volume</u> needed for PCB analysis is a minimum of 2 liters although a 4-liter sample volume is preferred (i.e., sample volume ≥ 2 L ≤ 4 L). There is greater benefit with the 4-liter sample in mitigating the effects of background PCBs on the final analytical results derived using the ultra low detection method. The ability to meet the objectives of the PCB monitoring guidance will be compromised if the defined sample volume is not collected.

Effluent samples are to be collected during dry conditions and wet events at municipal and industrial facilities that discharge final effluent susceptible to storm water input. Please note this is applicable to municipal wastewater that receives storm water input such as inflow and infiltration, and industrial process wastewaters that combine with storm water prior to discharge.

Dry weather condition is defined as no measurable (less than 0.1 inch) rain event has occurred within the past 72 hours. Wet weather condition is defined as a wet event that exceeds 0.1 inch and results in an increase in wastewater flow, and that occurs at least 72 hours from the previously measured (> 0.1 inch) storm event. While the guideline for the dry weather sampling should be simple to follow, adherence to the wet weather guideline may be more problematic for reasons such as variation in facility retention time, etc. Therefore, in determining the best time to collect samples, facility personnel may apply Best Professional Judgment (BPJ) as it relates to wet weather influencing the facility hydrograph (i.e., increase). A guideline to follow for wet weather sample collection coincides with a precipitation based event (>0.1 inch) where the influent flow increases by 10% or more from the base influent flow, and considers the retention time of the facility. BPJ can be supported with information such as facility retention time, plant hydrograph, local rain gage data, etc. during the sample collection period.

Industrial storm water applies to those outfalls noted in Part V. A. of the guidance. Storm water discharge associated with industrial activity means the discharge from any conveyance which is used for collecting and conveying storm water and that is directly related to manufacturing, processing or raw materials storage areas at an industrial plant. A grab sample must be taken during the first 30 minutes of the discharge, or within the first hour if impracticable. A resulting storm event must be at least 0.1 inch of precipitation (defined as a "measurable" event) and the interval from the preceding measurable storm is at least 72 hours.

3.0 Summary of Method

3.1 Twenty-four hour composite samples

Twenty-four hour composite samples are only applicable to continuously discharging effluent. Wastewater samples should be collected using a programmable automated sampler capable of capturing equal aliquots of a water based medium on a time-weighted basis. Flow-weighted samples are also acceptable. An equipment or rinsate blank shall be collected prior to effluent coming into contact with the sampling unit and is obtained by pumping a minimum of 2 liters of PCB free de-ionized (DI) water through the hoses directly into an appropriately sized (2 to 4 liter) certified pre-cleaned amber glass sample bottle. While it is preferred that PCB samples be collected directly into the certified clean bottle, an intermediate glass receptacle that is compatible with the composite sampler is also acceptable for sample collection provided the container is certified clean. The PCB free de-ionized water should originate from the analytical laboratory. Although not required, it is suggested that demonstration of PCB free sampling equipment be provided through analysis with the most current versions of method 1668 prior to effluent collection. Alternatively, the equipment blank can be collected on-site prior to effluent collection and will concurrently serve as a field blank (see Appendix D, Attachment I.B. for acceptance criteria). Twenty-four hour composite samples are to be collected by placing new, solvent rinsed Teflon lined tubing with an attached intake-strainer into the wastewater at the point, or as close as possible, where the permit compliance monitoring takes place. The objective is to capture the final effluent. For time-weighted samples the recommended frequency for collecting equal aliquots of effluent is one (1) per hour over a 24-hour period.

With use of extremely sensitive instrumentation for the analytical PCB method, it is critical that new, solvent washed, certified clean bottles and Teflon tube be used at each outfall/sampling site. It is equally important that once the Teflon coated tubing is used for PCB sampling, that it not be used again for this purpose as residue in the tube may contaminate future samples. After solvent is pumped through the Teflon tubing and emptied to an appropriate waste receptacle, the ends should be sealed with Teflon tape or aluminum foil to ensure the interior of the tubing remains free of contamination. The containers receiving the effluent should consist of certified pre-cleaned amber glass bottles with Teflon lined lids. It is highly recommended the sample bottles be obtained from the analytical laboratory. For increased assurance the bottle(s) are not contributing to the background PCB concentration, the recommended guideline for bottle cleanliness is for detected PCB congeners to be no greater than ½ of the laboratory's reporting level (i.e.,ML or minimum level).

3.2 Instantaneous grab samples

Instantaneous grab samples are applicable to continuous and intermittent outfalls and should be collected at or as close as possible to the permit compliance monitoring point. For continuous flowing dischargers, one grab sample is acceptable although two or more are preferred. If two or more grab samples are collected, they are to be maintained as individual samples until combined under controlled conditions at the analytical laboratory. For purposes of this guidance, the collective grab samples are referred to as a "manual composite" and is an option available that more closely mimics an automated composite sample but at a reduced cost. If a manual composite is collected, the recommendation is to obtain equal aliquots of effluent over a 24-hour period at a frequency that can be repeated at exact time-based intervals (e.g., 2 samples - 12 hour intervals, 3 samples - 8 hour intervals, etc.). The volume needed for each aliquot is attained by simply dividing the total volume (2 to 4 liters) by the number of samples collected. Each aliquot shall be collected in a separate, appropriately sized certified pre-cleaned amber glass bottle with a Teflon lined cap, preferably provided by the analytical laboratory. The aliquots must be composited under controlled conditions by the analytical laboratory.

3.3 Quality Assurance Samples

Use of quality assurance (QA) samples such as field/trip blanks or duplicate samples are recommended but are not required. If compositing equipment is utilized, an equipment blank is required which can also serve as a field blank. Due to the expense of collecting twenty-four hour composites samples, it is suggested that duplicate samples be collected 1) in case the QA requirements of the method are not attained by the laboratory or 2) to serve as a back-up sample in case breakage occurs during sample shipment.

3.4 Sample Preservation
Sample preservation is limited to immediate chilling on ice to < 6°C. PCB samples may be stored up to one year in the dark at < 6°C.

4.0 General

All sampling should be performed in a safe manner using the proper equipment. Safety requirements shall be in accordance with the facility specific safety plan. Attachment C1 contains a list of supplies needed for the PCB sampling. Examples of field forms with specific procedures outlined for sample preparation, sample set-up, 24-hr composite/grab sample collection and post-sampling (24-hr composites) are contained in Attachment C2. To maintain and ensure the integrity of the sample, a chain of custody form should accompany samples during transport.

4.1 Site access shall be coordinated with facility staff when sampling is to be conducted by regulatory agency staff or contractors.
4.2 Field sampling personnel shall wear protective gloves to prevent direct skin exposure and to minimize sample contamination. Protective eye ware is recommended.
4.3 Establish sampling approach.
  4.3.1 If automated 24-hour composite samples are to be collected, follow sections 5.0 and 6.1 below.
  4.3.2 If grab samples are collected, follow section 6.2 below.
4.4 It is highly recommended that duplicate samples be collected in case the QA requirements of the method are not attained or sample breakage occurs during shipment.
  4.4.1 The duplicate samples can be submitted to the laboratory or kept on site provided the prescribed storage conditions are maintained until it has been determined the samples have met the specified QA.
4.5 As a potential cost reduction measure, batch analysis of samples is recommended. Samples may be stored in the dark for up to 1 year at < 6°C. Therefore, samples may be shipped separately or held until the desired number are collected for batch analysis. Specific arrangements should be part of the analytical laboratory agreement.
4.6 Field Sampling Records - See Attachment C2, Forms 1, 2 and/or 3.

5.0 Equipment Preparation/Cleaning (24-h Composite Samples)

Disposable equipment such as the peristaltic pump flex tubing and the Teflon coated tubing should be solvent washed using the specified volume of fresh pesticide grade solvent (i.e., methanol or ethanol). All non-disposable equipment such as the intake screener and utility knife, etc. should be cleaned prior to field activities and between sites. When samples are collected by a third party, assurance should be provided that the sampling equipment is PCB free. The following cleaning procedures are recommended:

5.1 Tube Washing (new Flex and/or new Teflon coated)
  5.1.1 Be sure the flex tubing is compatible with the solvent (acetone may degrade tubing).
  5.1.2 Attach the flex tubing to the Teflon coated tubing.
    5.1.2.1 Fresh pesticide grade ethanol or methanol should be pumped through the tubing at a rate of 4 liters per 100 feet.
    5.1.2.2 The solvent should be captured in a receptacle as it clears the tubing and disposed of in an approved manner.
    5.1.2.3 The tubing can be reused for purposes other than PCB monitoring as described in 3.1 above.
  5.1.3 Cover the tube ends with Teflon tape or the dull side of aluminum foil.
  5.1.4 Double bag the Teflon coated and flex tube coils in a large polyethylene bag. Enclose by tying a loose knot at the opening of the bag and repeat using the second polyethylene bag.

5.2 Teflon coated tubing <u>should not</u> be reused for PCB monitoring (with Method 1668).
  5.2.1 The flex tubing should be disposed.

5.3 Other non-disposable equipment (knife, effluent strainer, etc):
  5.3.1 Wash contact surface with liquidnox or other laboratory grade detergent.
  5.3.2 Rinse contact surface with tap water.
  5.3.3 Rinse contact surface with pesticide grade ethanol or methanol (if in the field contain rinsate for appropriate disposal).
  5.3.4 Rinse contact surface with PCB free DI water.
  5.3.5 Air dry equipment and wrap in aluminum foil and place in sealable plastic bag.

5.4 Clean/decontaminate the sampler(s)
  5.4.1 Clean the inside of the sampler(s) with laboratory grade soap.
  5.4.2 Rinse with PCB free DI water and either wipe dry with a paper towel or air dry
  5.4.3 Clean the inside of the automated sampler lid as in 5.4.1 above. The lid can be used to hold any clean equipment during the field set-up of the 24h-composite sampler.

6.0 Sample Collection

6.1 <u>Automated 24-hour Composite Samples</u>
  6.1.1 Applies only to continuous flowing municipal and industrial dischargers.
  6.1.2 Disposable Nitrile gloves or equivalent should be placed on hands prior to handling equipment.
  6.1.3 Remove flex tubing from double bags and assemble the solvent washed flex intake line through the pump housing (length will be dependent on machine used but is typically 3 feet).
  6.1.4 Remove from bag and insert the Teflon coated tube into the flex tubing (a clamp or zip tie may be needed to hold these hoses together).
  6.1.5 Cut the Teflon lined tube to the desired length (site specific) and place Teflon tape or aluminum foil on the loose end of the hose.

- 6.1.5.1 Ensure the loose end of the hose does not touch the ground (i.e., final 2 feet). Either hold or place in the lid of the sampler.
- 6.1.5.2 Seal off the end of the remaining hose and place back in bags and close with a tie.

6.1.6 Rinsate or Equipment Blank

- 6.1.6.1 PCB free DI water shall be run thru the flex tubing and Teflon coated tubing prior to effluent collection.
  - 6.1.6.1.a This step is required for each automated sampler used.
- 6.1.6.2 Remove the lid(s) from the certified clean sample bottle(s), wrap the lid(s) in foil and hold in a plastic bag.
- 6.1.6.3 Remove the Teflon tape or aluminum foil from the loose end of the Teflon lined hose and place in the bottle containing the PCB free rinsate water.
- 6.1.6.4 Pump 2 liters of rinsate water through the hose into the receiving bottle.
- 6.1.6.5 Cap the sample bottle, tag and place bottle on ice in cooler.
  - 6.1.6.5.a If an intermediate sample collection bottle is used, the rinsate sample shall be transferred to the sample bottle.
- 6.1.6.6 Record date, time sample collected, and machine used.

6.1.7 Attach intake strainer to Teflon coated intake tubing. If necessary, use a clamp or zip tie to secure.

- 6.1.7.1 Use of an intake strainer is optional (it is very important to ensure strainer is cleaned properly as this could be a source of contamination).

6.1.8 Pre-sample collection & calibration - Verify the sample delivery volume. (*note: specific information provided below is intended <u>only as guideline</u> for effluent collected hourly over a 24 hour period)*

- 6.1.8.1 Dispense 2 manual rinsate samples to waste.
- 6.1.8.2 For each automated sampler that is used, pump a pre-set aliquot (83 mls for 2 liters and 166 mls for 4 liters) into a 250-ml graduated cylinder (a second sampler may be used if an optional duplicate sample is collected).
  - 6.1.8.2.a Place the graduated cylinder on a level surface, read and record volume.
  - 6.1.8.2.b Follow this procedure 5-6 times to obtain an average volume.
  - 6.1.8.2.c The expected volume is 83 mls or 166 mls**.** If the average volume exceeds ± 5 ml from the expected volume, calibrate the automated sampler in accordance with manufacturer instructions. Record all calibration volumes per the field data sheets in Attachment C2.

- 6.1.8.2.d If a sample splitter is used to collect two samples (main sample along with a duplicate sample) from a single machine, configure the splitter in accordance with the machine specific instructions to ensure the appropriate volume is administered each pumping cycle.

6.1.8.3 Place certified clean amber bottle(s) or a certified clean intermediate collection bottle in the composite sampler(s); or two certified clean amber bottles in a single sampler if a sample splitter is used.

6.1.8.4 Remove bottle cap(s), wrap in aluminum foil and place in a plastic bag.

6.1.8.5 Feed the flex tubing through the sampler lid.

- 6.1.8.5.a May not be applicable if a splitter is used.

6.1.8.6 Insert the flex tubing into the bottle(s); then wrap a piece of Teflon tape or aluminum foil around the mouth of the bottle and the tube.

- 6.1.8.6.a Is not applicable if a splitter is used. If an intermediate collection bottle is utilized, every effort should be made to minimize the air/bottle opening interface.

6.1.9 Pack wet ice around the sample bottle(s) to ensure effluent will be chilled.

6.1.10 Start the sampling routine. Complete required field data sheets (Attachment C2, Form 1) to document the sample event.

6.1.11 Post sample collection

6.1.11.1 Remove sample(s) from composite sampler(s).

- 6.1.12.1.a. When an intermediate collection bottle is utilized, the sample shall be transferred to certified clean bottle(s).

6.1.11.2 Remove lids from plastic bag, remove foil and place on bottle(s).

6.1.11.3 Label sample(s) and place on ice in a cooler and chill to $< 6\ ^{o}C$.

6.1.11.4 Post sample collection – verify the sample delivery volume (this step is only necessary if there is a significant shortage or excessive collection of sample).

- 6.1.11.4.a Pump 2 manual samples to waste (do not collect).
- 6.1.11.4.b Pump an aliquot into a 250-ml graduated cylinder, place on a level surface, read and record volume per the field data sheets in Attachment C2.
- 6.1.11.4.c It may be necessary to re-set the sampler and collect another 24h composite sample particularly if there is a shortage in sample volume (e.g. shortage $\leq$ 1.9 L).

6.1.12 Sample Storage

- 6.1.12.1 Place sample(s) in a darkened refrigerator capable of maintaining < 6°C.

6.2 Grab Samples

- 6.2.1 Applicable to all discharger types including municipal, industrial process and industrial storm water.
  - 6.2.1.1 A single grab sample is acceptable for all discharge types.
- 6.2.2 Select a sampling point.
  - 6.2.2.1 The sample should be collected at or as close as possible to the permit compliance monitoring point.
  - 6.2.2.2 For outfalls where the final effluent is not accessible, the next upstream component of the treatment train can be utilized for sample collection.
    - 6.2.2.2.a Grab samples should not be collected from a faucet or an existing non-teflon lined pumping apparatus containing a new or existing hose that has been used for permit compliance monitoring for other parameters.
- 6.2.3 Manual Composites for non-storm water based effluents *(optional).*
  - 6.2.3.1 The number of grab samples to be used should be pre-established and the same collection procedure must be applied to the first and subsequent samples.
  - 6.2.3.2 The sample bottle size and associated volume collected shall be in accordance with the number of grab samples targeted.
    - 6.2.4.2.a Example: if 4 individual samples are to be collected, 500 ml certified pre-cleaned amber glass bottles are recommended for a final 2-liter sample. For 4-liter samples, 4 x 1,000 ml bottles are recommended.
  - 6.2.3.3 All grab samples are to be composited under controlled conditions at the analytical laboratory.
  - 6.2.3.4 Each sample should be carefully labeled so that it is clear to the analytical laboratory which samples are to be composited (e.g., ID #, 1 of 4; same ID#, 2 of 4, etc).
- 6.2.4 Storm event sampling associated with industrial activity.
  - 6.2.4.1 Storm water outfalls are limited to a single grab sample.
  - 6.2.4.2 Grab samples shall be collected during the first 30 minutes of the discharge, or within the 1st hr if sampling during the first 30 minutes is not practical.
  - 6.2.4.3 A grab sample shall be taken from the discharge when a storm event yields at least 0.1 inch of precipitation.
  - 6.2.4.4 The time interval from the preceding measurable storm event shall be at least 72 hours.
- 6.2.5 Disposable Nitrile gloves, or equivalent, should be placed on hands.
- 6.2.6 Remove the Teflon lined cap and temporarily place in aluminum foil.
- 6.2.7 Submerge the certified pre-cleaned glass bottle.

6.2.7.1 Grab samples are to be collected directly into a certified pre-cleaned glass bottle through immersion in wastewater or storm water either by using a rope (with a weight if necessary) or by submerging a hand held bottle.

6.2.7.2 The final sample volume should be as close as possible to the targeted $\geq 2 \leq 4$ liter volume (note: 4 liters is preferred).

6.2.8 Upon collecting the sample(s), immediately place the cap back on the bottle(s), label and place on ice in a cooler and chill to < 6°C.

6.2.9 Complete required field data sheets (Attachment C2, Forms 2 or 3) to document the sample event.

6.2.10 Sample Storage

6.2.10.1 Place sample(s) in a darkened refrigerator capable of maintaining <6°C.

6.3 Shipping Procedure

6.3.1. Once sampling has been completed, PCB samples should be packaged in bubble wrap to prevent breakage during shipping. The bottle caps may be sealed using Teflon tape if needed.

6.3.2. Coolers shall be packed with sufficient amounts of bagged wet ice to ensure the temperature is maintained at <6°C for shipment to the analytical laboratory.

6.3.2.1 The shipping coolers should be adequately packed with ice such that sample temperatures are maintained at <6°C when received by the laboratory.

6.3.2.2 It is recommended that Blue Ice not be used for maintaining the temperature as breakage during shipping could result in contamination.

6.3.3 Chain of custody forms shall be completed and sealed in a waterproof bag (e.g., zip lock bag) to accompany all samples from the time of initial generation through delivery to the analytical laboratory.

6.3.4 The samples can be transferred to an appropriate shipping agent for conveyance to the PCB analytical laboratory for processing and analysis.

7.0 Intake / Influent Sampling

The following minimal guidelines shall be met for consideration of using PCB data generated from source water for purposes of determining environmental background concentrations:

7.1 Time and date of intake, influent and effluent monitoring.

7.1.1 The collection of intake, influent and effluent samples must be within a representative time-frame to account for the lag time necessary for the intake water to move through the entire system.

7.1.1.1 Retention time of the facility treatment train and how that corresponds to the final effluent collection shall be described.

7.1.1.2 At a minimum, thorough documentation shall be provided for items such as intake sample collection date/time, effluent sample collection date/time, weather conditions, etc.

7.2 Pollutant concentrations of the intake, influent and effluent must be reported as congener- specific and total PCB.

7.3 PCB samples shall be collected in accordance with the requirements specified in this Appendix.

Attachment 1

Table 1. Supplies for PCB Sampling with automated samplers (per event)

ITEM	Num.	✓
1. Automated sampler (ISCO, Sigma, etc.)	1 or 2	
2. Power packs or batteries for automated sampler).	1 or 2	
3. Operating and Maintenance Manual for automated sampler	1	
4. Solvent rinsed ½” OD X 3/8” ID Teflon tubing (or other tubing depending on composite sampler requirements) with appropriate length of flex tubing – double bagged kit.	1	
5. Intake strainer (if used) – cleaned in accordance with section 5.0.	1	
6. Utility knife – cleaned in accordance with section 5.0.	1	
7 Certified clean (solvent rinsed) 2+L amber sample collection bottles (1 per sample plus 1 for each rinsate blank); or a certified clean intermediate collection bottle.	1+	
8. Laboratory generated PCB free DI water contained in certified clean amber bottle (2 Liters) for rinsate blank (1 blank needed for each machine)	1 or 2	
9. Optional – Laboratory generated PCB free DI water contained in certified clean amber bottle (2 Liters) for trip blank	1	
10. Ziplock bag for cap storage	1	
11. Aluminum foil (use dull side)	1	
12. 250 ml graduated cylinder for flow calibration of automated sampler	1	
13. Disposable Nitrile gloves, or equivalent	1 box	
14. Cooler w/ wet ice for holding rinsate sample and sample transport; also for chilling composite sampler	1	
15. Field data sheets and waterproof notebook	1 set	
16. Chain of Custody Sheets	2	
17. Black indelible ink pens	2	
18. Roll of Teflon tape	1	
19. Zip Ties	1 pack	

Table 2. Supplies for PCB Grab Sampling (per event)

ITEM	Num.	✓
1. Certified clean (solvent rinsed) amber sample collection bottles; the size of the bottle(s) will be dependent on the final targeted volume (2 or 4 liters) and the number of grab samples collected over a 24h period (e.g., 1 grab = one - 2 L (or 4 L), 2 grabs = two - 1 L (or 2 L), 4 grabs = four - 0.5 L (or 1 L), etc.)	1+	
2. Optional – Laboratory generated PCB free DI water contained in certified clean amber bottle (2 Liters) for trip blank	1 or 2	
3. Aluminum foil	1	
4. Disposable Nitrile gloves, or equivalent	1 box	
5. Cooler w/ wet ice for holding samples during transport	1-2	
6. Field data sheets and waterproof notebook	1 set	
7. Chain of Custody Sheets	2	
8. Black indelible ink pens	2	
9. Rope (and weight - if needed) for submerging sample container(s). If a rope is not appropriate for the type of sample container used, other tools or methods can be used for immersing the bottle provided the mouth of the bottle does not contact the apparatus.	1	

Attachment 2

Form 1 - PCB TMDL Effluent Sampling (24h Composite Samples)

Facility:		VPDES #:	Outfall:
1st Day Arrival Date /		1st Day Departure Date/	
2nd Day Arrival Date /		2nd Day Departure Date/	
Location of Sampler:			
Latitude :		Longitude :	
Weather:			
Sampling Information	Sampler Type / SNN # :		
Sample Begin Date /		Sample End Date / Time :	
Sample Event Completed	YES NO	Type of Event :	WET DRY
Comments:			
Sample Id:		Blank Sample Id:	
Auto Sampler Settings	Set Clock :	YES NO	
Bottle Size :	2 or 4 Liter	Intermediate bottle used?	
Suction Line ID, Type:	______ID, Teflon	Suction Line Length:	____ ft
Liquid Detector Enabled:	Yes		
Rinse Cycle:	0	Sample Volume Information	
Enter Head Manually:	NO	Sample Volume	83 (or 166) ml
Retry :	0	Actual Calibrate	
Program Mode:	Extended	2nd Volume :	
Purge Counts:	Pre-150 Post 250	3rd Volume :	
Time Paced :	YES	4th Volume :	
Uniform Time Intervals:	YES	5th Volume :	
Sample Every :	1 Hour	6th Volume (after	
Bottles Per sample Event:		Number Samples set to	_______
Sample Continuously:	NO	Actual Number	
Hydrograph Information (Wet Weather Samples)			
Amt. of rain w/in last 72h:		Amt. of rain on day	
% Influent inc. & time		Facility Retention time:	

Preparations for Sample: *(may require modification)*

	DATE: **TIME:**
	1. Decontaminate the sampler ☐ Spray the inside of the ISCO with soapy water. Wipe the inside of the sampler with a paper wipe. ☐ Spray with DI water to rinse the inside of the sampler. ☐ Dry the sampler with a dry paper towel. ☐ Clean inside the sampler lid first, which then can be used to hold any clean equipment during the sampler set-up.
	2. Install solvent cleaned pump tubing in the sampler pump and distributor. Make sure tubing extends ¼" past end of distributor arm.
	3. Attach the Teflon sample tube to the Automatic Sampler by pushing the Teflon tubing into the pump tubing at least 1 inch. Do not twist and check for kinks at the connection. Do not use black electric tape on either end of the tubing.
	4. Rinsate blanks ☐ Place rinsate receiver bottle with lid off in the sampler bottom. Wrap in foil and place lid in plastic bag. ☐ Make sure the sampler is set to pump to receiving bottle. ☐ Open lab water bottle. ☐ Put automated sampler together. ☐ Place Teflon lined tubing in lab water, use the "Pump forward" button (or similar) on sampler to pump through the sampler to the ground approximately 50 mls of water from the bottle marked "Lab water". Be sure to remove the tubing from the water when back flushing the tubing ☐ Pump all the water from the lab bottle through the automated sampler and into the receiving bottle. ☐ Cap sample bottle, and store in cooler with ice. ☐ Date & time rinsate sample is completed:______________________

Sample Set-up:

	5. Attach strainer to end of hose by twisting. Verify that opening is clear of stray pieces of Teflon. Attach sample tube in effluent to sample as close as possible to Plant NPDES sample location.
	6. Verify that all the Teflon tape has been removed from the sample hose.
	7. Verify the sample delivery volume. Dispense 2 manual samples to waste. Pump a sample into to a 1 gallon jar. Pour the sample into a graduated cylinder (250-ml), place on a level surface, read and record volume. **Expected volume 83 (or 166) ml. Actual volume ___________** If actual volume is ± **5** ml use the calibration function of the automated sampler. See automated sampler manual for details on how to calibrate the unit. Record all calibration volumes below. Expected Volume 83 or 166 Actual Volume_________ Expected Volume 83 or 166 Actual Volume_________ Expected Volume 83 or 166 Actual Volume_________ Expected Volume 83 or 166 Actual Volume_________
	8. Place the certified clean amber glass bottle(s) into the automated sampler at the proper position.
	9. Remove lid, wrap with foil and place cap into a zipper seal polyethylene bag. Place the flex tubing inside the sample bottle.
	10. Start the sampling routine
	11. Make any necessary field note entries into logbook.

NOTES: (Include weather conditions)

Following Sample Collection:

	DATE: **TIME:**
	1. Confirm that Automatic Sampler has completed 24-hour sample routine. Yes No
	2. Check the bottom of the automated sampler for any signs of liquid. Note in log.
	3. Replace the bottle lids. Put sample bottles in cooler.
	4. Download Automatic Sampler electronic log (if applicable): **File Name** ______________________
	5. Verify sample delivery volume. Dispense 2 manual samples to waste. Then, pump a manual sample into to a graduated cylinder (250-ml), place on a level surface, read, and record volume. **Expected volume** _____________**ml**. **Actual volume** _________________
	6. Remove the Teflon sample tube & stainless steel strainer and place into clean plastic bag.
	7. Put plastic bag around automated sampler.
	8. Make necessary field note entries into logbook.
	9. Transport samples in a cooler packed with ice, directly to location for shipment to the laboratory. Samples must be stored in the dark at <6°C until shipment.
	10. For shipment to laboratory, pack samples in a cooler using sufficient bagged wet ice to maintain chilled samples.

NOTES:

Weather Conditions:

Form 2 - PCB TMDL Effluent Sampling (Grab Samples)

(Not applicable to industrial storm water dischargers)

Facility:		**VPDES #:**	
Outfall:	**Description:**		

Event Information

Arrival Date / Time:		**Departure Date/ Time:**	
Sample Location Point			
Latitude :		**Longitude :**	
Weather:			

Sampling Information

Type of Event :	**WET DRY**		
Number of grab samples collected (over 24h):	**1 2 3 4 other**	**Sample container Size:**	
Approach used to collect sample(s):			
Comments:			
Sample Id:		**(Optional) Trip Blank Sample**	

Grab sample information

# Collected	**Volume (L)**	**Date/Time**
1		
2		
3		
4		
6		
other		

Location of "Manual Composite" sample mixing:	

Hydrograph Information (Wet Weather Samples)

Amt. of rain w/in last 72h:		**Amt. of rain on day sampled:**	
% Influent inc. & time observed:		**Facility Retention Time:**	

Form 3 - PCB TMDL Effluent Sampling for Industrial Storm Water Dischargers

Facility:		**VPDES #:**	
Outfall:	**Description:**		
Event Information			
Arrival Date / Time:		**Event begin Date / Time:**	
Sample Location Point			
Latitude :		**Longitude :**	
Weather:			
Sampling Information			
Grab sample collected within the first 30 mins. of storm event?		**Yes No**	
If no, was grab sample collected within the first hour of event?		**Yes No**	
Time sample collected:		**Grab sample volume:**	
Comments:			
Sample Id:		**(Optional) Trip Blank Sample**	
Rain Information (Wet Weather Samples)			
Date of last significant (>0.1 inch) rain event:		**> 72h?**	**Yes No**
Amt. of rain on day sampled:			

Appendix D
Virginia PCB TMDL Analytical Quality Control Requirements
(2/22/2009)

This document provides a summary of the analysis quality control requirements for the Polychlorinated Biphenyls (PCBs) analysis employing the most recent version of EPA Method 1668 for Virginia's Total Maximum Daily Load (TMDL) source identification studies. EPA Method 1668 is a Performance Based method. Project specific requirements stated in this QC summary must be met to ensure high quality data and cannot be modified without approval from DEQ.

With the reduction or elimination of interfering compounds and background contamination, the Estimated Method Detection Limits (EMDLs) and the Estimated Minimum Levels (MLs) presented in Method 1668 can be significantly improved upon. Examples of applied steps that have led to reduced MLs include increasing the volume of sample analyzed, baking glassware at a high temperature, increasing pre-post extract injection rinses, minimizing solvents used in extraction by keeping the sample at the appropriate nominal volume or by utilizing the proper glassware for sample extraction, etc. By improving technical procedures as described above it is plausible to attain EMDLs of ≤ 5 pg/L and MLs of ≤10 pg/L, both on a congener specific basis.

Quality Control Item	**Requirements and Frequency**	**Acceptance Criteria**	**Corrective Action**
Sample Collection, Preservation, Storage and Holding Times, Equipment Blank	Samples must be collected by following sampling guidelines included in Appendix C of the TMDL PCB Monitoring Guidance. A targeted volume of 4 liters is preferred. • Two (2) to four (4) liters (L) of aqueous sample will be collected for this project. It is recommended that a 2 or 4 L certified clean amber glass containers (or other appropriately sized bottles equaling 2 - 4 L when combined for use in a "manual composite") for aqueous sample collection originate from the analytical laboratory. • Documentation showing the traceability and cleanliness of the containers must be maintained at the laboratory. • <u>Bottle cleanliness guideline</u> – each PCB congener shall be ≤ ½ of the reporting level (i.e., Minimum Level). • It is recommended that a total of 2 - 4 L for the sample and 2 - 4 L for a duplicate be collected at each site. The duplicate is available in the event re-extraction and re-analysis is necessary due to failing QC.	• Samples should be stored at < 6°C until delivery to the laboratory. • If stored in the dark at < 6°C aqueous samples may be stored for up to one year. • If stored in the dark at ≤10°C, extracts may be stored for up to one year. • Certified clean amber glass bottle recommended guideline for cleanliness: Detected PCB congeners shall be no greater than ½ of the laboratory's reporting level. ▪ Equipment blanks shall meet the requirements in Attachment 1.B.	• There are no demonstrated holding times for PCBs although for purposes of TMDL monitoring, samples should not be held for more than one year. • Resample if able or deemed necessary for project DQOs. Otherwise, qualify results based on professional judgment if the potential for a low bias exists due to excessive holding times. Note: Laboratories should strive to meet the bottle cleanliness guideline since the intent is to ensure contamination does not originate from the sample containers.

Quality Control Item	**Requirements and Frequency**	**Acceptance Criteria**	**Corrective Action**
Sample Collection, Preservation, Storage and Holding Times **Con't**	• If an automated 24h composite sampler is used, the laboratory must supply 2 - 4 L of PCB free reagent grade Deionized (DI) water for use in collection of rinsate blanks. Two (2) -4 L of reagent grade DI water should also be provided by the laboratory for all field blanks. • If "manual composite samples" are collected, individual samples must be composited under controlled conditions in the laboratory.		
Aqueous Sample Extraction and Cleanup	Extraction and cleanup of the sample must use one of the techniques described in EPA Method 1668. • The entire contents of the 2 – 4 L sample volume must be extracted (do not separate solids if greater than 1% as described in Section 11.5 of the Method: extract the sample as is). The exact volume extracted must be documented and used in calculation of the sample concentration. All spike additions must be added to the 2-4 L bottle containing the sample prior to extraction. • Samples for "Manual composites" should be combined under clean conditions and spiked once combined. • It is preferred laboratories purchase glassware /extraction equipment to accommodate the larger 2-4 L sample volume (Note: the method typically extracts 1-L of aqueous sample). The laboratory may serially extract the 2-4 L volume in the same device one-liter at a time or extract two - four 1 L portions simultaneously in two different apparatus set-ups. Combine the solvent if serial or sequential extractions were performed prior to extract concentration and cleanup. **(Note: Serial extraction requires the use of greater volumes of solvent which may increase the level of background contamination).** • Sample extracts will be conc. to a final volume of 20 ul.	• Method blanks, OPR samples, labeled surrogate recoveries, field blanks or other QC samples must be processed identically to the samples including the same extract cleanups.	• Contact the client for guidance if the sample size or matrix does not allow these conditions to be met.

Quality Control Item	Requirements and Frequency	Acceptance Criteria	Corrective Action
Retention Time Calibration	• This project requires the use of the SPB-octyl column or comparable column. • Each diluted individual congener solution (Section 7.10.2 of the method) is injected to establish the beginning and ending retention times for the scan descriptions in Table 7. • Monitor the RT for all 209 congeners by injecting the diluted combined 209 congener solution (Section 7.10.2.2) every 12 hours with continuing calibration (Section 15). . • Co-eluting congeners must be reported according to the scheme defined in the Qualifier Codes for the project.	• The RT and relative RT (RRT) for all congeners must be within the windows in Table 2 of the method and the column performance specifications in Sections 6.9.1 of the method must be met. •The absolute RTs of the labeled toxics/LOC/window-defining standard congeners in the verification test must be within ±15 seconds of the respective RTs in the calibration.	• If the absolute RT of CB 209 does not meet criterion, the GC temperature must be adjusted and the test repeated until the minimum RT criterion is met. • Adjust chromatographic conditions and scan descriptors until all criteria are met. • Adjust system or replace GC column and repeat the verification test or recalibrate. NOTE: Laboratories with newer injection technology such as Electronic Pressure Control (EPC) may render the RT requirement for CB 209 obsolete. The RT and RRT for all congeners and co-eluting congeners must be documented at the same frequency as stated in this section for systems using EPC.
Mass Spectrometer Resolution	• The instrument is tuned using perfluorokerosene (PFK, or other reference material). • Static resolving power checks must be performed at the beginning and at the end of each shift.	A minimum resolving power of 1,000 for a significant PFK fragment in the range of m/z 300-350. The deviation between the exact m/z and the theoretical m/z (Table 7 - method) for each exact m/z monitored must be < 5 ppm.	Any problems must be corrected before analyses can proceed. Any samples in the previous shift that may be affected by poor resolution must be reanalyzed.
Ion abundance ratios and signal-to-noise (S/N) ratios	The low calibration standard concentration for this project must be 1.0 ng/ml. [If available, 0.5 ng/ml would be the preferred low calibration standard.]	The mass spectrometer must be adjusted and this test repeated until the m/z ratios fall within the limits specified If the adjustment alters the resolution of the mass spectrometer, resolution must be verified prior to repeating the test.	The mass spectrometer must be adjusted and this test repeated until the m/z ratios fall within the limits specified If the adjustment alters the resolution of the mass spectrometer, resolution must be verified prior to the repeat of the test.

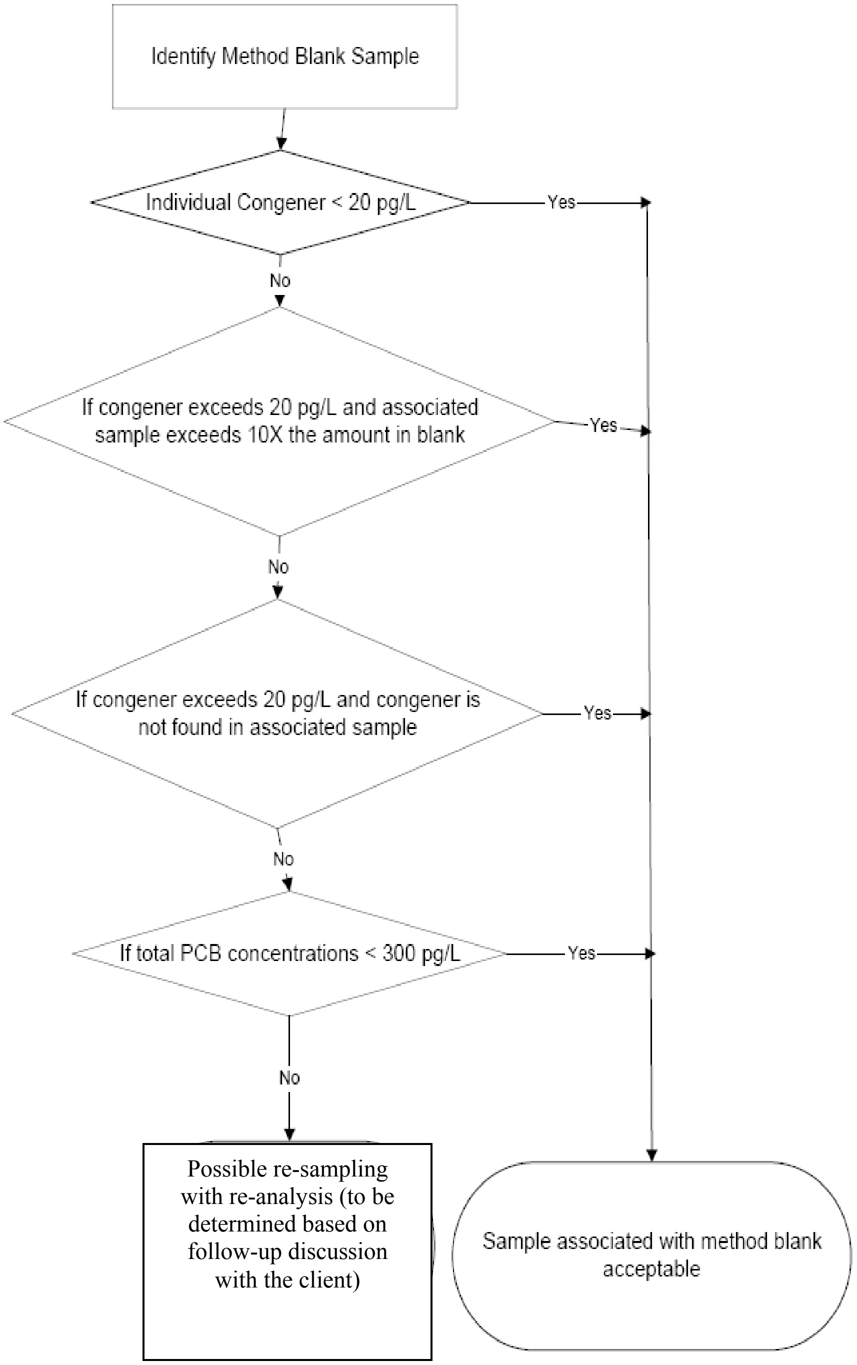
Quality Control Item	Requirements and Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	Established initially and when calibration verification fails criteria. Per the method, calibration is performed by isotope dilution and internal standard. • Calibration by isotope dilution is performed at a minimum of 5 (6 may be used) concentration levels for each of the toxic/level of chlorination (LOC) congeners (refer to Table 3 of the method). • The low calibration standard concentration for this project must be 1.0 ng/ml. (0.5 ng/ml is preferred). • Calibration by internal standard is performed for each native congener for which a labeled congener is not available, the labeled toxics/LOC/window-defining congeners, and the labeled cleanup congeners. For the native congeners, calibration is performed at a single point using the CS-3 standard. For the labeled congeners, calibration is performed using the data from the 5 (or 6) points in the calibration of the toxics/LOC congeners.	• %RSD≤20% among relative response (RR) for each native toxic/LOC congener in order to use the average RR (as calculated in Section 10.4.2 of the method). Otherwise, the complete calibration curve for that congener must be used over the calibration range. • Meet performance criteria specified in Table 6.	%RSD≤20% among relative response (RR) for each native toxic/LOC congener in order to use the average RR (as calculated in Section 10.4.2 of the method). Otherwise, the complete calibration curve for that congener must be used over the calibration range.
Calibration Verification	Performed at the beginning of each 12-hour shift during which analyses are performed. • The CS-3 calibration verification (VER) standard and the diluted combined 209 congener solution are analyzed.	• The m/z abundance ratios for all congeners must be within the limits in Table 8. • The GC peak representing each native CB and labeled compound in the VER solution must be present with a S/N ratio of at least 10. • The concentration of each compound must be within the limit in Table 6 of the method.	Adjust system, if necessary, and recalibrate. Criteria must be met before sample, blank, IPR, and OPR analysis may begin. • If the adjustment alters the resolution of the mass spectrometer, resolution must be verified prior to the repeat of the verification test.
GC Resolution and minimum analysis time	As part of calibration verification, the diluted combined 209-congener solution is analyzed.	Table 6 criteria for continuing calibration must be met.	Adjust GC analysis conditions until the specifications are met, or the column must be replaced and the calibration verification tests repeated or the system recalibrated. (See previous note concerning GC systems with EPC.)

Quality Control Item	**Requirements and Frequency**	**Acceptance Criteria**	**Corrective Action**
Mass Spectrometer Resolution	Beginning and end of each shift (Section 15.2)	• A minimum resolving power of **<u>10,000</u>** at m/z 330.9792 (Section 10.2.1). • The resolution must be $\geq$ 8,000 throughout the mass range (Section 10.2.3).	Samples affected by poor resolution should be reanalyzed.
Ongoing precision and recovery (OPR)	Prepared with each batch of samples (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples). • Analyzed prior the analysis of samples from the same batch.	The recoveries of the toxic/LOC CBs must be within the OPR limits given in Table 6 of the method.	If any individual concentration falls outside of the range, the extraction/concentration processes are not being performed properly. The problem must be corrected and the sample batch must be re-prepared, extracted, and cleaned up and the OPR test repeated.
Method Blank	Prepared with each batch of samples (samples started through the extraction process on a given 12-hour shift, to a maximum of 20 samples). • Analyzed prior to the analysis of samples from the same batch immediately following the analysis of the OPR. • The reference matrix must simulate, as closely as possible, the sample matrix under test. • When a reference matrix that simulates the sample matrix under test is not available, reagent water can be used to simulate water samples; playground sand or white quartz sand can be used to simulate soils; filter paper can be used to simulate papers and similar materials; and corn oil can be used to simulate tissues.	Method blanks must meet the decision rules specified in Attachment 1.A. of this Quality Control Requirements document (i.e., Appendix D).	If the method blank acceptance criteria are exceeded, analysis of samples must halt until the source of background contamination is eliminated or reduced. Extraction and analysis of duplicate samples is required. All samples must be associated with an acceptable method blank before the results for those samples may be reported; or the specific conditions preventing the ability to achieve the method blank acceptance criteria should be discussed with the client.

Quality Control Item	Requirements and Frequency	Acceptance Criteria	Corrective Action
Labeled Toxics/LOC/window defining standard spike	All samples must be spiked with labeled compounds to monitor method performance. The spiking of the extraction standards must occur prior to extracting the sample. The addition of the cleanup standards must occur before the fractionation, while the addition of the injection standards is conducted prior the GC/MS analysis.	The recovery of each labeled compound must be within the limits in Table 6.	If any labeled compound falls outside of limits, the method performance is unacceptable for that compound in that sample. Additional cleanup procedures must be employed to attempt to bring the recovery within the normal range. If the recovery cannot be brought within the normal range after all cleanup procedures have been employed, water samples are diluted and sediments, and other matrices are analyzed.
Qualitative/ Quantitative Issues	Identification of a CB or labeled compound in a standard, blank or sample occurs when all criteria are met. • Report results for all 209 PCB congeners adhering to the IUPAC nomenclature. • Report results according to specified Electronic Data Deliverable (EDD) found in Appendix E. Coeluting congeners shall be reported in accordance to the rules specified in Attachment D1. • Report results using data qualifying flags as included in Attachment 3.	The signals for the two exact m/z's in Table 7 must be present and must maximize within the same two scans. • The S/N for the GC peak at each exact m/z must be ≥ 2.5 for each CB detected in a sample extract, and ≥ 10 for all CBs in the calibration and verification standards. • The ratio of the integrated areas of the two exact m/z's specified in Table 7 must be within the limit in Table 8, or within ±15% of the ratio in the midpoint (CS3) calibration or VER, whichever is most recent. • The RRT of the peak for a CB must be within the RRT QC limits specified in Table 2, or if an alternate column or column type is employed, within its respective RRT QC limits for the alternate column or column system. • With congener overlap and the potential for interfering substances, it is possible that all of the identification criteria above may not be met. It is also possible that loss of one or more chlorines from a highly chlorinated congener may inflate or produce a less-chlorinated congener that elutes at the same retention time. If identification is ambiguous, an experienced spectrometrist must determine the presence or absence of the congener.	Congeners that are not detected are to be reported to the sample specific Estimated Detection Limit (EDL). EDLs must be calculated as described in described in Attachment 2 of this Appendix. • Estimated Minimum Levels (MLs) or reporting levels must be calculated as described in Attachment 2. • If a peak does not meet the qualitative identification criteria (most commonly the ion abundance ratio criteria), the quantitative result for that congener must be reported as an Estimated Maximum Possible Concentration (EMPC).

Attachment 1.A.

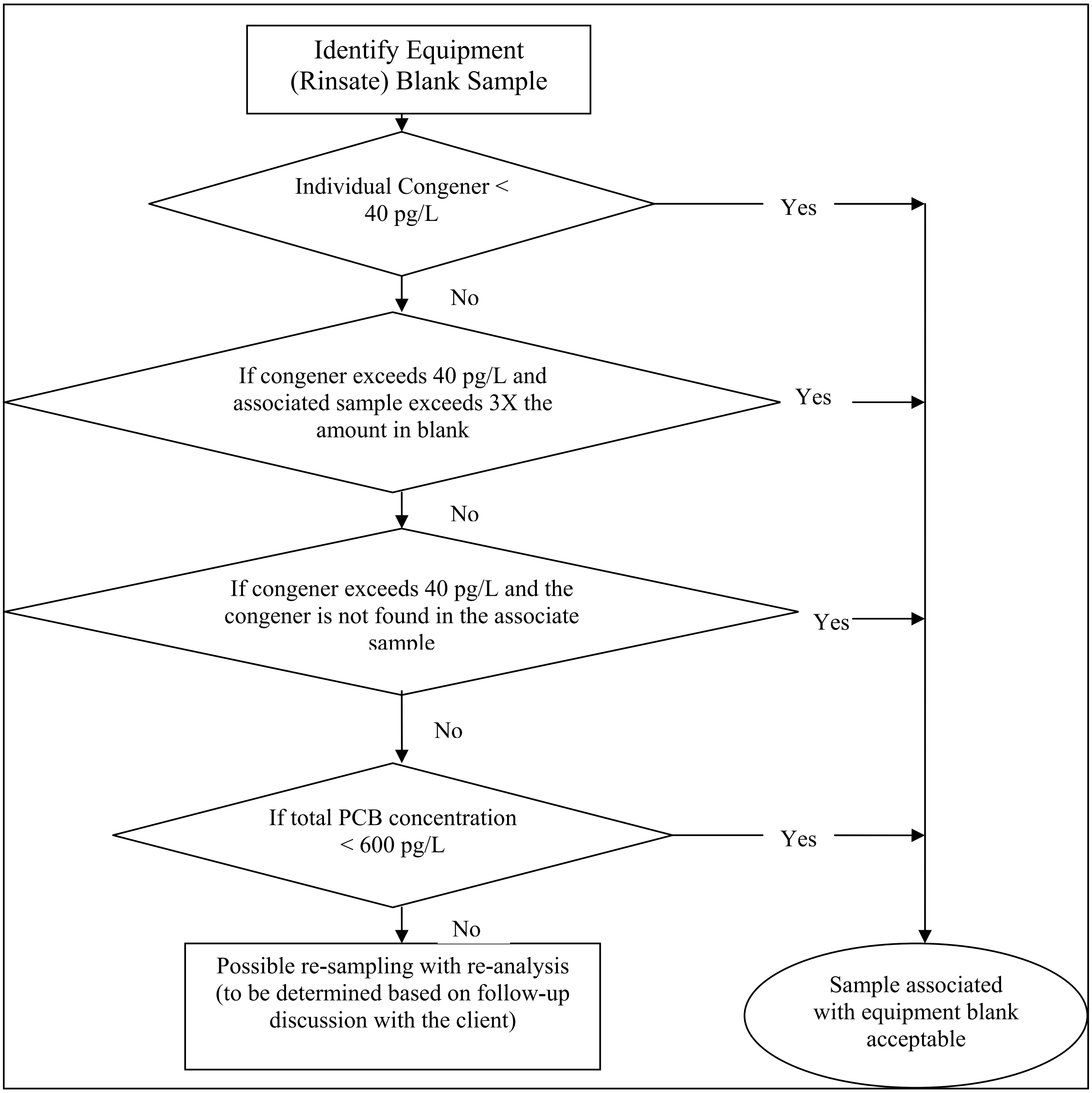
(based on Delaware River Basin Commission (DRBC) PCB monitoring)



Attachment 1.B.

(based on Delaware River Basin Commission (DRBC) PCB Monitoring)

Equipment Blank Contamination Decision Rules



Attachment 2

Estimated Detection Limit

For analyte ‘x’, the EDL is calculated by the following formula:

$$EDL_x = 2.5 \bullet \frac{(Na \bullet Qis \bullet Rah)}{(Ais \bullet RRF \bullet wv)}$$

Where:

Na = Analyte peak to peak noise heght.
Qis = Concentration of internal standard.
Rah = Area of Height Ratio
Ais = Area of internal standard
RRF = initial calibration average relative response factor for the congener of interest.
wv = sample weight/volume.
2.5 = Mimimum signal to noise ratio.

Noise calculations are to be taken from the discrete sections of the chromatogram rather than the entire chromatograph for mass descriptor. No peak smoothing of the chromatograph is to be undertaken. Peak identification is to be conducted on the raw chromatograph.

Estimated Minimum Level (ML)

For analyte ‘x’, the ML is calculated by the following formula:

$$ML_x = (1000\ pg/ng) \bullet (1\ ng/mL) \bullet (0.02\ mL)/4\ L = 5\ pg/L$$

Where:

1 ng/mL = low calibration standard
0.02 mL = sample extract volume
4 L = sample volume

The method sensitivity is not limited by the instrumentation but rather by interferences present in the sample or background PCB contamination levels. Decision rules have been established to minimize the impact of congener specific background contamination (see Attachment 1). If background contamination exceeds the established thresholds and compromises the results, it will be necessary to reanalyze a duplicate effluent sample once background contamination has been controlled.

Attachment 3

Data Qualifiers

Qualifier Flag		Description
J	-----	The reported result is an estimate. The value is less than the minimum calibration level but greater than the estimated detection limit (EDL).
U	------	The analyte was not detected in the sample at the estimated detection limit (EDL).
E	------	Exceeds calibration range.
D	------	Dilution data. Result was obtained from the analysis of a dilution.
B	------	Analyte found in sample and associated blank.
C	------	Co-eluting congener
Cxx	------	Co-elutes with the indicated congener, data is reported under the lowest IUPAC congener. 'Xx' denotes the IUPAC number with the lowest numerical designated congener.
NR	------	Analyte not reported because of problems in sample preparation or analysis.
V	------	Surrogate recovery is not within method control limits.
X	------	Results from reinjection/repeat/recolumn data.
EMPC	------	Estimated maximum possible concentration. Indicates that a peak is detected but did not meet all the method required criteria.

Total PCBs

For purposes of this guidance, Total PCB is defined as the summation of all detected PCB congeners. This includes "J" flagged congeners as well as those identified with "EMPC". PCB congeners flagged with a "U" are not included in the total PCB value.

Appendix E
Reporting Requirements for Analytical (PCB) Data Generated Using the most current EPA Method 1668

(1/12/2009)

Objective

The data reporting guidelines presented in this Standard Operating Procedure (SOP) are to ensure PCB results generated with the most recent version of U.S. EPA Method 1668 are electronically submitted to the Department of Environmental Quality (DEQ) with consistent content and format. Congener specific PCB data are to be generated based on uniform sample collection (Appendix C) and analytical requirements (Appendix D). Since different HRGC/HRMS laboratories will be providing data, it is essential that a consistent format be used for reporting PCB results. The specific formatting and reporting requirements of this appendix are applicable to sample collection personnel as well as the analytical laboratories generating the data. Personnel from the permitted facility will be responsible for ensuring PCB data are delivered in the requested format.

Electronic Data Deliverables (EDDs)

Data shall be delivered to DEQ in two electronic data formats to address aqueous sample collection and analysis associated with point source discharge samples. For the first reporting format, three Electronic Deliverable Data (EDD) files are required and include the following data deliverables: a sample "*Location*" file, a sample "*Chain of Custody*" file, and an "*Analytical Results*" file. Descriptive information for these three files can be found in Tables 1-3 and are available for download from the DEQ TMDL website (http://www.deq.state.va.us/tmdl/pcb.html). Each file consists of an Excel spreadsheet and includes data fields with embedded formatting rules and tabs for providing formatting definitions for each field.

1. Data for the "Location" and "Chain_Custody" EDDs are to be submitted in the Excel format specified in the file.
2. Laboratories must deliver analytical results which adhere to the formatting guidelines specified in the "Analytical_Results" EDD file as Quote Comma Separated Value (*.csv) files.

In addition to the three EDD tables, it is also requested that an electronic "hard copy" data report be provided to DEQ by the analytical laboratory generating the PCB data. The framework for this report, for which the order of presentation and content are provided below, partly mimics the US EPA Contract Laboratory Program Statement of Work (CLP SOW). The report is to be submitted in PDF format and shall include the following:

1. Cover Letter or Letter of Transmittal signed by the laboratory manager.
2. Sample Delivery Group (SDG) Narrative.
    a. Laboratory name.
    b. SDG number.
    c. Field identification and laboratory identification numbers for samples.
    d. Extraction method.

- e. Detailed documentation of any quality control, sample shipment, and/or analytical problems encountered in processing (preparing and analyzing) and corrective actions taken for the samples reported in the data package.
    - i. Include a glossary of qualifier codes used in the SDG.
    - ii. Include technical and administrative problems encountered, corrective actions taken and method of resolution, and an explanation of all flagged edits on quantitation reports.
- f. The SDG narrative must be signed and dated by the laboratory manager or other appointed laboratory personnel that reviewed and approved the release of the data.

3. Field Chain-of-Custody Records and Sample Receipt Documentation Log
    - a. Copies of field Chain-of-Custody Records for all samples within the SDG must be included in the deliverables.
        - i. Upon receipt at the laboratory, a description of the condition and temperature for samples from each cooler must be provided.
4. HRGC/HRMS PCB Data
    - a. Quality Control (QC) Summary with qualification or identification of compounds not meeting the criteria (applies to each sample batch and includes the submission of all PCB congener data).
        - i. Method Blank Analysis Summary
        - ii. Ongoing Precision and Recovery (OPR) Summary
        - iii. Duplicate Precision Summary
        - iv. Instrument Calibration Information (Initial and Continuing)
            1. Example calculation
            2. Information should be retained at the laboratory and only to be submitted if requested.
    - b. Sample Data
        - i. Analytical results summary (raw data) with reported concentrations and qualifiers.
            1. Use coeluting congener rules specified in Attachment 1.
            2. Includes homolog data summary.
        - ii. Estimated Method Detection Levels (EMDLs) and Estimated Minimum Levels (MLs), and concentration of target analytes.
            1. Provide example calculations as to how derived.
        - iii. Compound/Peak relative retention times (RRTs), peak co-elution information, ion abundance ratios (retained at laboratory and only to be submitted if requested).

Data Submission

Upon compiling the aforementioned electronic information, the three EDD Excel files and the “hard copy” data report (PDF file) should copied to a CD-ROM. All CD-ROMS should be submitted to DEQ using the Data Transmittal Form found as an attachment to this Appendix (Attachment 2).

Table 1. Electronic Data Deliverables for Location Information

DATA FIELDS	FORMAT	DEFINITIONS
Permit Number	Numeric	VPDES # (example VA0123456)
Location ID	Alpha-Numeric	VPDES # and outfall (example VA0123456-001)
Facility Name	Text	XYZ Inc.
Facility Designation	Text	Industrial/Municipal Major or Minor
Owner	Text	Legal entity responsible for facility, or State and Agency
Street Address	Text	Facility's Address
City	Text	City name
Zip Code	Numeric	5 digit zip code
Point of Contact	Text	Facility contact name and title
Contact Phone Number	Numeric	Phone number (example (804) 888-9500 ext 270)
Outfall	Numeric	outfall number (example 001)
Receiving Stream	Text	Stream that receives effluent discharge
Type of sampling location	Text	(LOOKUP TABLE) Type of sampling location; Industrial, Municipal, Tributary, Estuary (tidal mainstem), River (non-tidal mainstem), Air-Urban, Air-Rural
Latitude	Numeric	Decimal degrees of outfall located to an accuracy of ± 1 meter at 95% confidence level relative to NAD83. Please provide 6 digits after the decimal (example 40.123456). A conversion equation from degrees/minutes/seconds to decimal degrees is on the "Lat&Long Conversion" tab of this Excel Worksheet.
Longitude	Numeric	Decimal degrees of outfall located to an accuracy of ± 1 meter at 95% confidence level relative to NAD83. Please provide 6 digits after the decimal (example -75.123456). A conversion equation from degrees/minutes/seconds to decimal degrees is on the "Lat&Long Conversion" tab of this Excel Worksheet.
River Mile	Numeric	VADEQ River mile location if known. Please provide two digits after the decimal point (example 75.25)
Drainage Area	Numeric	Applicable to Storm Water discharges (in square miles). Please provide two digits after the decimal point (example 2.22)

Table 2. Electronic Data Deliverables for Chain of Custody Information

DATA FIELDS	FORMAT	DEFINITIONS
Location ID	Alpha Numeric	State abbreviation VPDES # and outfall (example VA0123456-001)
Location ID (second outfall)	Alpha Numeric	State abbreviation VPDES # and outfall (example VA0123456-002) if necessary
Sample_ID	Alpha Numeric	Consists of 9 digit VPDES #, outfall, sample type (e.g. Dry Weather), and collection date (Example: VA0123456-001-DW-02042008)
Sample_ID (second Outfall)	Alpha Numeric	Consists of 9 digit VPDES #, outfall, sample type, and collection date (Example: VA0123456-002-DW-02042008) if necessary
Sample_Date	MM/DD/YY	Date format example 02/04/08 (Date grab sample collected or end date for 24-hr Manual Composite or 24-hr composite samples)
Sample_Time	HH:MM	Time Format example 2:30 PM (Collection time for grab sample or end Time for composite samples)
Sample Collection Type	Lookup	(LOOK UP) Single Grab, Manual Composite, 24-hr Comp
Manual Composite Sample #	Lookup	(LOOK UP) The number of grab samples that form the 24-hr Manual Composite (2, 3, 4, 6, 8, other,NA=Not Applicable)
Weather category	Lookup	(LOOKUP TABLE) WW= Wet Weather, DW= Dry Weather, NA=Not Applicable
Sample Type	Lookup	(LOOKUP TABLE) Samples types: SA= Sample TB= Trip Bank, RB Rinsate Blank, INF=Influent Sample, OTH= Other
Sample_Matrix	Lookup	(LOOKUP TABLE) Water = water based sample including dissolved and suspended particulate fraction, Sediment = solids which are collected sub-aqueously, Soil = soils which are collected sub-aerially; sludge = solids collected as part of a WWTP process
Sample_Size	Numeric	Volume in Liters, Mass in grams
Sample_Units	Lookup	(LOOKUP TABLE) Units: Liters – L , grams - g
Precipitation (total amount for sampling event)	Numeric	Amount of precipitation to (e.g., 0.0 = no precipitation; 0.01")
Precipitation station	text	Location of precipitation station or gage used to determine amount of precipitation
Flow	Numeric	Total discharge from outfall during 24-hour event for continuous discharges or total flow during non-continuous event up to 24-hours (in MGD)
Notes	Text	Sampler's notes regarding conditions, description of collection methodology, identify previous rainfall event in excess of 0.1", any anomalies encountered
Analysis Requested	Lookup	(LOOKUP TABLE) 1668, Other
Laboratory Undertaking Analysis	Text	Laboratory name
Laboratory Address	Text	Street address (no P.O. Box addresses)
Laboratory Contact	Text	Laboratory contact name and title
Laboratory Contact Phone Number	Text	Phone number (example (609) 883-9500 ext 270)
Date sent to lab	MM/DD/YY	Date sample sent to lab Date format example 12/31/2004
Sampler	Text	Name of sampler
sampler affiliation	Text	Employer of sampler

Table 3. Electronic Data Deliverables for Analytical Information

DATA FIELDS	FORMAT	DEFINITION
Sample_ID	ALPHA NUMERIC	Sample ID from Chain of Custody
Lab_Sample_Id	TEXT	Laboratory Identification to be provided by Lab
Laboratory Name	TEXT	Name of Laboratory to be provided by Lab
Sample_Matrix	TEXT	(LOOKUP TABLE) Sample Matrix must match sample matrix from Chain of Custody. Water = water based sample including dissolved and suspended particulate fraction, Sediment = solids which are collected sub-aqueaously, Soil = soils which are collected sub-aerially; sludge = solids collected from the WWTP process
Percent_Moisture	ALPHA NUMERIC	Percentage of moisture content of sample (NA=Not Applicable)
Qc_Code	Lookup	(LOOKUP TABLE) SA = sample, QADU = duplicate, MB = method blank, OPR = spike, MS = matrix spike, MSD = matrix spike duplicate, CCV = cal ver
Sample_Date	DATE (MM/DD/YY)	Sample Date from Chain of Custody
Sample_Time	TIME (HH:MM)	Sample Time from Chain of Custody
Analysis_Perfomed	TEXT	Analysis performed on sample – EPA Method (to be provided by Lab)
Extraction Date	DATE (MM/DD/YY)	Date sample was extracted
Analysis_Date	DATE (MM/DD/YY)	Sample analysis Date (to be provided by Lab)
Analysis_Time	TIME (HH:MM)	Sample analysis Time (to be provided by Lab)
Sample_Size	NUMERIC	Volume or Weight of the sample (To two decimal places)
Size_Units	Lookup	(LOOKUP TABLE) Sample size units (L, mL, uL, g)
Initial_Cal_Date	DATE (MM/DD/YY)	Date the initial calibration was run (to be provided by Lab)
Instrument Id	TEXT	Instrument identification (to be provided by Laboratory)
GC Column Id	TEXT	Gas Chromatograph Column identification (to be provided by the Laboratory- Example SPB-Octyl, DB-1)
Test Type	TEXT	Type of test includes: Initial, Reextraction and Reanalysis, Other
Test Batch Type	Lookup	(LOOKUP TABLE) Lab Batch Type values include “preparatory or analysis”
Batch_ID	TEXT	Batch Id (to be provided by the lab)
Cal_Ver_Lab_Sample_ID	TEXT	Calibration Verification Lab Sample ID associated with the sample (instrument run id to be provided by Laboratory)
Method_blank_lab _sample_ID	TEXT	Method Blank Lab Sample ID associated with the sample (instrument run id to be provided by Laboratory)
Compound	TEXT	Enter Full chemical compound name
IUPAC_PCB_#	TEXT	IUPAC PCB number from Table 1 EPA Method 1668 Revision A. 1999, for labeled analogs identify with “L” Example “3L”,”77L”
CAS_#	TEXT	CAS number from Table 1 EPA Method 1668 Revision A. 1999
Concentration Found	NUMERIC	Analyte concentration found
Dilution Factor	NUMERIC	Numeric Dilution Factor applied to extract
UNITS	TEXT	Reporting units (PG/L, PCT_REC, PG/G_DRYWT, NG/ML, etc.)
Data_Qualifiers	Lookup	(LOOKUP TABLE) See Data Qualifier Tab on Spreadsheet
EDL	NUMERIC	Estimated Detection Limit See EDL Definition Tab on Spreadsheet
Minimum_Level	NUMERIC	Minimum Level as calculated by Lab
Conc_Lower_Limit	NUMERIC	Used for spikes and cal vers to show limits values are in percent recovery
Conc_Upper_Limit	NUMERIC	Used for spikes and cal vers to show limits values are in percent recovery

Attachment 1

Reporting Rules for coeluting congeners

1. If a congener coelutes with another congener, qualify the result with CXXX (where XXX is the lowest numbered target in the coelution). No value will be entered into the concentration field for CXXX.
2. The CXXX designation will be identified in the data qualifier flag column.
3. If the congener is the lowest numbered congener in a coelution, then it will be identified with a C in the data qualifier flag column.

Attachment 2

Virginia Department of Environmental Quality
PCB Data Transmittal Form

Date: ____________________________

Mail To:
TMDL Program
Virginia Department of Environmental Quality
P. O. Box 1105
Richmond, VA 23218

Please find enclosed ________ CD-ROM(s) containing PCB data submittal and accompanying supporting documentation for:

FACILITY NAME:____

VPDES NUMBER:______________________________________

We are submitting a total of ______ documents to satisfy the voluntary request to generate low level PCB data to determine the potential for contribution of PCBs from our facility for the period beginning on __________ and ending on ___________.

These documents are described as follows:

Name of Document	**File Name of Document on Disk**	**Size of Document**	**Date of Document**
Data "Hard Copy" Report (PDF)			
Location Table EDD (.xls)			
Chain of Custody EDD (.xls)			
Analytical Results EDD (*.csv)			

If any problems or questions should arise during DEQ's processing of this/these CD-ROMS, please contact:

Technical Contact:________________________ Phone Number:______________

Signature of Authorized Official:________________________ Date:_______________

Title of Authorized Official:_________________________________