California Regional Water Quality Control Board
San Francisco Bay Region

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Edmund G. Brown, Jr.
Governor

Sent Via Email

May 17, 2011

To: Attached Mailing List

Subject: Sampling Analysis and Reporting Protocols Using EPA Method 1668C for Final Order No. R2-2011-0012, NPDES Permit No. CA0038849

This letter requires that dischargers regulated by Order No. R2-2011-0012 follow the attached *POTW PCBs Sampling, Analysis, & Reporting Protocols Using EPA Method 1668C*, prepared by Bay Area Clean Water Agencies, in collaboration with the Regional Water Board, when conducting sampling, analysis, and reporting of PCBs with EPA Method 1668C. The purpose of these protocols is to ensure that dischargers collect and report data that are readily comparable.

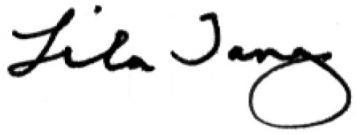
In reporting PCBs data, for dischargers reporting to the State Water Board's electronic reporting system (eSMR), or dischargers who will be reporting to eSMR, this letter allows them to upload PCB congener data simply as an attachment until sometime in 2012. In other words, dischargers may use the attachment tab of eSMR to upload laboratory data sheets that include data for all PCB congeners. Because we are only now phasing in eSMR for this region, this interim period will allow us to work out any issue that may exist with the upload of PCB congener data. We will notify eSMR dischargers once issues have been resolved and when eSMR dischargers will be required to switch from attaching data to uploading PCBs congener data in a retrievable format (i.e., as a data file).

For dischargers not participating in eSMR, this letter clarifies that they must provide PCB congener data with their routine self-monitoring reports. Sometime next year, we intend to require PCB congener data in electronic format in Microsoft Excel. Non-eSMR dischargers will receive notification of this requirement at that time.

Finally, there has been some confusion on sampling frequency requirements because Order No. R2-2011-0012 became effective on April 1, 2011. To clarify, for dischargers with annual sampling requirements, at least one sample must be collected between April 1 and December 31, 2011. For dischargers with semi-annual sampling requirements (where semi-annual is defined between January 1 through June 30, and July 1 through December 31), at least two samples must be collected in 2011.

If you have any questions regarding this letter, please contact Robert Schlipf at (510) 622-2478 or email him at rschlipf@waterboards.ca.gov.

Sincerely,



Lila Tang
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Attachment: Sampling Analysis and Reporting Protocols

MAY 16, 2011

BAY AREA CLEAN WATER AGENCIES

POTW PCBs Sampling, Analysis & Reporting Protocols Using EPA Method 1668C

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Summary

On March 9, 2011, the San Francisco Bay Regional Water Quality Control Board (Regional Water Board) adopted Order No. R2-2011-0012, *Amendment to Add PCBs Waste Discharge Requirements for Municipal and Industrial Wastewater Discharges of Mercury to San Francisco Bay, NPDES Permit No. CA0038849*. This Order includes requirements for monitoring and reporting of PCBs using both the approved EPA Method 608, and proposed EPA Method 1668C by Publicly Owned Treatment Works (POTWs). The following Sampling, Analysis & Reporting Protocols (Protocols) were prepared to provide a guidance document for POTWs and laboratories when generating data using EPA Method 1668C. They are intended as a supplement to the official EPA Method 1668C instructions (U.S. EPA, 2010), which, at this time, has not been confirmed as final nor promulgated as a rule by the U.S. EPA. Protocols for analysis and reporting using EPA Method 608 are not discussed in this document.

BACKGROUND

The February 2008 San Francisco Bay Basin Plan Amendment for the polychlorinated biphenyls Total Maximum Daily Load (PCBs TMDL) included a waste load allocation (WLA) for municipal wastewater dischargers of 2.0 kg/yr (kilograms per year). The estimated aggregate existing loading from municipal dischargers based on 2003 flow data is 2.3 kg/yr, but the final load for municipal wastewater dischargers was promulgated at 2.0 kg/yr by the Regional Water Board to incorporate expected reductions. The TMDL also splits the 2.0 kg/yr into individual POTW waste load allocations by dividing each POTW's flowrate by a group flowrate (grouped according to secondary and advanced-secondary treatment) and multiplying that value by the total POTW aggregate waste load allocation. The proposed individual and group allocations were developed from available PCB effluent concentration data for nine POTWs as presented in the PCBs TMDL Project Report (RWQCB, 2004). The data were collected using EPA Method 1668A from five POTWs with secondary treatment and four POTWs with advanced secondary treatment. A total of nine samples were collected over a three month period in 2000-2001 to characterize PCB effluent levels for secondary treatment and a total of fourteen samples were collected over a nine month period to characterize PCB effluent levels for advanced secondary treatment in 1999-2000. No data is available to characterize effluent quality from the remaining 30 facilities that received WLAs in the February 2008 TMDL.

IMPORTANCE OF COLLECTING ADDITIONAL DATA

A larger dataset (obtained from EPA Method 1668C analyses) may allow the Regional Water Board to assess variability in PCB monitoring data due to different treatment processes, diurnal patterns, flow changes (dry weather versus wet weather), and other factors. This variability will be useful in recalculating WLAs, or incorporating uncertainty factors when translating WLAs into NPDES permit limits.

Sampling Program

SAMPLING PROTOCOLS

The following protocols must be followed when collecting, holding, and transporting PCBs samples for analysis using EPA Method 1668C. The sample locations and sampling frequency are specified for POTWs in Order No. R2-2011-0012.

Sample Location

Samples shall be collected at the locations indicated as compliance monitoring points in the individual POTW NPDES permits (often, but not always, EFF-001).

Sample Collection

Samples shall be collected as 1-liter (L) grab samples in amber glass containers supplied by the laboratory[1] following applicable clean sampling techniques from EPA Method 1669 (U.S. EPA, 1996), such as the following, to reduce the likelihood of sample contamination:

- Wear clean, powder-free gloves
- Minimize time bottle is open
- Don't set lid down
- Don't allow rainwater to drip into bottle
- Don't touch inside of bottle/lid

Care shall also be taken to keep bottles dust-free prior to sample collection. At a minimum three 1-L grab samples shall be collected at each sampling location. Two of the grabs should be sent to the analytical laboratory and one sample should be retained until the analysis is completed and the data are accepted for reporting to the Regional Water Board. The extra sample (sent to the laboratory) will be for back-up purposes and general batch QC use by the laboratory. However, if a replicate analysis is desired, four 1-L grab samples should be collected and three of the grabs should be sent to the laboratory with instructions to analyze two of the bottles as replicates.

Sample Handling

If residual chlorine is present, each 1-L sample must contain 80 mg of sodium thiosulfate. Samples must be kept in the dark at less than 6°C from the time of collection until receipt at the laboratory. If the sample will be frozen, allow room for expansion. EPA Method 1668C allows samples to be held for up to 1 year prior to extraction if stored in the dark at less than 6°C.

Sampling Frequency

The effluent monitoring requirements for POTWs using proposed EPA Method 1668C are shown in **Table 1** (excerpted from Order No. R2-2011-0012). Major dischargers are required to sample more than once per year to ensure collection of samples during the wet and dry seasons. Minor dischargers may collect a sample anytime during the calendar year. All dischargers must

[1] Use bottles supplied by a laboratory with a EPA Method 1668C monitoring program to ensure no contamination is introduced by bottles.

follow Attachment G requirements (adopted as Order No. R2-2010-0054 and attached to NPDES permits) to collect effluent samples on days that are coincident with influent sampling and during periods of day-time maximum peak effluent flows.

Table 1. PCBs Monitoring Requirements for POTWs
(footnote numbering corresponds to Table E-2A in Order No. R2-2011-0012)

Parameter	Sample Type	Minimum Sampling Frequency[1]
Total PCBs (as congeners)[4]	Grab[3]	Quarterly for Major Dischargers with Design Flow > 5.0 mgd
		Semi-annually for Major Dischargers with Design Flow ≤ 5.0 mgd
		Annually for Minor Dischargers

1. Intermittent or seasonal dischargers shall collect samples during those months for which a discharge occurs. Major and minor discharge designations are indicated on each Discharger's individual permit and are also shown on Tables 1A and 1B of R2-2007-0077.
3. Grab Samples shall be collected coincident with composite samples collected for the analysis of other regulated parameters.
4. This monitoring is for informational purposes. Dischargers shall use USEPA Proposed Method 1668c and report the results for each of the 209 congeners. For congeners that co-elute, Dischargers shall report the sum of these congeners. A summation for Total PCBs is not required.

ANALYTICAL LABORATORIES

All samples shall be analyzed using proposed EPA Method 1668C (April 2010 version) to provide consistent concentration information for all 209 PCB congeners. Several analytical laboratories have the capabilities to analyze samples using EPA Method 1668C.

Dischargers must collect samples according to the above protocols and ensure sample temperature is less than 6°C during transport to the analytical laboratory. Sample bottles, shipping instructions, chain of custody templates, and billing information will be provided by the analytical laboratory.

Quality Assurance/Quality Control

Quality assurance and quality control samples are recommended in conjunction with PCB samples to verify data quality. In most cases, the volume collected for the grab samples and submitted to the laboratory (2 x 1-L) will allow for a backup sample and the random selection of samples for appropriate laboratory QA/QC. Collection of an additional sample as a field replicate is recommended during the first year or during the first two sample events. Even though analytical costs will be doubled, the field replicate results may be useful to determine sample variability. Field replicate analyses are recommended because PCBs are typically associated with solids and the results may be highly variable. Knowing the precision associated with a given sample is useful in terms of increasing the confidence of the concentrations reported to the Regional Water Board. Once this variability is known, replicate analyses may no longer be required.

Container Labeling

All samples must be identified with a unique identification code to ensure results are properly reported and interpreted. Samples must be identified such that the sampling location, matrix, and sample type (i.e., actual sample or QC sample) can be distinguished by a data reviewer or user. Container labels shall contain the following information:

- POTW Name
- Date
- Time
- Sample Number (alphanumeric code)
- Sampling Personnel
- Analytical Requirements
- Preservation Requirements
- Laboratory Conducting Analysis

Laboratory Controls

To ensure laboratories produce uniform data at known quality under low concentrations, the following quality control samples have been identified for each analytical batch:

1) Method Required Controls

   - Method Blank (as a batch contamination check)
   - Spike Blank (method recovery assessment)
   - Mid-Calibration Range Standards performed at the start and each and every 12 hours of instrument operation within a batch as per the method. An additional closing calibration verification, above that specified by the method is requested (so that calibration is maintained during the entire run.)

2) Recommended Controls

   - 10% of Samples Tested should be analyzed in duplicate (to assess precision in a sample matrix)

Additionally, chain of custody forms will ensure appropriate transfer and logging of samples.

Reporting

As specified in Order No. R2-2011-0012:

> *Each Discharger subject to PCBs effluent limitations of this Order shall submit PCBs monitoring data collected in its regular monthly or quarterly Self-Monitoring Reports (SMR) required in that Discharger's individual permit. These data shall include detection limits, reporting levels, estimated values, or quantified values for…all 209 PCB congeners using EPA Proposed Method 1668c.*

In the near future, the Regional Water Board will require all EPA Method 1668C data be submitted electronically. The following information is provided to generate a standard spreadsheet format for use by analytical laboratories, ensuring EPA Method 1668C data are reported consistently, and in a manner compatible with the California Integrated Water Quality System (CIWQS) Electronic Self-Monitoring Report module (eSMR2).

General requirements for data reporting and acceptability:

1) The Minimum Level of Quantitation reported for each congener for this analysis shall be the published Method Minimum Level (ML) (Table 2 of EPA Method 1668C). The laboratory analyzing the samples must have on file data demonstrating an ML, calculated as specified in 68 FR 11790 (published 3/12/2003, not yet approved by the EPA), that is equal to or less than the published Method ML. (Laboratories may revise MLs based on final published values, when EPA Method 1668C is fully approved and promulgated.)

2) The Method Detection Limit (MDL) reported for each of the congeners for this analysis shall be established by the laboratory using the MDL protocol described in 40 CFR 136 Appendix B. The MDLs reported by the laboratory shall be less than the ML (i.e., the laboratory must report 40 CFR Part 136 MDLs that are less than the reported MLs). If a laboratory determines Estimated Detection Limits (EDLs), these may be reported but only in addition to the MDL and the ML.

3) Specific qualifiers are available in CIWQS to describe analytical results or quality control issues. To report analytical results with qualifiers (i.e., values below MDL, less than the ML, co-elutions) specific, mandatory codes are available under the CIWQS Category of PARVQ. To report analytical uncertainties and quality control issues, another set or qualifiers is available under the CIWQS Category of QA CODE. The PARVQ and QA CODE qualifiers are listed and defined in **Table 2**. Dischargers may also attach laboratory-specific qualifiers to their data. However, these qualifiers are not available through the CIWQS drop-down menu and must be entered in the CIWQS "Comment" field. The analytical laboratory may also translate conditions into CIWQS-defined qualifiers when formatting data uploads to CIWQS or submit the information directly to the discharger in a separate report. A list of possible qualifiers that fall under this category is also provided in **Table 2**.

Table 2. PCBs EPA Method 1668C Qualifiers Defined in CIWQS or Applied by Analytical Laboratory

Qualifiers	Qualifier Description
Mandatory Qualifiers for CIWQS Reporting of Analytical Results (PARVQ)	
ND	The analyte was not detected in the sample at the method detection limit (MDL)
DNQ	The reported result is an estimate. The concentration is greater than the Method Detection Limit (MDL) but is less than the ML.
C	Co-eluting congener. This is the lowest numerically designated congener in a co-elution. Concentration of the co-eluting congeners is reported with this congener.
Cxxx	Co-elutes with the indicated congener. Concentration is reported under the lowest numerically designated congener. 'xxx' denotes the congener number for the lowest numerically designated congener in the co-elution.
Mandatory Qualifiers for CIWQS Reporting of Laboratory Quality Control Data (QA CODE)	
B	Analyte found in sample and associated method blank
U	Compound was analyzed for, but was not detected
R	Data rejected
J	Estimated value
BS	Insufficient sample available to follow standard QC procedures
GB	Matrix spike recovery not within control limits
IM	Method does not include this analyte as part of the compound lis
HR	Post digestion spike
BB	Sample > 4x spike concentration
GN	Surrogate recovery is outside of control limits
EB	Value is estimated
Additional Qualifiers for Analytical Laboratory Reporting[1]	
EMPC	Estimated maximum possible concentration. Indicates that a peak is detected but did not meet the Method's Theoretical Ion Abundance Ratio QC Limits.
D	Dilution data. Result obtained from the analysis of a dilution
V	Surrogate recovery is not within method control limits
X	Results from reinjection/repeat/recolumn data

[1] These qualifiers are not available for entry in CIWQS. If the analytical laboratory wishes to qualify data under these (or other) conditions, the information should either be translated into one of the mandatory CIWQS qualifiers or submitted in a separate report to the discharger. This is not a comprehensive list of all possible qualifiers. Additional qualifiers may be used by the laboratory.

4) When reporting data through CIWQS, only one qualifier can be reported per analytical result. The ND, DNQ, C, and Cxxx qualifiers have been selected for use in CIWQS reporting. The ND and DNQ qualifiers take precedence over all other qualifiers. ND must be reported when a value is less than the MDL. DNQ must be reported when a value is greater than or equal to the MDL but less than the ML. For co-eluting congeners, results shall be reported on the line of the lowest numeric congener and "C" shall be selected in the drop-down menu for qualifiers. For the higher numeric congeners that are part of this co-elution, the congener number for which the result is reported shall be selected in the drop-down menu. If the congeners co-elute, but the result is DNQ, "DNQ" shall be selected for the lowest numeric congener, and Cxxx shall be selected for the other congeners in that co-elution.

For example, when reporting a result that is part of a co-elution, the following convention shall be used:

Congener	Result	Qualifier
PCB 040	10	C
PCB 041	---	C040
PCB 071	---	C040

If the co-elution result is DNQ, the following convention shall be used:

Congener	Result	Qualifier
PCB 040	1.0	DNQ
PCB 041	---	C040
PCB 071	---	C040

5) Results shall be reported without blank-corrections.

6) Concentrations shall be reported in pg/L.

7) Options for handling replicate sample results: (1) If replicates are submitted to the laboratory as a single sample composed of multiple containers and instructions are provided to analyze the sample as a batch quality control duplicate, duplicate results do not have to be reported to the Regional Water Board. (2) If field replicates are submitted to the laboratory as separate samples (2 or more sample containers) and instructions are provided to analyze as separate samples, the duplicate results must be reported to the Regional Water Board.[2]

8) Consistent with Attachment G of NPDES permits, data should not be reported by an agency if data have been invalidated based on QA/QC results and analysis. If data are invalidated, it is recommended that documentation be prepared and filed internally to explain the process and reasons for invalidation. If data quality is discovered to be insufficient after the data have been reported to the Regional Water Board, the data invalidation procedure indicated in Attachment G should be followed.

[2] Handling replicate results (examples):

(a) Three 1-L containers are collected as grabs and submitted as a single sample (same location, date, time) to the laboratory without instructions. In this case, the laboratory will report a single result for that sample. The laboratory may or may not use the additional containers for batch QC purposes. Only the single sample result must be reported to the Regional Water Board.

(b) Three 1-L containers are collected as grabs and submitted as a single sample (same location, date, and time) to the laboratory with instructions to analyze one of the containers as a batch duplicate. The laboratory may charge for analyzing this duplicate and will report the result with the batch QC. The batch duplicate does not have to be reported to the Regional Water Board.

(c) Three 1-L containers are collected as grabs. One container is kept by the discharger for archiving. Two containers are submitted to the laboratory with instructions to analyze as separate samples. The laboratory will report two results and both results must be reported (individually) to the Regional Water Board.

PCBs Concentrations and Use of Qualified Data

PCBs shall be reported as individual congener concentrations with assigned qualifiers. A summation of individual congeners to produce a "Total PCBs" value is not required at this time. However, POTW reporting requirements could change under future permits and rules will be established regarding use of qualified data. At this time if a summation of Total PCBs is required by the Regional Water Board, or if summation is performed by any agency, data qualified with DNQ or ND should be set to "0" for the purpose of performing the summation. However, data qualified with B, C, EMPC, D, V, or X should be summed using the reported concentration.

Congener Naming Convention

Analytical laboratories and dischargers shall report PCBs congener results using the "Congener Number" (i.e., 1-209) as specified in Table 1 of EPA Method 1668C, available on-line at http://water.epa.gov/scitech/methods/cwa/upload/M1668C_11June10-PCB_Congeners.pdf. To ensure consistent recordkeeping and reporting formats, each congener shall be identified using the acronym "PCB" following by the three digit congener number as in PCB XXX (e.g., PCB 001, PCB 028, PCB 117).

The following congener names in Table 1 of EPA Method 1668C were found to be incorrect and should be changed to:

1) Congener 055 (74338-24-2) should be 2,3,3',4-TeCB and not 2,3,3',4'-TeCB
2) Congener 103 (60145-21-3) should be 2,2',4,5',6-PeCB and not 2,2',4,5,'6-PeCB
3) Congener 121 (56558-18-0) should be 2,3',4,5',6-PeCB and not 2,3',4,5,'6-PeCB
4) Congener 154 (60145-22-4) should be 2,2',4,4',5,6'-HxCB and not 2,2',4,4',5',6-HxCB
5) Congener 171 (52663-71-5) should be 2,2',3,3',4,4',6-HpCB and not 2,2'3,3',4,4',6-HpCB

Reporting Rules for Co-Eluting Congeners

- Report all congeners individually.
- The numeric result of the co-eluted congeners will be entered into the concentration field and reported as the congener with the lowest numerically designated congener number. The result will be qualified with a C. (Refer to General Rule #8 above for CIWQS procedures.)
- If a congener co-elutes, no value will be entered into the concentration field for the higher numerically designated congener. The "no value" result will be qualified with Cxxx (where xxx is the congener number for the lowest numerically designated congener in the co-elution). (Refer to General Rule #8 above for CIWQS procedures.)

Acceptance Criteria for Method Blanks

Method blank concentrations should generally be less than the MDL. Data can be invalidated by a discharger if the total concentration of the method blank is greater than the total concentration of the MLs. When summing the total concentrations for the method blanks and the samples, the individual congener concentrations must be counted as zero if less than the ML.

When a method blank is above this criterion, the discharger should submit the stored back-up sample to the analytical laboratory for re-analysis. If the analysis cannot be completed before a Self-Monitoring Report is required, the discharger shall address the situation in a cover letter to the Regional Water Board. The discharger should indicate sampling was completed, but analytical results are not yet available due to method blank contamination. If similar issues are encountered with the re-analysis, the data should still not be reported and an internal data invalidation should be prepared as indicated above, with a similar note in the subsequent applicable Self-Monitoring Report.

Sampling Program Objectives

The objective of these Protocols is to produce and report data that represent as closely as possible actual concentrations of PCBs in POTW effluent. This objective will be achieved, in part, by using established methods for sample collection and laboratory analysis, accepted QA/QC procedures, and consistent reporting practices. The ability to meet this objective will be accomplished by evaluating the resulting laboratory measurements in terms of reporting limits, precision, accuracy, representativeness, comparability, and completeness.

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