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UPDATE ON THE BENEFITS OF PCB CONGENER-SPECIFIC ANALYSES

Ecological Risk Assessment Support Center
Office of Research and Development
U.S. Environmental Protection Agency
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NOTICE

Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Note Relating to PCB Congeners by Low-Resolution GC-MS - Method 1628 (Not yet approved).

The Office of Water of EPA is in the process of developing a low-resolution GC/MS method for congener specific PCB analysis: [Method 1628. Polychlorinated Biphenyl \(PCB\) Congeners in Water, Soil, Sediment, Biosolids, and Tissue by Low-Resolution GC/MS using Selected Ion Monitoring](#) (EPA 821-R-21-002). The present document (*Update on the Benefits of PCB Congener-Specific Analyses*) was drafted while method 1628 was under development. The benefits of method 1628 are a) improved sensitivity over Methods 8082A and 608.3, b) total PCBs determined from the sum of the individual PCB congeners and not from the sum of Aroclors, and c) lower analysis costs in comparison to Method 1668C because of the use of low-resolution mass spectrometry instrumentation. Methods 1628 and 1668C are likely to provide similar total PCB determinations; however, for sites where the total dioxin toxicity equivalence (TEQ) of the PCBs are of interest, Method 1668C is likely to provide greater accuracy due to its ability to eliminate interferences from higher homologues for the dioxin-like PCB congeners.

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Background:

In April 2020, the Ecological Risk Assessment Forum (ERAF) submitted a request to the Ecological Risk Assessment Support Center (ERASC) to provide a review of ERASC [*Memorandum: Response to Ecological Risk Assessment Forum Request for Information on The Benefits of PCB Congener-Specific Analyses*](#) NCEA-C-1315, ERASC-002F, developed in March 2005. Specifically, a review was needed to 1) ensure that the memorandum reflects the latest state of the science, and 2) update the information as necessary.

Results of analyses for PCB contamination on environmental matrices may be expressed in terms of PCB congener-specific, total PCB, and Aroclor equivalent concentrations. Given the cost ramifications and potential overlap in results from each analysis, should expressing results in terms of all three types of analyses be our standard approach? The product of this work, a few-page memo updating the information in the 2005 ERASC publication, is designed to assist risk assessment practitioners to choose, in a cost-efficient manner, analyses that meet the objectives of the assessment.

Review:

After reviewing the 2005 ERASC [*Memorandum: Response to Ecological Risk Assessment Forum Request for Information on The Benefits of PCB Congener-Specific Analyses*](#), three updates are needed:

- 1) Toxicity Equivalence Factors (TEFs) in Table 4 have been updated.
- 2) Method 1668A migration to Method 1668C
- 3) Discussion of chemical bioavailability

Update 1: Revised Toxicity Equivalence Factors (TEFs)

In 2006, the World Health Organization updated their mammalian TEFs for PCDD/Fs and PCBs [1,2]. The updated/revised mammalian TEFs are provided in Table 1.

Table 1. Original [1] and revised [2] World Health Organization Toxicity Equivalence Factors (TEFs) for Mammals. Values for Birds and Fish were not changed.

Congener	TEF Mammals [1]	Revised TEF Mammals ^a [2]	TEF Birds [1]	TEF Fish [1]
Chlorinated dibenzo-p-dioxins				
2378-TCDD	1	1	1	1
12378-PeCDD	1	1	1	1
123478-HxCDD	0.1	0.1	0.05	0.5
123678-HxCDD	0.1	0.1	0.01	0.01
123789-HxCDD	0.1	0.1	0.1	0.01
1234678-HpCDD	0.01	0.01	0.001	0.001
OCDD	0.0001	0.0003	0.0001	0.0001
Chlorinated dibenzofurans				
2378-TCDF	0.1	0.1	1	0.0001
12378-PeCDF	0.05	0.03	0.1	0.05
23478-PeCDF	0.5	0.3	1	0.5
123478-HxCDF	0.1	0.1	0.1	0.1
123678-HxCDF	0.1	0.1	0.1	0.1
123789-HxCDF	0.1	0.1	0.1	0.1
234678-HxCDF	0.1	0.1	0.1	0.1
1234678-HpCDF	0.01	0.01	0.01	0.01
1234789-HpCDF	0.01	0.01	0.01	0.01
OCDF	0.0001	0.0003	0.0001	0.0001
Non-ortho-substituted PCBs				
PCB-77 3,3',4,4'-TeCB	0.0001	0.0001	0.05	0.0001
PCB-81 3,4,4',5-TeCB	0.0001	0.0003	0.1	0.0005
PCB-126 3,3',4,4',5-PeCB	0.1	0.1	0.1	0.005
PCB-169 3,3',4,4',5,5'-HxCB	0.01	0.03	0.001	0.00005
Mono-ortho-substituted PCBs				
PCB-105 2,3,3',4,4'-PeCB	0.0001	0.00003	0.0001	0.000005
PCB-114 2,3,4,4',5-PeCB	0.0005	0.00003	0.0001	0.000005
PCB-118 2,3',4,4',5-PeCB	0.0001	0.00003	0.00001	0.000005
PCB-123 2',3,4,4',5-PeCB	0.0001	0.00003	0.00001	0.000005
PCB-156 2,3,3',4,4',5-HxCB	0.0005	0.00003	0.0001	0.000005
PCB-157 2,3,3',4,4',5'-HxCB	0.0005	0.00003	0.0001	0.000005
PCB-167 2,3',4,4',5,5'-HxCB	0.00001	0.00003	0.00001	0.000005
PCB-189 2,3,3',4,4',5,5'-HpCB	0.0001	0.00003	0.00001	0.000005

^a Revised values are in bold font.

Update 2: Method 1668A migration to Method 1668C

EPA has updated Method 1668A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS [3] twice since the publication of the 2005 ERASC Memorandum. With Revision B [4], the key change was updating the quality control (QC) acceptance criteria based upon validation studies of the method. The revision also pointed the user to the updated mammalian TEFs [2]. With Revision C [5], the QC acceptance criteria were revised slightly. With both revisions, minor clarifications and corrections for typos in the method were provided.

With all three versions of the method, reports contain:

- Concentrations of the 12 polychlorinated biphenyls (PCBs) designated as the most toxic by the World Health Organization (WHO): congeners 77, 81, 105, 114, 118, 123, 126, 156, 157, 167, 169, and 189.
- Concentrations of the remaining 197 PCBs, approximately 125 of which are resolved adequately on an SPB-octyl gas chromatographic column to be determined as individual congeners. The remaining approximately 70 congeners are determined as mixtures of isomers (i.e., co-eluting isomers).
- Estimates of homolog totals by level of chlorination (LOC), and estimates of total PCBs in a sample by summation of the concentrations of the PCB congeners and congener groups.

Update 3: Discussion of chemical bioavailability

In the 2005 ERASC Memorandum, there is a fair amount of discussion on the weathering of PCBs from the standpoint of PCB mixtures in environmental samples not resembling the commercial Aroclor mixtures. Clearly, environmental fate and transport processes change the composition of the mixtures after their release into the environment. There is also some discussion of the impacts of bioaccumulation processes on the patterns of PCB congeners in biota where higher chlorinated congeners bioaccumulate more than lower chlorinated PCB congeners, and some congeners are biotransformed more quickly than others. Since the publication of the 2005 ERASC Memorandum, much has been learned about the partitioning behavior of

PCBs and the importance of different types of organic carbon in sediments and soils. These other organic carbon phases, beyond that arising from the diagenesis of plant materials, are black carbon (e.g., soot, chars, charcoal, coke, lampblack) and coal. When present, black carbon dramatically lowers the bioavailability of the PCB congeners in sediments and soils [6]. Therefore, when present, these other phases will further alter compositions of the congeners accumulated in biota. One should note that the addition of activated carbon phases to sediments have been evaluated as remedial options at some contaminated sediment sites [7]. The addition of the activated carbon lowers bioavailability of the contaminants, and subsequently, lowers the residues in the biota exposed to the sediments, either directly or indirectly via their diet. Most advances in understanding bioavailability of PCBs arises from the use of passive samplers to directly measure the bioavailable PCBs in sediments and if further details on passive sampling are needed, the reader should consult US-EPA (2012) [8].

Discussion of ERASC Request No. 22 Question

The 2005 ERASC Memorandum recommends "... results of analyses for PCB contamination in environmental matrices be expressed in terms of PCB congener-specific, total PCB, and Aroclor equivalent concentrations."

The question in the review request is "Given the cost ramifications and potential overlap in results from each analysis, does the recommendation really reflect what should be our standard approach?"

Response:

EPA's congener specific Methods 1668A, 1668B, and 1668C (henceforth referred to as Method 1668C) reports concentrations of PCBs on a congener-specific basis, by total PCB, and by homologue totals. In addition, concentrations of the 12 polychlorinated biphenyls (PCBs) designated as toxic by the World Health Organization (WHO) and their total dioxin toxicity equivalence (TEQ) are reported. Note, the TEQ is computed by summing, for all 12 congeners, the products of their individual concentrations and individual TEF values. Method 1668C does not report concentrations on an Aroclor basis. However, as described later

in the document, Aroclor equivalent concentrations can be calculated from the congener-specific PCB concentrations. Method 1668C requires sample cleanup and analysis using high resolution gas chromatography with high resolution mass spectrometry detection. Minimum levels of detection for sediments and soils are approximately 1-2 ng/kg-dw per congener and rise to 7.4 ng/kg-dw for several co-eluting congeners [5].

SW-846 Method 8082A [9] provides quantifications of total PCB based upon Aroclors. In the method, Aroclors most representative of the PCBs in the sample are determined and are subsequently used to quantify the amount of PCB as Aroclors in the sample. Identification of the representative Aroclors can be problematic and their determination depends upon the experience of the analyst, complexity of the samples from the site, technique used to identify the composition, and the degree of similarity of the PCBs in the sample to Aroclors as sold. Once identified, individual Aroclors are quantified using 3 to 5 characteristic peaks in the mixture and one of the characteristic peaks is considered “unique” to the individual Aroclor. The quantifications from the 3 to 5 characteristic peaks in each Aroclor are averaged to yield a reported concentration for the individual Aroclor and the sum of the quantifications for the individual Aroclors yields total PCB. Depending upon Aroclors selected as being representative and the characteristic peaks used by the laboratory, analyses by different laboratories can yield different compositions of the Aroclors. For example, one laboratory may report a total PCB concentration of XX ug/kg with AA:BB composition of Aroclors 1242:1254 while another laboratory might report total PCB concentration of YY ug/kg with CC:DD:EE composition of Aroclors 1242:1248:1254. Further, there are cases where one laboratory reports the PCBs in the sample as Aroclor 1242 and another laboratory reports the PCBs as Aroclor 1248. Method 8082A uses gas chromatography (GC) with electron capture detection (ECD) and is subject to interferences from other contaminants including organochlorine pesticides, phthalates, and other chemicals as well as some sediment constituents (e.g., sulfides). When PCBs are weathered such that the Aroclor mixtures (as sold) don't match the sample's actual pattern, accurate assignment of the representative Aroclor(s) can become challenging [10-12]. Minimum levels of detection for sediments and soils are typically 50-70 ug/kg per Aroclor using Method 8082A [10].

Depending upon what is selected when ordering analyses with Method 8082A, analyses may be performed with or without sample cleanup. Without sample cleanup, the sample is simply extracted with an organic solvent and the extract concentrated to volume that enables analysis with GC/ECD. With sample cleanup

(see SW-846 3600 methods [13]), the sample is extracted, cleaned-up by removing interferences, and then, analyzed using GC/ECD. Data from Method 8082A with cleanup will be more robust and dependable than those from the method without cleanup. Additionally, lower detection limits will be available when sample cleanup is performed.

Uncertainties in the analytical results are larger with Method 8082A in comparison to Method 1668C due to several factors. First, Method 1668C measures mass of the individual PCBs accurately, i.e., minimum resolving power of 10,000 (10% valley) at mass 330.9792 (permits measurement of ions with of mass 330.9461 with 10 % overlap in responses). This level of resolution eliminates interferences from chemicals such as chlorinated pesticides, brominated organics, chlorinated diphenyl ethers and other chemicals because of their differences in mass from PCBs. In contrast, Method 8082A uses ECD detection and all halogenated organics respond strongly and yield interfering responses in the quantification data. Second, Method 1668C has much lower detection limits in comparison to Method 8082C, and Method 1668C, with lower detection limits provides more certainty in the results especially when concentrations become low. Third, there is no need to determine the representative Aroclors in the sample with Method 1668C because the quantifications are based upon responses of the individual congeners. With Method 8082A, selection of representative Aroclors can be problematic and can be a source of uncertainty. Fourth, with Method 8082A, if the representative Aroclors don't exactly match the PCB pattern in the sample, there will be some inaccuracy in the quantification. From Table 9 in Method 8082A, accuracy in quantification of soils spiked with Aroclor 1254 at 50 ug/kg ranged from 38% to 144% of the spiked level.

As discussed by Erickson [11] and in the method itself [9], Method 8082A provides quantifications of total PCBs and the representative Aroclors used in the quantification. Method 8082A should not, except in some extremely rare cases, be used for source identification or for forensic purposes at contaminated sites [11, 14, 15].

The recommendation in the 2005 ERASC Memorandum states: "It is recommended that the results of analyses for PCB contamination in environmental matrices be expressed in terms of PCB congener-specific, total PCB, and Aroclor equivalent concentrations". The recommendation does not inherently require analysis in the laboratory by a method that quantifies PCBs in the samples on an Aroclor basis but rather an expression of "Aroclor equivalent concentrations". Aroclor equivalents could be determined by post-

processing of the laboratory data using the compositions of the Aroclor mixtures (see Frame et al [16, 17] for composition of Aroclors) and some type of regression/pattern matching approach to yield total PCBs on an Aroclor basis. This type of determination could be performed on a congener basis or homologue basis (Note, Table 3 in the 2005 ERASC Memorandum provides homologue compositions of 5 Aroclor mixtures). Currently, this type of post-processing is not done. An example of this type of processing is provided in Appendix A of this document.

From a cost perspective, Method 1668C when performed for all congeners is the most expensive analysis. Other options are available depending upon laboratory abilities and these include limiting the Method 1668C analyses to just the 12 PCBs with dioxin-like activity or to the homologue totals for mono- through deca-homologue groups. The latter two analyses will be less expensive, e.g., $\frac{1}{2}$ to $\frac{2}{3}$ the cost of the complete Method 1668C for all congeners. Analyses by Method 8082A will be the cheapest, and analyses without cleanup being lower in cost in comparison to those with cleanup.

The major differences between Methods 8082A and 1668C are 1) minimum detection levels and 2) accuracy of the total PCBs quantifications. For samples with concentrations greater than approximately 1 mg/kg total PCBs, Methods 8082A with sample cleanup and 1668C will provide similar quantifications of total PCB. When concentrations become less than approximately 1 mg/kg, Method 1668, with its much lower detection limit, is superior to Method 8082A and will provide better accuracy in the quantification of total PCB.

Because of the cost differences and time for results to be reported by laboratories, can defensible correlations between total PCBs determined by Methods 8082A and 1668C be developed? Yes, a defensible correlation at most sites can be developed. With the correlation, one could potentially have a majority of analyses performed using the less expensive method, i.e., Method 8082A, and then, adjusted using the correlation to total PCBs determined using the Method 1668.

To develop a correlation between total PCBs by Methods 8082A and 1668C, one would need run sufficient analyses with both methods to develop a strong relationship. The correlation/relationship will be site specific and will need to be developed for each site. Paramount in developing the correlation is that the mixture of PCBs across the site is relatively constant, and for most sites, this will be true. In Appendix B, a detailed

discussion is provided using data from Portland Harbor Superfund site on developing a correlation/relationship between total PCBs measured using Methods 8082A and 1668C.

The above discussion has been focused on EPA's Methods 8082A and 1668C. However, site managers/risk assessors have the flexibility to use other analytical methods for PCB analysis on Superfund site samples. The critical points in using other methods are that they need to be consensus-based, e.g., ASTM, EPA, Standard Methods, or European HORIZONTAL [18] official methods. These methods must provide appropriate and applicable QA/QC, respond to documented DQO needs, undergo interlaboratory validations/round robin trials, formal peer review, and be officially approved and published. Methods using benchtop GC/MS systems instead of high-resolution GC/MS systems like Method 1668C could easily be developed and provide data with more accuracy and reliable results in comparison to Method 8082A.

Conclusions

At Superfund sites, use of Method 1668C is **recommended on samples used in a site's risk assessment**. It is not a requirement that all samples be analyzed using Method 1668C but enough samples need to be analyzed to adequately perform the risk assessment and characterize the PCB congener distribution at the site. As discussed above, Method 1668C provides congener level data for the 12 PCBs designated as toxic by the World Health Organization (WHO) and allows determination of the dioxin PCB toxic equivalents (TEQ_{PCB}) using their TEFs. Method 1668C also provides total PCBs values. With these data, risks can be evaluated on a total PCB basis, on a total dioxin TEQ basis, and if desired, for any other PCB toxicological endpoint using the appropriate PCB congeners. Following the risk assessment, preliminary remediation goals (PRGs) are developed. PRGs for PCBs are expressed on a total PCB basis.

As the site transitions into site cleanup, site managers must choose between Methods 1668C and 8082A for measuring total PCBs. Total PCBs are measured because PRGs are expressed on a total PCB basis. Use of Method 1668C is recommended because of its lower detection limits, accuracy, and reliability in comparison to Method 8082A. However, Method 8082A is **acceptable** for the determination of total PCBs, and the method provides quicker laboratory response times and lower analytical costs in comparison to Method 1668C. Selection of the appropriate analytical method will be a site-specific decision.

In developing and establishing a long-term monitoring plan for a site, careful consideration of analytical methods is suggested for measuring total PCBs. Use of Method 1668C for the monitoring plan is recommended because of its lower detection limits. Method 8082A can also be used for monitoring quite successfully provided the method is applied similarly over time, e.g., same Aroclors are quantified and sample cleanup is used. What one wants to avoid is switching analytical methods after long term trends are established post remedial completion, e.g., after 15 years of monitoring, switching from Methods 8082A to 1668C because of detection limit issues. When switching occurs, difficulties in understanding long-term trends can occur and careful consideration of analytical methods initially can avoid this issue. If switching methods from Method 8082A to Method 1668C is required, analyzing samples in parallel for one or two years and then continuing with Method 1668C is recommended.

Appendix A:

Examples of data processing to derive Aroclor equivalences from congener specific PCB Data

To compute the amounts of individual Aroclors best matching the distribution of PCBs in a sample, a linear regression can be performed using the equation:

$$Y_i = \sum(Aroclor_m \times f_{m,i})$$

Where $Aroclor_m$ is the amount of the Aroclor “m”, Y_i is the amount of the “i” homologue in the sample, and $f_{m,i}$ is the fraction of homologue “i” in Aroclor “m”. Nine Aroclor mixtures were manufactured, i.e., Aroclor 1221, 1232, 1016, 1242, 1248, 1254, 1260, 1262, and 1268. Table 2A presents the homologue distributions provided in Table 3 in the 2005 ERASC Memorandum for five Aroclors. These values were abstracted from the analyses of Frame et al. [16]. In Table 2B, homologue distributions for eight Aroclor mixtures are provided and these values were obtained by averaging all values for an individual Aroclor mixture in the report by Frame et al [16]. Frame et al. [16] did not analyze the composition of Aroclor 1668 and amounts of decachlorobiphenyl (homologue 10) were not reported as it was used as an internal standard in the method.

The regressions need to constrain the amount of individual Aroclors to be nonnegative and have the intercept set to zero. Further, significance testing on the coefficients is recommended.

To illustrate the approach, linear regressions were performed for eight sediment samples from Portland Harbor Superfund site (Table 2C; from [19], as cited in [20]). The regressions provide “Aroclor equivalence” for the eight samples without having separate analyses by method 8082A for the Aroclor quantification. In Table 2D: First Regression, regression results using all eight Aroclor mixtures in Table 2B are provided for the eight sediment samples. All eight sediment samples have negative amounts for a few individual Aroclor mixtures. For each sample, the Aroclor with largest negative amount was removed, and regression was redone with the remaining seven Aroclor mixtures. These regression results are in Table 2E: Second Regression, and the removed Aroclor (not included in the regression) are marked with “-” symbol in Table 2E. This process of removing the most negative Aroclor mixture and redoing the regressions with fewer Aroclors in the regression was repeated for each sediment sample sequentially until all regression

coefficients (amounts for each Aroclor mixture) were non-negative (summarized in Table 2F). The number of regressions ranged from 3 to 6 depending upon the individual samples.

Once all regression coefficients (amounts for each Aroclor mixture) were non-negative, additional regressions were done by eliminating coefficients (Aroclor amounts) not significantly different from zero ($\alpha = 0.05$). The least significant coefficient (smallest t-value) was removed (marked with “>” symbol in Tables 2), and the regression redone. The coefficients were checked again, and if another Aroclor coefficient was not significantly different from zero, the process was redone by removing the least significant coefficient (Aroclor). This process was redone until all regression coefficients (amounts for individual Aroclors) were significantly greater than zero, summarized in Table 2G. For clarity, the third, fourth, ... and eighth regressions are provided in Tables 2H through Table 2M to show each regression in the process.

As shown in Table 2G, the predicted total PCB from the regressions using homologue amounts align very well with the measured values; i.e., 90 to 104% for regression results using only non-negative criteria for the regression coefficients and 84% to 99% for regression results using non-negative and coefficients significantly greater than zero criteria for the regression coefficients. The Aroclor proportions/equivalences are provided in Table 2G and the standard errors for the regression coefficients averaged approximately 13% of their coefficient values.

In Table 4, quantifications using method 8082A are provided for the samples in Table 2. With Method 8082A, the most representative Aroclors were Aroclors 1254 and 1260, and these Aroclors were used in quantification of total PCB in the samples. The regressions with homologues yielded slightly different Aroclor compositions, e.g., some combination of Aroclors 1248, 1254 and 1262 (Table 2G). A more refined analysis of the eight samples was done using individual PCB congener amounts and Frame et al. [16] composition data with a similar regression technique with outlier detection [21]. These more refined results (Table 3) suggested compositions similar to the those determined as most representative with method 8082A, i.e., predominantly Aroclors 1254 and 1260. Based on this analysis, when determining Aroclors equivalences from Method 1668C congener data, regressions with congener data is recommended/preferred to use of homologues.

These results might or might not be illustrative of other sites and samples.

Table 2. Measured and predicted homologue concentrations in Portland Harbor Superfund site samples using homologue Aroclor composition data

Table 2A. Homologue distributions from Table 3 in the 2005 ERASC Memorandum

Aroclor homologue	A1221	A1232	A1242	A1248	A1254	A1260	A1262	A1016
1			0.008	0	0	0		0.007
2			0.15	0.004	0.002	0.001		0.175
3			0.449	0.22	0.013	0.002		0.547
4			0.326	0.566	0.164	0.005		0.266
5			0.064	0.186	0.53	0.086		0.005
6			0.003	0.02	0.268	0.434		0
7			0	0.006	0.027	0.385		0
8			0	0	0	0.083		0
9			0	0	0	0.007		0
10			0	0	0	0		0

Table 2B. Homologue distributions from Frame et al. [16], average values for each Aroclor

Aroclor homologue	A1221	A1232	A1242	A1248	A1254	A1260	A1262	A1016
1	0.69947	0.35236	0.01029	0.00058	0.00015	0.00025	0.00021	0.00859
2	0.24193	0.20984	0.12741	0.00840	0.00141	0.00047	0.00094	0.21839
3	0.04145	0.27673	0.52527	0.27588	0.01035	0.00179	0.00426	0.56202
4	0.01354	0.13413	0.27435	0.49656	0.15213	0.00293	0.01627	0.20789
5	0.00361	0.02532	0.06035	0.19803	0.57952	0.05028	0.01114	0.00312
6	0	0.00146	0.00233	0.01663	0.22723	0.32112	0.48070	0
7	0	0.00016	0	0.00393	0.02841	0.38672	0.31230	0
8	0	0	0	0	0.00022	0.22889	0.16226	0
9	0	0	0	0	0.00059	0.00755	0.01192	0
10	0	0	0	0	0	0	0	0

Table 2C. Measured concentrations in Portland Harbor sediments by homologue group [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E-ALT	LW3-GCR05W	LW3-GCR10W
homologue	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
1	0.0801	3.088	0.4102	0.02977	0.00519	0	0	0.2362
2	0.3384	2.65	5.5577	0.3123	0.0949	0	0.1659	0.4665
3	1.1388	2.4952	41.2783	2.0806	0.2078	0.0834	0.6365	1.9626
4	3.0038	6.4452	226.8955	17.9069	0.7501	0.3808	2.2747	9.2717
5	4.1368	11.5658	663.4454	51.993	2.9382	0.3377	3.6040	20.1122
6	4.01765	15.018	2610.276	222.6493	7.0997	0.1702	2.736	21.4176
7	2.5436	12.0966	2360.599	243.2407	24.1603	0.0538	1.5431	13.8986
8	0.7285	3.5583	685.869	67.786	23.427	0	0.3881	4.2331
9	0.1837	0.4994	41.6	3.392	3.695	0	0.0679	0.9201
10	0.0878	0.246	0.525	0.0564	0.107	0	0.0537	0.454
total	16.2592	57.6625	6636.456	609.4470	62.4853	1.0259	11.4699	72.9726

Table 2D. First Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E- ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	2088	12891	2976508	339230	-52805	98	1611	11008
A1232	-4189	-25856	-5972242	-680651	105950	-196	-3232	-22085
A1242	170	1058	250087	28483	-4448	7.5	131	914
A1248	302	1846	423580	48286	-7505	14.8	233	1578
A1254	46	266	58346	6603	-1027	2.2	36	243
A1260	24	152	33907	3960	-430	0.9	18	128
A1262	-28	-186	-42458	-5037	746	-1.3	-22	-147
A1016	1603	9887	2278740	259716	-40419	75.1	1236	8434
total	16	57	6804	597	56	0.990	11	73

Table 2E. Second Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E- ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	0.133	4.500	-1.733	-0.037	-	0.005	0.020	0.382
A1232	-	-	-	-	0.124	-	-	-
A1242	-5.459	-25.616	-125.982	-33.633	-8.657	-0.728	-4.282	-11.595
A1248	5.389	12.456	116.499	23.982	7.563	0.893	4.020	11.756
A1254	5.590	16.585	831.130	47.729	-6.375	0.356	5.126	30.358
A1260	1.823	15.579	2458.322	376.120	127.618	-0.107	1.118	11.769
A1262	4.429	13.147	3483.006	199.171	-68.625	0.233	2.461	22.462
A1016	4.328	21.461	80.862	19.352	4.966	0.382	3.041	7.746
total	16.233	58.114	6842.102	632.684	56.615	1.034	11.503	72.878

Table 2F. Regression results after elimination of all coefficients with negative values: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E- ALT	LW3-GCR05W	LW3-GCR10W
Regression Coefficients								
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	0.210	4.861	-	-	-	-	0.044	0.389
A1232	-	-	-	-	-	-	-	-
A1242	-	-	-	-	0.485	-	-	-
A1248	4.037	6.116	69.545	9.427	0.099	0.545	2.695	7.724
A1254	5.534	16.323	836.242	49.916	-	0.416	5.190	30.712
A1260	1.736	15.171	2458.084	376.285	55.773	-	1.080	11.715
A1262	4.544	13.685	3482.431	198.603	-	0.126	2.497	22.467
A1016	0.079	1.523	-	-	-	-	-	-
total	16.140	57.678	6846.301	634.232	56.357	1.087	11.505	73.006
Standard error of regression coefficients								
A1221	0.402	2.007	--	--	--	--	0.248	1.671
A1232	--	--	--	--	--	--	--	--
A1242	--	--	--	--	16.395	--	--	--
A1248	0.851	4.252	426.478	48.893	16.620	0.065	0.358	2.416
A1254	0.619	3.095	423.591	48.562	--	0.065	0.355	2.397
A1260	1.623	8.114	1220.474	139.919	10.765	--	1.024	6.907
A1262	1.523	7.612	1141.937	130.916	--	0.060	0.958	6.462
A1016	0.698	3.488	-	-	--	--	--	--
total	0.811	4.053	525.646	60.262	14.649	0.077	0.499	3.369

Table 2G. Regression results after elimination of all coefficients not significantly great than zero: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E- ALT	LW3-GCR05W	LW3-GCR10W
Regression coefficients								
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	>	>	-	-	-	-	>	>
A1232	-	-	-	-	-	-	-	-
A1242	-	-	-	-	>	-	-	-
A1248	4.122	>	>	>	>	0.526	2.697	7.737
A1254	5.509	20.120	>	>	-	0.464	5.195	30.764
A1260	>	28.083	>	597.333	55.788	-	>	>
A1262	6.078	>	5949.706	>	-	>	3.445	32.763
A1016	>	>	-	-	-	-	-	-
Total	15.709	48.202	5949.706	597.333	55.788	0.990	11.337	71.264
Standard error of regression coefficients								
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	--	--	--	--	--	--	--	--
A1232	--	--	--	--	--	--	--	--
A1242	--	--	--	--	--	--	--	--
A1248	0.501	--	--	--	--	0.077	0.335	2.569
A1254	0.497	3.916	--	--	--	0.072	0.333	2.551
A1260	--	4.529	--	48.818	9.470	--	--	--
A1262	0.461	--	489.835	--	--	--	0.308	2.364
A1016	--	--	--	--	--	--	--	--
Total	0.594	4.952	489.835	48.818	9.470	0.074	0.572	3.046
R-Squared	0.991	0.922	0.943	0.943	0.794	0.957	0.993	0.990
F value	254.516	47.437	147.534	149.720	34.701	89.590	320.856	221.688
Proportions by Aroclor mixture								
A1221	>	>	-	-	-	-	>	>
A1232	-	-	-	-	-	-	-	-
A1242	-	-	-	-	>	-	-	-
A1248	26%	>	>	>	>	53%	24%	11%
A1254	35%	42%	>	>	-	47%	46%	43%
A1260	>	58%	>	100%	100%	-	>	>
A1262	39%	>	100%	>	-	>	30%	46%
A1016	>	>	-	-	-	-	-	-
Total PCB: Method 1668C (ug/kg-dw)								
	16.2592	57.6625	6636.456	609.4470	62.4853	1.0259	11.4699	72.9726
Total PCB: Regression results after elimination of all coefficients with negative values (ug/kg-dw)								
	16.140	57.678	6846.301	634.232	56.357	1.087	11.505	73.006
% of Method 1668C	99%	100%	103%	104%	90%	106%	100%	100%
Total PCB: Regression results after elimination of all coefficients with negative values and after elimination of all coefficients not significantly great than zero (ug/kg-dw)								
	15.709	48.202	5949.706	597.333	55.788	0.990	11.337	71.264
% of Method 1668C	97%	84%	90%	98%	89%	97%	99%	98%

Table 2H. Third Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E-ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	0.210	4.861	0.038	0.436	-	0.015	0.080	0.545
A1232	-	-	-	-	1.446	-	-	-
A1242	-	-	-	-	13.030	-	-	-
A1248	4.037	6.116	85.315	15.657	5.643	0.713	2.960	8.886
A1254	5.534	16.323	829.837	47.384	-10.290	0.348	5.082	30.239
A1260	1.736	15.171	2456.315	375.584	59.086	-0.119	1.050	11.584
A1262	4.544	13.685	3485.651	199.877	-	0.248	2.551	22.705
A1016	0.079	1.523	-17.201	-6.828	-15.151	-0.185	-0.292	-1.280
total	16.140	57.678	6839.956	632.111	53.763	1.021	11.430	72.680

Table 2I. Fourth Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E-ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	0.220	5.047	-2.070	-0.401	-	-0.007	0.044	0.389
A1232	-	-	-	-	0.674	-	-	-
A1242	-	-	-	-	-5.580	-	-	-
A1248	4.109	7.499	69.693	9.456	10.012	0.545	2.695	7.724
A1254	5.505	15.760	836.192	49.907	-9.984	0.417	5.190	30.712
A1260	1.728	15.015	2458.076	376.283	58.959	-0.100	1.080	11.715
A1262	4.558	13.968	3482.452	198.607	-	0.214	2.497	22.467
A1016	>	>	-	-	-	-	-	-
total	16.120	57.290	6844.342	633.852	54.081	1.068	11.505	73.006

Table 2J. Fifth Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E-ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	>	5.057	-	-	-	-0.007	>	>
A1232	-	-	-	-	-0.079	-	-	-
A1242	-	-	-	-	0.540	-	-	-
A1248	4.124	7.268	69.545	9.427	0.087	0.545	2.698	7.752
A1254	5.500	16.315	836.242	49.916	-	0.416	5.189	30.702
A1260	1.727	29.035	2458.084	376.285	55.773	-	1.079	11.714
A1262	4.560	>	3482.431	198.603	-	0.126	2.497	22.471
A1016	>	>	-	-	-	-	-	-
total	15.912	57.674	6846.301	634.232	56.322	1.080	11.463	72.639

Table 2K. Sixth Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E-ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	>	5.305	-	-	-	-	>	>
A1232	-	-	-	-	-	-	-	-
A1242	-	-	-	-	0.485	-	-	-
A1248	4.122	>	>	>	0.099	0.545	2.697	7.737
A1254	5.509	20.051	872.275	54.801	-	0.416	5.195	30.764
A1260	>	28.098	2457.383	376.190	55.773	-	>	>
A1262	6.078	>	3474.210	197.489	-	0.126	3.445	32.763
A1016	>	>	-	-	-	-	-	-
Total	15.709	53.454	6803.869	628.480	56.357	1.087	11.337	71.264

Table 2L. Seventh Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E-ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221		>	-	-	-	-		
A1232		-	-	-	-	-		
A1242		-	-	-	0.564	-		
A1248		>	>	>	>	0.526		
A1254		20.120	883.611	>	-	0.464		
A1260		28.083	>	379.073	55.778	-		
A1262		>	5633.731	214.547	-	>		
A1016		>	-	-	-	-		
total		48.202	6517.343	593.620	56.341	0.990		

Table 2M. Eighth Regression: Amounts of Aroclors determined using linear regression with measured homologue concentrations in sediments [19]

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E-ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221			-	-	-			
A1232			-	-	-			
A1242			-	-	>			
A1248			>	>	>			
A1254			>	>	-			
A1260			>	597.333	55.788			
A1262			5949.706	>	-			
A1016			-	-	-			
Total			5949.706	597.333	55.788			

Table 3. Regression with congener data and outlier detection

Sample ID	LW3-GCA05 W-C00	LW3-GCA10 W-C00	LW3-GCA11E -C00	LW3-GCA12E -C00-R	LW3-GCA12 W-C00	LW3-GCR01E- ALT	LW3-GCR05W	LW3-GCR10W
Aroclor	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw	µg/kg-dw
A1221	-	>	-	-	-	-	-	>
A1232	>	8.91	-	-	-	-	-	>
A1242	-	-	-	-	>	-	-	>
A1248	3.26	>	>	>	>	0.60	1.90	3.93
A1254	4.92	15.16	154.80	-	3.56	0.48	4.92	23.95
A1260	5.22	26.50	4666.74	504.73	10.45	-	3.28	29.87
A1262	>	-	>	35.73	-	-	>	>
Total	13.40	50.57	4821.54	540.47	14.00	1.08	10.11	57.75

Standard error of regression coefficients

Aroclor	µg/kg-dw							
A1221	-	-	-	-	-	-	-	-
A1232	-	0.49	-	-	-	-	-	-
A1242	-	-	-	-	-	-	-	-
A1248	0.29	-	-	-	-	0.12	0.22	1.00
A1254	0.31	0.74	58.98	-	0.18	0.10	0.23	1.04
A1260	0.31	0.74	66.43	16.40	0.25	-	0.20	1.04
A1262	-	-	-	16.15	-	-	-	-
Total	0.40	0.99	76.07	15.66	0.28	0.11	0.27	1.38

Appendix B:

Developing a relationship between total PCBs by Methods 8082A and 1668C

To develop a correlation/relationship between total PCBs by Methods 8082A and 1668C, one would need to run sufficient analyses with both methods to develop a strong relationship. The number of samples analyzed by both methods will be a function of a number of factors including the quality of the analytical data for Method 8082A, range in total PCB concentrations in the samples, and variabilities in composition of the PCBs across the site. If the range in concentrations is too narrow, e.g., less than two orders of magnitude, developing a useful relationship will be difficult. Minimum number of analysis pairs is difficult to assign *a priori* without background information on the site, and it is recommended that a statistician be consulted in developing a relationship.

To develop a relationship, the measurement pairs should be visually examined using an X-Y plot. The relationship between the two total PCB values might be linear, quadratic, or require some type of transformation. Once the form of the relationship is determined, fitting of an appropriate curve to the data would be done using standard regression techniques. One should develop a regression equation with all coefficients being significant (e.g., $\alpha = 0.05$) and the regression itself being highly significant. As mentioned in the preceding text, developing a relationship will not be a trivial effort, will take a large amount of analytical effort, and potentially, be costly because of the duplication in sample analyses.

To illustrate some of the complexities in developing a relationship, data for sediments from Round 3 sampling in Portland Harbor Superfund RI/FS [19] are shown in Table 4. One of the remarkable points in the data set are the low detection limits for 8082A, i.e., approximately 1.5 ug/kg-dw, and these detection limits are much lower than those traditionally observed with Method 8082A [10]. In the data set, 4 of the 30 samples were nondetects for all Aroclors and the range in total PCB spans a little over three orders of magnitude. Regression was performed with and without an intercept using \log_{10} transformed data with the nondetect values excluded (Table 5). The intercept was not significantly different from zero, and after eliminating the intercept, the resulting relationship for this data set is:

$$\log_{10} C_{M-1668C} = 0.9496538 \times \log_{10} C_{M-8082A} \quad r^2 = 0.905, n = 26 \quad (1)$$

where $C_{M-1668C}$ is the total PCB concentration measured using Method 1668C and $C_{M-8082A}$ is the total PCB concentration measured using Method 8082A. Equation 1 is shown in Figure 1 with the 95% confidence and prediction levels for the regression. The 95% prediction levels are slightly larger than an order of magnitude, factor of approximately 13.4, while the confidence levels for the regression are much smaller and range from 1.2 to 2.8-fold for the regression. Eleven of 26 data points (excluding non-detects in Method 8082A) reside within the narrow confidence limits for the regression line, and 25 of the 26 data points reside with the prediction limits for the regression. In Figure 2, the data and equation 1 are shown with lines that are 3-fold and 10-fold above and below the regression line. Twenty-one and 24 of the 26 data points reside within ± 3 -fold and ± 10 of the regression line, respectively. Closer examination of the data reveals two potential outliers in this data set. At your site, such outliers would require further work to resolve and understand such observations.

In applying a developed regression equation at your site, you must know the level of uncertainty acceptable for predictions of total PCB from the measurements performed with method 8082A. This will be a site-specific decision and require some internal discussion. With the Portland Harbor data set, the Methods 8082A and 1668C provide very similar results since the coefficient in Equation 1 is not significantly different from 1.00.

Table 4. Quantification Results for Total PCBs by Method 8082A and Method 1668C for Portland Harbor Superfund site

Sample ID	Method 8082A (ug/kg-dw) ⁺										Method 1668C
	Aroclor 1016	Aroclor 1221	Aroclor 1232	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Aroclor 1262	Aroclor 1268	Total PCB (ug/kg-dw)	Total PCB [#] (ug/kg-dw)
LW3-GCA01E-C00						2.3	1.7			4	3.26099
LW3-GCA02W-C10						3.3	3.3			6.6	3.44917
LW3-GCA03W-C00						7.9	9.3			17.2	15.36144
LW3-GCA04W-C00						9.9	8.5			18.4	11.21967
LW3-GCA05E-C00						16	15			31	19.38426
LW3-GCA05W-C00						20	8.6			28.6	16.25923
LW3-GCA10W-C00						29	36			65	57.66246
LW3-GCA11E-C00							3500			3500	6636.4567
LW3-GCA12E-C00-R						31	140			171	609.44696
LW3-GCA12W-C00						3.2	2.3			5.5	62.4853
LW3-GCR01E-ALT										0.8*	1.02594
LW3-GCR05W						16	8.6			24.6	11.46993
LW3-GCR10W						120	98			218	72.97256
LW3-GCR12W						21				21	6.83068
LW3-GCRSP01W-1							5.1			5.1	6.55823
LW3-GCRSP06W						11				11	11.88543
LW3-GCRSP08W				82		47	34			163	133.1186
LW3-GCRSP11E							900			900	10.65876
LW3-GCRSP12E						42	92			134	912.14302
LW3-GSP01E										0.7*	2.95495
LW3-GSP03E							31			31	32.13624
LW3-GSP04W										0.75*	160.26001
LW3-GSP05E						14	14			28	18.52396
LW3-GSP07E							16			16	26.24116
LW3-GSP07W										0.65*	1.20213
LW3-GSP08E							7.9			7.9	8.79936
LW3-GSP09W						190				190	699.4293
LW3-GSP10E							16			16	7.73636
LW3-GSP10W							32			32	52.47157
LW3-GSP12W-ALT						29				29	12.19023

⁺ Aroclors with no values were not detected. * ½ Method detection limit. [#] Total PCB found by summing only the detected congeners. Congeners not detected were not included in the total PCB value.

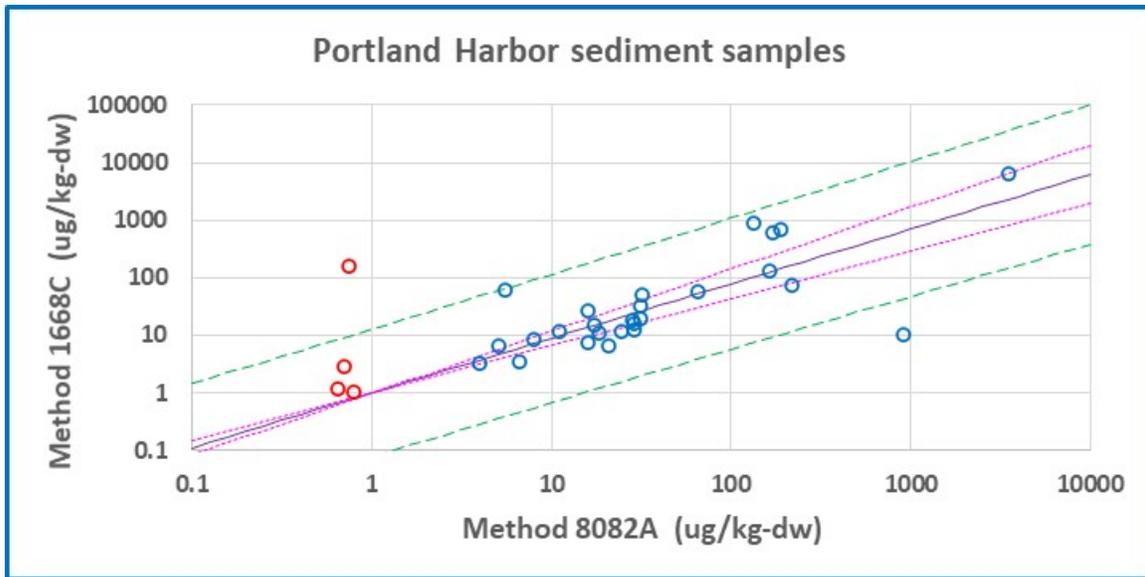


Figure 1. Concentrations of total PCBs measured using Methods 8082A and 1668C for sediment samples from Portland Superfund site. Blue circle – measurements are above detection limits for Methods 1668C and 8082A. Red circles – measurements are above detection limits for Method 1668C and less than the detection limits for Method 8082A. For Method 8082A, $\frac{1}{2}$ of the detection limit are plotted for samples where Aroclors were not detected. The solid line is the regression fit to the data, the dotted lines are the 95% confidence limits for the regression lines, and the dashed lines are 95% prediction lines for the regression.

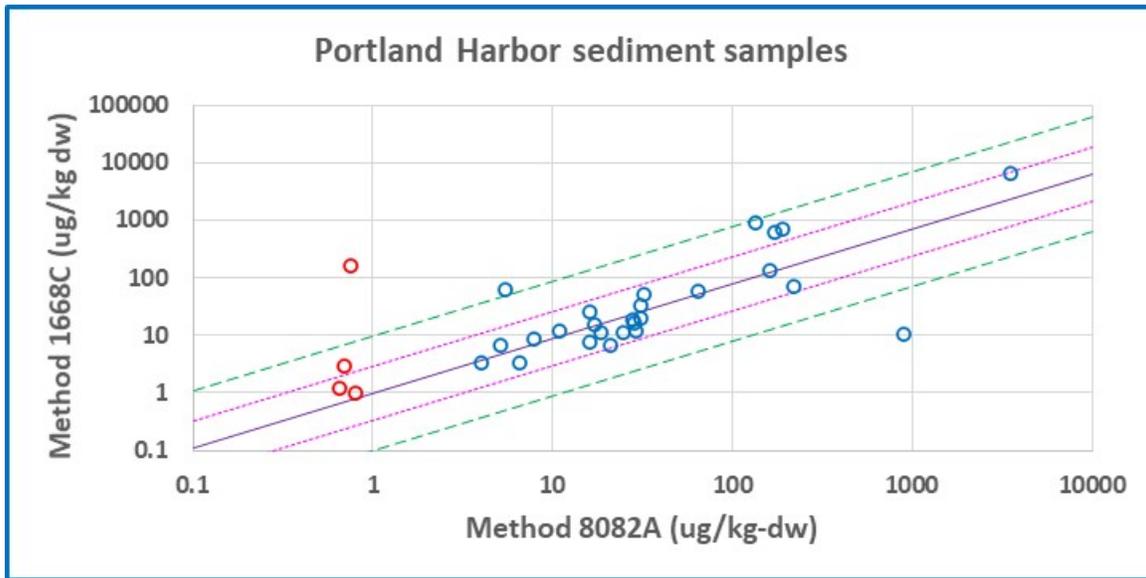


Figure 2. Concentrations of total PCBs measured using Methods 8082A and 1668C for sediment samples from Portland Superfund site. Blue circle – measurements are above detection limits for Methods 1668C and 8082A. Red circles – measurements are above detection limits for Method 1668C and less than the detection limits for Method 8082A. For Method 8082A, $\frac{1}{2}$ of the detection limit are plotted for samples where Aroclors were not detected. The solid line is the regression fit to the data, the dotted lines are 3-fold from the regression line, and the dashed lines are 10-fold from the regression line.

Table 5. Regression statistics for Portland Harbor total PCB data by Method 1668C and Method 8082A.
SUMMARY OUTPUT

<i>Regression Statistics</i>	
Multiple R	0.9513213
R Square	0.9050122
Adjusted R Square	
Standard Error	0.5378003
Observations	26

ANOVA					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	68.891946	68.891946	238.1916	5.829E-14
Residual	25	7.2307277	0.2892291		
Total	26	76.122673			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 99.0%</i>	<i>Upper 99.0%</i>
Intercept	0	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A
X Variable 1	0.9496538	0.0615321	15.433457	2.751E-14	0.822926	1.0763816	0.7781369	1.1211707

SUMMARY OUTPUT

<i>Regression Statistics</i>	
Multiple R	0.7528105
R Square	0.5667237
Adjusted R Square	0.5486705
Standard Error	0.5450046
Observations	26

ANOVA					
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>
Regression	1	9.3243382	9.3243382	31.391906	9.116E-06
Residual	24	7.1287203	0.29703		
Total	25	16.453058			

	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 99.0%</i>	<i>Upper 99.0%</i>
Intercept	0.1553836	0.2651486	0.5860246	0.5633302	-0.3918562	0.7026235	-0.586221	0.8969882
X Variable 1	0.8666943	0.1546882	5.602848	9.116E-06	0.5474336	1.185955	0.4340409	1.2993478

References

1. Van den Berg M, Birnbaum L, Bosveld A, Brunström B, Cook P, Feeley M, Giesy JP, Hanberg A, Hasegawa R, Kennedy SW. 1998. Toxic equivalency factors (TEFs) for PCBs, PCDDs, PCDFs for humans and wildlife. *Environmental Health Perspectives* 106:775-792.
2. Van den Berg M, Birnbaum LS, Denison M, De Vito M, Farland W, Feeley M, Fiedler H, Hakansson H, Hanberg A, Haws L. 2006. The 2005 World Health Organization reevaluation of human and mammalian toxic equivalency factors for dioxins and dioxin-like compounds. *Toxicological Sciences* 93:223-241.
3. US-EPA. 1999. Method 1668, Revision A: Chlorinated biphenyl congeners in water, soil sediment, bio-solid and tissue by HRGC/HRMS. EPA-821-R-00-002., Office of Water, Washington, DC. USA.
4. US-EPA. 2008. Method 1668B: Chlorinated biphenyl congeners in water, soil sediment, bio-solid and tissue by HRGC/HRMS. EPA/821/R-08/020., Office of Water, Washington, DC. USA.
5. US-EPA. 2010. Method 1668C: Chlorinated biphenyl congeners in water, soil sediment, bio-solid and tissue by HRGC/HRMS. EPA-820-R-10-005, Office of Water, Washington, DC. USA.
6. Cornelissen G, Gustafsson Ö, Bucheli TD, Jonker MT, Koelmans AA, van Noort PC. 2005. Extensive sorption of organic compounds to black carbon, coal, and kerogen in sediments and soils: mechanisms and consequences for distribution, bioaccumulation, and biodegradation. *Environmental Science & Technology* 39:6881-6895.
7. Ghosh U, Luthy RG, Cornelissen G, Werner D, Menzie CA. 2011. In-situ sorbent amendments: a new direction in contaminated sediment management. *Environmental Science & Technology* 39:6881-6895.
8. US-EPA. 2012. Sediment Assessment and Monitoring Sheet (SAMS) #3: Guidelines for using passive samplers to monitor organic contaminants at Superfund sediment sites. OSWER Directive 9200.1-110 FS. Office of Superfund Remediation and Technology Innovation and Office of Research Development, Washington, DC.
9. US-EPA. 2007. Polychlorinated Biphenyls (PCBs) by Gas Chromatography. EPA Method 8082A. Revision 1, February 2007. Final Update IV to the Third Edition of the Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846. US Environmental Protection Agency, Washington, DC. USA.
10. Beliveau AF. 2001. PCB analyses needs for risk evaluation. In Robertson L, Hansen L, eds, *PCBs—Recent Advances in Environmental Toxicology Health Effects*. University Press of Kentucky, Lexington, KY.

11. Erickson MD. 2018. Aroclor misidentification in environmental samples: how do we communicate more effectively between the laboratory and the data user? *Environmental Science Pollution Research* 25:16291-16299.
12. Narquis C, Hyatt J, Prignano A. 2007. Generating the Right Data: Determination of Aroclors Versus PCB Congeners. Vol 13. Prepared for the US Department of Energy, Assistant Secretary for Environmental Management. Fluor, Richland, WA.
13. US-EPA. 2015. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015). <https://www.epa.gov/hw-sw846/sw-846-compendium>
14. Erickson MD. 2020. Environmental PCB forensics: processes and issues. *Environmental Science Pollution Research* 27:8926-8937.
15. Battelle. 2012. A Handbook for Determining the Sources of PCB Contamination in Sediments TECHNICAL REPORT TR-NAVFAC EXWC-EV-1302. see https://clu-in.org/download/contaminantfocus/pcb/pcb_sediment_handbook.pdf Accessed 21-June-2021. p. p 164.
16. Frame GM, Cochran JW, Bøwadt SS. 1996. Complete PCB congener distributions for 17 Aroclor mixtures determined by 3 HRGC systems optimized for comprehensive, quantitative, congener-specific analysis. *Journal of High Resolution Chromatography* 19:657-668.
17. Frame GM, Wagner RE, Carnahan JC, Brown Jr JF, May RJ, Smullen LA, Bedard DL. 1996. Comprehensive, quantitative, congener-specific analyses of eight Aroclors and complete PCB congener assignments on DB-1 capillary GC columns. *Chemosphere* 33:603-623.
18. Kalbe U, Lehnik-Habrink P, Bandow N, Sauer A. 2019. Validation of European horizontal methods for the analysis of PAH, PCB and dioxins in sludge, treated biowaste and soil. *Environmental Sciences Europe* 31:29.
19. Integral Consulting Inc. 2008. Portland Harbor RI/FS Round 3B Fish and Invertebrate Tissue and Collocated Surface Sediment Data Report Draft. Prepared for the Lower Willamette Group, Portland, OR. Windward Environmental LLC, Seattle WA, and Integral Consulting, Inc., Mercer Island, WA.
20. US-EPA. 2016. Portland Harbor RI/FS. Remedial Investigation Report. Final.
21. Burkhard LP, Weininger D. 1987. Determination of polychlorinated biphenyls using multiple regression with outlier detection and elimination. *Analytical Chemistry* 59:1187-1190.