Appendix D: Water Quality Monitoring Analytical PCB Methods

Technical Memorandum

Prepared For: US EPA Region 10 Spokane and Little Spokane Rivers Polychlorinated Biphenyls Total Maximum Daily Loads TMDL Team

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Acronyms & Abbreviations

Acronym/Abbreviation	Definition
GC/MS	Gas chromatography/mass spectrometry
µg/kg	Microgram per kilogram
MDL	Method detection level
ML	Minimum level
ng/g	Nanogram per gram
ng/kg	Nanogram per kilogram
ng/L	Nanogram per liter
PCBs	Polychlorinated biphenyls
pg/L	Picogram per liter
ppb	Parts per billion
ppm	Parts per million
ppq	Parts per quadrillion
ppt	Parts per trillion
QL	Quantitation level
SIM	Selected ion monitoring

1 Analytical PCB Methods for Water Quality Monitoring Overview

Eight methods are summarized as part of this appendix, including but not limited to methods approved under 40 CFR 136.3 for use in NPDES permit reporting and applications. Some of the methods discussed herein are more commonly used outside of the Clean Water Act. Most current PCB methods use some form of gas chromatography connected to a detector (e.g., electron capture detector or mass spectrometer) to detect, characterize, and determine or quantify individual PCB congeners or aroclors within a variety of matrices such as, water, tissue, soil, sediment, and biosolids. The detection limits of the methods presented below range from parts per quadrillion (ppq) to parts per billion (ppb) (Table D-1). As a result, it can be challenging to define a single detection limit for total PCBs since the total reflects the sum of PCB congeners with potentially different detection limits. This is relevant because total PCBs is used to set water quality thresholds and criteria, and is the parameter used to set TMDL targets. See Sections 2 – 9 for details on individual methods including a short description, detection and quantitation limits and any identified studies that have used any of these methods within the Spokane Watershed including Water Resource Inventory Areas (WRIAs) Lower Spokane (54), Little Spokane (55), Hangman Creek (56) and Middle Spokane (57).

Table D-1: Summary of PCB Congener and Aroclor Methods

Method	**Detection Limit Magnitude (ppt, ppm, ppb, ppq)**	**Measures Congeners or Aroclors?**
1628	ppt	Congeners
1668C	ppq	Congeners
8082A	ppt	Aroclors and some congeners
608.3*	ppt	Aroclors
625.1*	ppb	Aroclors
6410 B-2000*	ppb	Aroclors
608-3M0222*	ppb	Aroclors
Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater*	ppb	Aroclors
*Note: Methods are approved under 40 CFR 136.3 for NPDES permit reports and applications, while remaining methods remain unapproved for this use. See sections below for details on method detection limits and minimum or quantitation levels		

2 Method 608.3

2.1 Description of Method

EPA Method 608 can analyze for PCB aroclors (PCB-1016, PCB-1221, PCB-1232, PCB-1242, PCB-1254, PCB-1260, PCB-1268) and several organochlorine pesticides in water using gas chromatography with a halogen-specific detector. The most recent version of this method is Method 608.3 (US EPA, 2016). Method 608 was originally published in 1984, and the updated Method 608.3 was promulgated in the 2017 Methods Update Rule (82 FR 40836). The method appears in Appendix A (40 CFR Part 136).

Method 608.3 is approved for use with NPDES permit reports and applications (40 CFR 136.3), and it is the most sensitive among such approved methods for PCB aroclors. Thus, the sufficiently sensitive methods rule (79 FR 49001) will generally require the use of this method for NPDES permit reports and applications.

2.2 Detection and Quantitation Limits

A method detection limit (MDL) and minimum level (ML) are published directly in method 608.3 for only one PCB aroclor: PCB-1242. The published MDL is 65 ng/L (65,000 pg/L) and the published ML is 195 ng/L (195,000 pg/L).[1] Individual NPDES permits issued to point sources discharging to the main stem Spokane River in 2022 list Method 608.3 as the recommended analytical protocol and list the MDL and quantitation level (QL) of all PCB aroclors as 65 ng/L and 195 ng/L, respectively. The QL is synonymous with the ML (79 FR 49001). Note: the MDL value published within method 608.3 is unchanged from the MDL published in Method 608, which was promulgated in 1984. The MDL of 65 ng/L or 65,000 pg/L is much higher than any of the applicable human health water quality criteria for the Spokane River, as shown in **Error! Reference source not found.**.

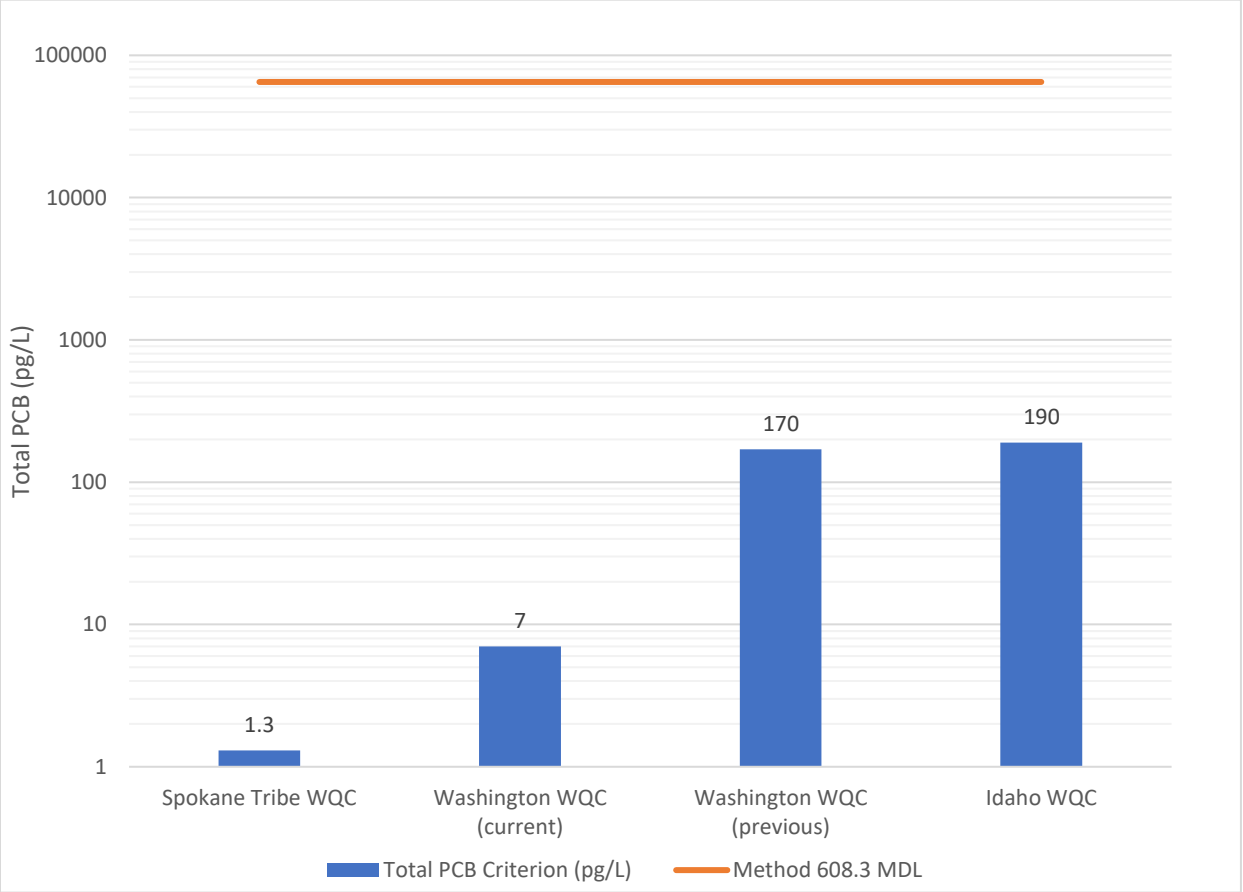

Figure D-1: PCB water quality criteria (WQC) relative to MDL of EPA Method 608.3

[1] The method lists the ML for PCB-1242 as 95 ng/L, however, this is a typographical error. The actual ML is 195 ng/L (3 times the MDL). See: https://www.epa.gov/cwa-methods/2017-methods-update-rule-frequent-questions

2.3 Use Within the Spokane Watershed

As stated in Section 2.2, above, Method 608.3 is used for determining compliance with PCB effluent limits in permits for discharge to the main stem Spokane River in the State of Washington. In addition to this method's use in NPDES permits, a search of Washington's Environmental Information Management (EIM) database identified three studies within the watershed (WRIAs 54, 55, 56, and 57) which included data collected using EPA Method 608, as shown in Table D-2.

Table D-2: Studies in EIM Using EPA Method 608 or 608.3

Study Name	Study ID	Field Collection Date Range
Heglar Kronquist Landfill RI/FS, Mead, WA	AO6557	5/4/2010 - 7/28/2011
1992 Lakes Toxics Screening Survey	DSER0002	6/8/1992 - 2/2/1993
Riverfront Park Spokane	VCEA0318	4/4/2016 - 6/29/2020

3 Method 1628

3.1 Description of Method

Method 1628 is used for determination of all 209 polychlorinated biphenyl (PCBs) congeners in water, soil, sediment, biosolids, and tissue, by low-resolution gas chromatography/mass spectrometry (GC/MS) using selected ion monitoring (SIM) (US EPA, 2021).

Method 1628 detects all 209 PCB congeners and quantifies them either directly or indirectly. A total of 29 carbon-13 labeled PCB congeners are used as isotope dilution quantification standards. An additional 19 congeners are quantified by an extracted internal standard procedure, using one of the isotope dilution standards. The remaining 144 congeners are quantified against a labeled standard in the same homolog. This approach strikes a balance between enabling the laboratory to detect and quantify all 209 congeners, while not making the method too arduous. Method performance was similar across all the congeners, regardless of the quantification approach.

Although the EPA has completed a multi-laboratory validation study on Method 1628, it is not yet approved for use with NPDES permits under 40 CFR Part 136.

3.2 Detection and Quantitation Limits

MDLs and MLs for aqueous, solid, and tissue matrices published in Method 1628 are shown in Table D-3.

Table D-3: Method Detection Limits and Minimum Levels for EPA Method 1628.

Congener	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
	MDL	ML	MDL	ML	MDL	ML
PCB-1	1.75	5	0.63	2	0.11	0.2
PCB-2	0.71	2	0.06	0.2	0.13	0.5
PCB-3	0.69	2	0.10	0.2	0.11	0.2
PCB-4+10	1.90	5	0.15	0.5	0.23	0.5
PCB-8+5	1.00	2	0.22	0.5	0.18	0.5
PCB-6	0.57	2	0.09	0.2	0.10	0.2
PCB-7+9	1.17	5	0.24	1	0.22	0.5

Congener	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
	MDL	ML	MDL	ML	MDL	ML
PCB-11	0.72	2	0.42	1	0.06	0.2
PCB-12+13	1.11	5	0.21	0.5	0.13	0.5
PCB-14	0.64	2	0.11	0.2	0.07	0.2
PCB-15	0.44	1	0.09	0.2	0.06	0.2
PCB-16+32	0.80	2	0.14	0.5	0.18	0.5
PCB-17	0.49	2	0.07	0.2	0.08	0.2
PCB-18	0.46	1	0.07	0.2	0.09	0.2
PCB-19	0.63	2	0.08	0.2	0.07	0.2
PCB-33+20+21	1.11	5	0.30	1	0.20	0.5
PCB-22	0.39	1	0.08	0.2	0.10	0.2
PCB-34+23	1.00	2	0.11	0.2	0.13	0.5
PCB-24+27	0.64	2	0.09	0.2	0.11	0.5
PCB-25	0.46	1	0.08	0.2	0.08	0.2
PCB-26	0.43	1	0.09	0.2	0.07	0.2
PCB-28	0.69	2	0.15	0.5	0.14	0.5
PCB-29	0.49	2	0.06	0.2	0.08	0.2
PCB-30	0.61	2	0.08	0.2	0.08	0.2
PCB-31	0.50	2	0.07	0.2	0.09	0.2
PCB-35	0.89	2	0.21	0.5	0.14	0.5
PCB-36	0.54	2	0.17	0.5	0.10	0.2
PCB-37	0.44	1	0.18	0.5	0.12	0.5
PCB-38	1.66	5	0.14	0.5	0.13	0.5
PCB-39	0.53	2	0.10	0.2	0.06	0.2
PCB-40	1.12	5	0.16	0.5	0.13	0.5
PCB-41+64	0.97	2	0.17	0.5	0.15	0.5
PCB-42	0.73	2	0.10	0.2	0.09	0.2
PCB-49+43	1.06	2	0.24	1	0.22	0.5
PCB-44	0.40	1	0.11	0.5	0.09	0.2
PCB-45	0.31	1	0.09	0.2	0.07	0.2
PCB-46	0.36	1	0.06	0.2	0.07	0.2
PCB-47+48+75	1.71	5	0.24	1	0.23	0.5
PCB-50	0.58	2	0.07	0.2	0.07	0.2
PCB-51	0.48	2	0.06	0.2	0.07	0.2
PCB-52+73	0.97	2	0.17	0.5	0.24	1.0
PCB-53	0.33	1	0.05	0.2	0.05	0.2
PCB-54	0.58	2	0.06	0.2	0.06	0.2
PCB-55	0.39	1	0.08	0.2	0.10	0.2
PCB-56+60	0.74	2	0.13	0.5	0.09	0.2
PCB-57	0.47	1	0.10	0.2	0.07	0.2
PCB-58	0.46	1	0.11	0.5	0.09	0.2
PCB-59	0.60	2	0.07	0.2	0.08	0.2

Congener	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
	MDL	ML	MDL	ML	MDL	ML
PCB-74+61	0.96	2	0.14	0.5	0.12	0.5
PCB-62	0.49	2	0.11	0.5	0.06	0.2
PCB-63	0.38	1	0.08	0.2	0.08	0.2
PCB-65	0.57	2	0.10	0.2	0.07	0.2
PCB-66+80	0.91	2	0.19	0.5	0.16	0.5
PCB-67	0.45	1	0.11	0.2	0.07	0.2
PCB-68	0.66	2	0.16	0.5	0.10	0.2
PCB-69	0.53	2	0.12	0.5	0.06	0.2
PCB-70	1.32	5	0.08	0.2	0.09	0.2
PCB-71	1.09	2	0.14	0.5	0.07	0.2
PCB-72	0.50	2	0.10	0.2	0.11	0.2
PCB-76	0.53	2	0.11	0.2	0.08	0.2
PCB-77	0.50	2	0.07	0.2	0.09	0.2
PCB-78	0.51	2	0.10	0.2	0.11	0.5
PCB-79	0.48	2	0.08	0.2	0.06	0.2
PCB-81	0.49	2	0.09	0.2	0.07	0.2
PCB-82	0.61	2	0.06	0.2	0.08	0.2
PCB-83+109	0.76	2	0.14	0.5	0.10	0.2
PCB-84	2.53	10	0.07	0.2	0.06	0.2
PCB-85+120	1.19	5	0.15	0.5	0.17	0.5
PCB-97+86	1.67	5	0.11	0.2	0.07	0.2
PCB-87+115+116	2.23	5	0.37	1	0.22	0.5
PCB-88+121	0.93	2	0.12	0.5	0.13	0.5
PCB-90+101+89	3.36	10	0.24	1	0.10	0.2
PCB-91	0.39	1	0.05	0.2	0.05	0.2
PCB-92	0.53	2	0.06	0.2	0.05	0.2
PCB-95+93	2.01	5	0.12	0.5	0.10	0.2
PCB-94	0.32	1	0.06	0.2	0.03	0.1
PCB-96	0.37	1	0.06	0.2	0.05	0.1
PCB-98+102	0.77	2	0.12	0.5	0.12	0.5
PCB-99	1.30	5	0.10	0.2	0.06	0.2
PCB-100	0.50	2	0.17	0.5	0.06	0.2
PCB-103	0.48	2	0.15	0.5	0.06	0.2
PCB-104	0.51	2	0.05	0.2	0.05	0.2
PCB-105+127	1.23	5	0.19	0.5	0.14	0.5
PCB-118+106	3.21	10	0.39	1	0.12	0.5
PCB-107+108	0.86	2	0.16	0.5	0.13	0.5
PCB-110	3.94	10	0.31	1	0.06	0.2
PCB-111+117	1.33	5	0.21	0.5	0.16	0.5
PCB-112	0.34	1	0.09	0.2	0.06	0.2
PCB-113	0.34	1	0.08	0.2	0.04	0.1

Congener	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
	MDL	ML	MDL	ML	MDL	ML
PCB-114	0.28	1	0.06	0.2	0.07	0.2
PCB-119	0.42	1	0.08	0.2	0.09	0.2
PCB-122	0.19	0.5	0.07	0.2	0.05	0.2
PCB-123	0.31	1	0.09	0.2	0.06	0.2
PCB-124	0.35	1	0.08	0.2	0.06	0.2
PCB-125	0.81	2	0.07	0.2	0.05	0.2
PCB-126	0.42	1	0.07	0.2	0.10	0.2
PCB-128	1.27	5	0.08	0.2	0.08	0.2
PCB-129	0.33	1	0.07	0.2	0.08	0.2
PCB-130	0.35	1	0.07	0.2	0.06	0.2
PCB-131+142	1.46	5	0.10	0.2	0.19	0.5
PCB-132+168	1.91	5	0.18	0.5	0.14	0.5
PCB-133	0.39	1	0.07	0.2	0.07	0.2
PCB-134	0.75	2	0.08	0.2	0.06	0.2
PCB-144+135	1.26	5	0.19	0.5	0.11	0.5
PCB-136	1.39	5	0.06	0.2	0.05	0.1
PCB-137	0.38	1	0.08	0.2	0.06	0.2
PCB-138+163+164	3.95	10	0.34	1	0.17	0.5
PCB-149+139	4.98	20	0.20	0.5	0.12	0.5
PCB-140	4.00	10	0.06	0.2	0.06	0.2
PCB-141	1.35	5	0.09	0.2	0.07	0.2
PCB-143	0.40	1	0.07	0.2	0.07	0.2
PCB-145	0.43	1	0.08	0.2	0.06	0.2
PCB-146	0.57	2	0.07	0.2	0.04	0.1
PCB-147	0.30	1	0.08	0.2	0.07	0.2
PCB-148	0.44	1	0.07	0.2	0.06	0.2
PCB-150	0.46	1	0.07	0.2	0.11	0.2
PCB-151	1.97	5	0.08	0.2	0.05	0.2
PCB-152	0.50	2	0.07	0.2	0.06	0.2
PCB-153	3.90	10	0.20	0.5	0.09	0.2
PCB-154	0.42	1	0.08	0.2	0.06	0.2
PCB-155	0.43	1	0.05	0.1	0.05	0.2
PCB-156	0.37	1	0.06	0.2	0.07	0.2
PCB-157	0.60	2	0.07	0.2	0.08	0.2
PCB-158+160	0.73	2	0.12	0.5	0.13	0.5
PCB-159	0.51	2	0.06	0.2	0.06	0.2
PCB-161	0.43	1	0.07	0.2	0.07	0.2
PCB-162	0.60	2	0.06	0.2	0.05	0.2
PCB-165	1.51	5	0.07	0.2	0.04	0.1
PCB-166	0.37	1	0.08	0.2	0.09	0.2
PCB-167	0.94	2	0.06	0.2	0.06	0.2

Congener	Aqueous (ng/L)		Solid (ng/g)		Tissue (ng/g)	
	MDL	ML	MDL	ML	MDL	ML
PCB-169	0.34	1	0.10	0.2	0.06	0.2
PCB-170+190	1.95	5	0.14	0.5	0.15	0.5
PCB-171	0.60	2	0.07	0.2	0.13	0.5
PCB-172+192	0.59	2	0.13	0.5	0.12	0.5
PCB-173	0.33	1	0.07	0.2	0.21	0.5
PCB-174	3.12	10	0.09	0.2	0.08	0.2
PCB-175	0.33	1	0.07	0.2	0.08	0.2
PCB-176	0.56	2	0.06	0.2	0.07	0.2
PCB-177	1.57	5	0.07	0.2	0.06	0.2
PCB-178	0.50	2	0.09	0.2	0.09	0.2
PCB-179	1.54	5	0.05	0.2	0.05	0.2
PCB-180	0.37	1	0.07	0.2	0.19	0.5
PCB-181	3.10	10	0.07	0.2	0.08	0.2
PCB-187+182	2.28	5	0.15	0.5	0.13	0.5
PCB-183	0.92	2	0.08	0.2	0.07	0.2
PCB-184	0.49	2	0.05	0.2	0.06	0.2
PCB-185	0.36	1	0.07	0.2	0.06	0.2
PCB-186	0.35	1	0.07	0.2	0.05	0.2
PCB-188	0.39	1	0.06	0.2	0.05	0.2
PCB-189	0.26	1	0.06	0.2	0.07	0.2
PCB-191	0.22	0.5	0.07	0.2	0.05	0.2
PCB-193	0.39	1	0.07	0.2	0.06	0.2
PCB-194	3.16	10	0.18	0.5	0.10	0.2
PCB-195	0.43	1	0.07	0.2	0.08	0.2
PCB-196+203	1.13	5	0.15	0.5	0.17	0.5
PCB-197	0.43	1	0.06	0.2	0.04	0.1
PCB-198	0.80	2	0.10	0.2	0.06	0.2
PCB-199	0.88	2	0.08	0.2	0.10	0.2
PCB-200	0.44	1	0.06	0.2	0.05	0.2
PCB-201	0.59	2	0.06	0.2	0.14	0.5
PCB-202	0.26	1	0.05	0.2	0.05	0.2
PCB-204	0.65	2	0.07	0.2	0.09	0.2
PCB-205	0.75	2	0.06	0.2	0.11	0.5
PCB-206	0.64	2	0.06	0.2	0.06	0.2
PCB-207	0.62	2	0.06	0.2	0.06	0.2
PCB-208	0.90	2	0.05	0.2	0.05	0.2
PCB-209	0.50	2	0.26	1	0.09	0.2

3.3 Use Within the Spokane Watershed

Method 1628 is relatively new, and the EPA could find no examples of its use within the Spokane watershed.

4 Method 1668C

4.1 Description of Method

EPA Method 1668C determines PCB congeners in water, soil, sediment, biosolids, tissue, and other sample matrices by high resolution gas chromatography/high resolution mass spectrometry (US EPA, 2010). Using an SPB-octyl gas chromatographic column, this method can determine approximately 125 PCBs as individual congeners, including the 12 PCB designated as toxic by the World Health Organization. The remaining congeners co-elute as mixtures of isomers.

4.2 Detection and Quantitation Limits

MDLs and MLs for water and other matrices published in Method 1668c are shown in Table D-4.

Table D-4: Method Detection Limits and Minimum Levels for EPA Method 1668C.

Cl No.	Congener No.	Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
		MDL	ML	MDL	ML	ML
1	1	10	20	1.0	2	1
1	2	7	20	0.7	2	1
1	3	11	50	1.1	5	2.5
2	4	13	50	1.3	5	2.5
2	10	13	50	1.3	5	2.5
2	9	7	20	0.7	2	1
2	7	8	20	0.8	2	1
2	6	7	20	0.7	2	1
2	5	8	20	0.8	2	1
2	8	15	50	1.5	5	2.5
2	14	8	20	0.8	2	1
2	11	34	100	3.4	10	5
2	13	19	50	1.9	5	2.5
2	12					
2	13/12					
2	15	16	50	1.6	5	2.5
3	19	8	20	0.8	2	1
3	30	16	50	1.6	5	2.5
3	18					
3	30/18					
3	17	9	20	0.9	2	1
3	27	8	20	0.8	2	1
3	24	10	20	1.0	2	1
3	16	9	20	0.9	2	1
3	32	8	20	0.8	2	1
3	34	7	20	0.7	2	1
3	23	7	20	0.7	2	1
3	29	12	50	1.2	5	2.5
3	26					
3	29/26					
3	25	8	20	0.8	2	1

Cl No.	Congener No.	Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
		MDL	ML	MDL	ML	ML
3	31	18	50	1.8	5	2.5
3	28	22	50	2.2	5	2.5
3	20					
3	28/20					
3	21	21	50	2.1	5	2.5
3	33					
3	21/33					
3	22	9	20	0.9	2	1
3	36	8	20	0.8	2	1
3	39	8	20	0.8	2	1
3	38	7	20	0.7	2	1
3	35	9	20	0.9	2	1
3	37	10	20	1.0	2	1
4	54	14	50	1.4	5	2.5
4	50	25	100	2.5	10	5
4	53					
4	50/53					
4	45	22	50	2.2	5	2.5
4	51					
4	45/51					
4	46	10	20	1.0	2	1
4	52	15	50	1.5	5	2.5
4	73	14	50	1.4	5	2.5
4	43	14	50	1.4	5	2.5
4	69	26	100	2.6	10	5
4	49					
4	69/49					
4	48	14	50	1.4	5	2.5
4	65	40	100	4.0	10	5
4	47					
4	44					
4	65/47/44					
4	62	37	100	3.7	10	5
4	75					
4	59					
4	62/75/59					
4	42	16	50	1.6	5	2.5
4	41	42	100	4.2	10	5
4	71					
4	40					
4	41/71/40					
4	64	13	50	1.3	5	2.5
4	72	13	50	1.3	5	2.5
4	68	14	50	1.4	5	2.5

Cl No.	Congener No.	Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
		MDL	ML	MDL	ML	ML
4	57	11	50	1.1	5	2.5
4	58	14	50	1.4	5	2.5
4	67	12	50	1.2	5	2.5
4	63	12	50	1.2	5	2.5
4	61	59	200	5.9	20	10
4	70					
4	76					
4	74					
4	61/70/76/74					
4	66	17	50	1.7	5	2.5
4	55	12	50	1.2	5	2.5
4	56	15	50	1.5	5	2.5
4	60	14	50	1.4	5	2.5
4	80	11	50	1.1	5	2.5
4	79	13	50	1.3	5	2.5
4	78	16	50	1.6	5	2.5
4	81	18	50	1.8	5	2.5
4	77	14	50	1.4	5	2.5
5	104	14	50	1.4	5	2.5
5	96	15	50	1.5	5	2.5
5	103	11	50	1.1	5	2.5
5	94	13	50	1.3	5	2.5
5	95	77	200	7.7	20	10
5	100					
5	93					
5	102					
5	98					
5	95/100/93/102/98					
5	88	22	50	2.2	5	2.5
5	91					
5	88/91					
5	84	11	20	1.1	2	1
5	89	13	50	1.3	5	2.5
5	121	12	50	1.2	5	2.5
5	92	13	50	1.3	5	2.5
5	113	47	200	4.7	20	10
5	90					
5	101					
5	113/90/101					
5	83	29	100	2.9	10	5
5	99					
5	83/99					
5	112	14	50	1.4	5	2.5
5	119	74	200	7.4	20	10

Cl No.	Congener No.	Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
		MDL	ML	MDL	ML	ML
5	109					
5	86					
5	97					
5	125					
5	87					
5	119/109/86/97/125/87					
5	117	38	100	3.8	10	5
5	116					
5	85					
5	117/116/85					
5	110	39	100	3.9	10	5
5	115					
5	110/115					
5	82	15	50	1.5	5	2.5
5	111	14	50	1.4	5	2.5
5	120	13	50	1.3	5	2.5
5	108	29	100	2.9	10	5
5	124					
5	108/124					
5	107	17	50	1.7	5	2.5
5	123	17	50	1.7	5	2.5
5	106	17	50	1.7	5	2.5
5	118	30	100	3.0	10	5
5	122	12	50	1.2	5	2.5
5	114	15	50	1.5	5	2.5
5	105	17	50	1.7	5	2.5
5	127	14	50	1.4	5	2.5
5	126	16	50	1.6	5	2.5
6	155	14	50	1.4	5	2.5
6	152	14	50	1.4	5	2.5
6	150	15	50	1.5	5	2.5
6	136	16	50	1.6	5	2.5
6	145	16	50	1.6	5	2.5
6	148	14	50	1.4	5	2.5
6	151	46	100	4.6	10	5
6	135					
6	154					
6	151/135/154					
6	144	15	50	1.5	5	2.5
6	147	35	100	3.5	10	5
6	149					
6	147/149					
6	134	33	100	3.3	10	5
6	143					

Cl No.	Congener No.	Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
		MDL	ML	MDL	ML	ML
6	134/143					
6	139	29	100	2.9	10	5
6	140					
6	139/140					
6	131	17	50	1.7	5	2.5
6	142	17	50	1.7	5	2.5
6	132	16	50	1.6	5	2.5
6	133	12	50	1.2	5	2.5
6	165	13	50	1.3	5	2.5
6	146	14	50	1.4	5	2.5
6	161	13	50	1.3	5	2.5
6	153	30	100	3.0	10	5
6	168					
6	153/168					
6	141	17	50	1.7	5	2.5
6	130	13	50	1.3	5	2.5
6	137	15	50	1.5	5	2.5
6	164	15	50	1.5	5	2.5
6	138	63	200	6.3	20	10
6	163					
6	129					
6	160					
6	138/163/129/160					
6	158	16	50	1.6	5	2.5
6	166	29	100	2.9	10	5
6	128					
6	128/166					
6	159	14	50	1.4	5	2.5
6	162	13	50	1.3	5	2.5
6	167	13	50	1.3	5	2.5
6	156	23	100	2.3	10	5
6	157					
6	156/157					
6	169	15	50	1.5	5	2.5
7	188	15	50	1.5	5	2.5
7	179	14	50	1.4	5	2.5
7	184	14	50	1.4	5	2.5
7	176	12	50	1.2	5	2.5
7	186	15	50	1.5	5	2.5
7	178	14	50	1.4	5	2.5
7	175	14	50	1.4	5	2.5
7	187	17	50	1.7	5	2.5
7	182	13	50	1.3	5	2.5
7	183	28	100	2.8	10	5

Cl No.	Congener No.	Water (pg/L)		Other (ng/kg)		Extract (pg/µL)
		MDL	ML	MDL	ML	ML
7	185					
7	183/185					
7	174	15	50	1.5	5	2.5
7	177	11	50	1.1	5	2.5
7	181	13	50	1.3	5	2.5
7	171	30	100	3.0	10	5
7	173					
7	171/173					
7	172	13	50	1.3	5	2.5
7	192	13	50	1.3	5	2.5
7	193	30	100	3.0	10	5
7	180					
7	193/180					
7	191	13	50	1.3	5	2.5
7	170	12	50	1.2	5	2.5
7	190	14	50	1.4	5	2.5
7	189	13	50	1.3	5	2.5
8	202	24	100	2.4	10	5
8	201	20	50	2.0	5	2.5
8	204	21	50	2.1	5	2.5
8	197	43	100	4.3	10	5
8	200					
8	197/200					
8	198	37	100	3.7	10	5
8	199					
8	198/199					
8	196	20	50	2.0	5	2.5
8	203	18	50	1.8	5	2.5
8	195	22	50	2.2	5	2.5
8	194	18	50	1.8	5	2.5
8	205	15	50	1.5	5	2.5
9	208	16	50	1.6	5	2.5
9	207	19	50	1.9	5	2.5
9	206	16	50	1.6	5	2.5
10	209	16	50	1.6	5	2.5

4.3 Use Within the Spokane Watershed

A search of Washington's Environmental Information Management (EIM) database identified 20 studies within the watershed (WRIAs 54, 55, 56, and 57) which included data collected using EPA Method 1668C, as shown in Table D-5.

Table D-5: Studies in EIM Using EPA Method 1668C.

Study Name	Study ID	Field Collection Data Range
Spokane River Toxics Preliminary Monitoring 2012 through 2013 – In Support of the Long-term Toxics Monitoring Strategy	BERA0009	10/09/2012 – 06/13/2013
Spokane River PCBs and other Toxics: Long-Term Monitoring at the Spokane Tribal Boundary	BERA0012	04/29/2015 – 06/09/2016
Spokane River – PCB Atmospheric Deposition Study	BERA0013	05/11/2016 – 09/06/2017
Cochran Basin DO TMDL Stormwater Sampling	Cochran Basin TMDL	08/09/2016 – 12/07/2019
Spokane Fish Hatchery PCB Evaluation	mifr0003	04/12/2016 – 10/11/2016
PBT Chemical Trends in Washington State Determined from Age-Dated Lake Sediment Cores, 2016 Sampling Results	SEDCORE16	08/31/2016 – 09/07/2016
Spokane River Regional Toxics Task Force 2014 Synoptic Dry Weather Survey and Confidence Testing for PCBs in Surface Water	SRRTTF-2014	05/13/2014 – 08/24/2014
Spokane River Regional Toxics Task Force 2018 Continued ID of Potential Unmonitored Dry Weather Sources	SRRTTF-2018	08/04/2018 – 08/08/2018
Spokane River Urban Waters- Spokane River Source Trace Study Regarding PCB, PBDE, Metal and Dioxin/Furan Contamination	SRUW-Spokane	06/03/2009 – 08/29/2013
Spokane River Biofilm PCB Screening Study	SWON0001	08/27/2018 – 08/08/2019
Assessment of Methods for Sampling Low-Level Toxics in Surface Waters	WHOB003	06/08/2016 – 02/09/2017
Washington State Toxics Monitoring Program: Exploratory Monitoring 2012	WSTMP12	04/26/2012 – 11/05/2012
Spokane River urban Waters Investigation of PCBs in Soils and Stormwater Associated with Demolition Activities	ABOR0001	03/14/2017 – 05/31/2017
Little Spokane River PCBs in Fish Tissue Verification Study	MIFR0002	10/21/2014 – 10/22/2014
Spokane River Regional Toxics Task Force 2016 Monthly Monitoring	SRRTTF-2016	03/24/2016 – 12/13/2016
Spokane River Regional Toxics Task Force 2015 Synoptic Dry Weather Survey	SRRTTF-2015	08/18/2015 - 08/22/2015
Upriver Dam PCB Sediments Site	UPRVRDAM	05/21/2003 - 08/19/2020
Kaiser Trentwood Remedial Investigation, Spokane, WA	FS53481373	01/23/2006 - 05/23/2022
Washington State Toxics Monitoring Program: Exploratory Monitoring 2012	WSTMP12	04/26/2012 - 11/05/2012
Freshwater Fish Contaminant Monitoring Program 2013	FFCMP13	06/04/2013 - 11/13/2013

5 Method 8082A

5.1 Description of Method

EPA Method 8082A is published in the EPA's Test Methods for Evaluating Solid Waste: Physical/Chemical Methods Compendium, also known as SW-846 or the Compendium (US EPA, 2007). These methods are primarily for use with the Resource Conservation and Recovery Act (RCRA).

EPA Method 8082A uses gas chromatography with electron capture detection or electrolytic conductivity detection to determine the concentrations of PCBs as aroclors or individual PCB congeners in extracts from solid, tissue and aqueous matrices.

EPA Method 8082A has been used to determine aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260 and PCB congeners 1, 5, 18, 31, 44, 52, 66, 87, 101, 138, 141, 151, 153, 170, 180, 183, 187, and 206. The method may be appropriate for additional congeners and aroclors.

5.2 Detection and Quantification Limits

No detection or quantification limits are published directly in EPA Method 8082A. The ATSDR Toxicological Profile for PCBs lists a detection limit of 57 – 70 µg/kg for analyses of PCBs in soil using this method (6). Ecology found the method to be more sensitive for sediment analysis, with a detection limit of 904 ng/kg (0.904 µg/kg) and a quantitation limit of 5700 ng/kg (5.7 ng/kg) (Coots, 2014).

5.3 Use Within the Spokane Watershed

A search of Washington's Environmental Information Management (EIM) database identified 27 studies within the watershed (WRIAs 54, 55, 56, and 57) which included data collected using EPA Method 8082, as shown in Table D-6.

Table D-6: Studies in EIM Using EPA Method 8082.

Study Name	Study ID	Field Collection Date Range
Spokane River Urban Waters Investigation of PCBs in Soils and Stormwater Associated with Demolition Activities	ABOR0001	3/14/2017 - 5/31/2017
Spokane River Biological Effects	AJOH0019	10/23/2000 - 10/25/2000
Heglar Kronquist Landfill RI/FS, Mead, WA	AO6557	5/4/2010 - 7/28/2011
Deadman Creek - City of Mead, Spokane County - Sediment Sampling	CDC Deadman Creek	11/10/2010 - 11/11/2010
Spokane River PCB Source Assessment 2003-2007 (formerly Spokane River PCB TMDL)	DSER0010	9/15/2003 - 7/14/2004
Persistent Organic Pollutants in Feed and Rainbow Trout from Selected Trout Hatcheries	DSER0015	3/29/2005 - 6/17/2005
Evaluation of Candidate Freshwater Sediment Reference Sites	NBLA0006	7/21/2008 - 8/21/2008
Spokane River Sediments October 2000	SPOK2000	10/23/2000 - 10/25/2000
Spokane River Urban Waters-Spokane River Source Trace Study Regarding PCB, PBDE, Metal, and Dioxin/Furan Contamination	SRUW-Spokane	6/3/2009 - 8/29/2013
BNSF Hillyard Lead Soil Site	VCEA0117	9/17/2008 - 10/8/2009

Study Name	Study ID	Field Collection Date Range
Washington State Toxics Monitoring Program: Exploratory Monitoring 2002	WSTMP02	6/19/2001 - 10/31/2002
Washington State Toxics Monitoring Program: Exploratory Monitoring 2006.	WSTMP06	9/19/2006 - 12/8/2006
Washington State Toxics Monitoring Program: Exploratory Monitoring 2012	WSTMP12	4/26/2012 - 11/5/2012
Cheney Super Stop Lots 8 & 9, Cheney, WA	VCEA0281	9/30/2014 - 9/7/2017
1999 Spokane River fish and crayfish PCB'S and METALS	AJOH0022	7/27/1999 - 10/14/1999
Verification of 303(d) Listed Sites in NWRO, CRO and ERO	BERA0001	10/9/2003 - 12/3/2003
Lake Spokane PCBs in Carp	BERA0011	9/28/2014 - 9/29/2014
PCBs, PBDEs, and Selected Metals in Spokane River Fish, 2005	DSER0016	8/22/2005 - 11/3/2005
NAVY AND MARINE CORPS RESERVE READINESS CENTER SOIL EXCAVATION AND DISPOSAL IN SPOKANE, WASHINGTON	FS99996936	7/22/2016 - 7/22/2016
West Medical Lake PCBs, Dioxins and Furans in Fish, Sediment, and Wastewater Treatment Plant Effluent	RCOO0008	2/12/2008 - 10/28/2008
Metals and PCBs in Long Lake Fish	RJAC002	6/18/2001 - 6/19/2001
Spokane Area Point Source PCB Survey, May 2001	SGOL005	5/1/2001 - 5/2/2001
SS059 Fitness Center TPH Contamination	VCEA0308	2/13/2016 - 3/9/2018
Spokane Transit Authority Old Railroad Right of Way Contaminated Soil Removal	VCEA0370	6/21/2022 - 8/25/2022
Washington State Toxics Monitoring Program: Exploratory Monitoring 2003.	WSTMP03	5/27/2003 - 10/23/2003
Washington State Toxics Monitoring Program: pre-QAPP Trend Monitoring	WSTMP03T	9/17/2001 - 9/16/2003
Washington State Toxics Monitoring Program: Exploratory Monitoring 2005.	WSTMP05	3/1/2005 - 11/29/2005

6 Method 625.1

6.1 Description of Method

The EPA promulgated Method 625.1 for use in wastewater compliance monitoring under NPDES (40 CFR part 136, Appendix A). Method 625.1 can be used for determining semi-volatile organic pollutants in aqueous environmental samples using base-neutral and acid extractions followed by a gas chromatograph coupled to a mass spectrometer (GC/MS) for sample analysis and is applicable under 40 CFR 136. Method 625.1 can be used to characterize the following PCBs as aroclors (1016, 1221, 1232, 1242, 1248, 1254, 1260, 1268), however extraction and/or gas chromatography of these analytes may present challenges for quantitative determination. Consequently, other methods may be more effective for PCB analysis. See Method 625.1 for complete details about the methodology (US EPA, 2016).

6.2 Detection and Quantitation Limits

The following MDL and ML values are published for PCB-1221 (30 and 90 µg/L, respectively) and PCB-1254 (36 and 108 µg/L, respectively) in Method 625.1 (40 CFR 136, Appendix A, Table 3). Note: the MDL values published within 625.1 are unchanged from the MDLs published in Method 625, which was promulgated in 1984.

6.3 Use Within Spokane Watershed

No studies were found or identified that have used or are currently using Method 625.1 within the Spokane Watershed (WRIAs 54, 55, 56 or 57).

7 Method 6410 B-2000

7.1 Description of Method

Method 6410 B-2000 uses liquid-liquid extraction followed by gas chromatography and mass spectrometry for detecting/characterizing up to 84 semi-volatile, organic analytes including the following PCB aroclors (PCB-1016, PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260) in municipal and industrial discharges or water matrices. See the complete methodology for details (Standard Methods Committee of the American Public Health Association, American Water Works Association, and Water Environment Federation).

7.2 Detection or Quantitation Limits

Method 6410 B-2000 has MDLs published for two aroclors: PCB-1221 (30 µg/L), and PCB-1254 (36 µg/L) (NEMI).

7.3 Use Within Spokane Watershed

No studies were found using Method 6410 B-2000 in the Spokane Watershed (WRIAs 54, 55, 56 or 57).

8 Method 608: Alternate Test Method 3M0222

8.1 Description of Method

The Organochlorine Pesticides and PCBs in Wastewater Using 3M Empore Extraction Disks method (i.e., EPA Method 608- Alternate Test Method 3M 0222 or the Empore Disk Method) was approved in 1995 and is appropriate for use in NPDES permit applications (i.e., effluent testing), discharge monitoring reports, and state certification (40 CFR 136.3(a) Table ID) on a variety of matrix profiles including different wastewater sources, solids and wide-ranging pH.

EPA Method 608 – Alternate Test Method 3M 0222 was promulgated as an alternative method to EPA Method 608 to determine nineteen organochlorine pesticides and seven PCB aroclors, specifically, 1016, 1221, 1232, 1242, 1248, 1254 and 1260, using disk extraction followed by gas chromatography and an electron capture detector. See methodology for further details (US EPA 1978).

8.2 Detection or Quantitation Limits

Method 608 – Alternate Test Method 3M 0222 published a single MDL for PCB-1254 of 0.26 µg/L.

8.3 Use Within Spokane Watershed

No studies were found or identified that have used or are currently using Method 608-Alternate Test Method 3M 0222 within the Spokane Watershed (WRIAs 54, 55, 56 or 57).

9 Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater

9.1 Description of Method

Using a gas chromatograph equipped with an electron capture, microcoulometric or electrolytic conductivity detector, 29 analytes associated with pesticides and PCBs (i.e., aroclors = PCB-1016, PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260) can be determined and quantified from a liquid-liquid co-extraction originating in water and wastewater.

9.2 Detection or Quantitation Limits

No detection or quantitation limits are published within *Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater (13)*.

9.3 Use Within Spokane Watershed

No studies were found or identified that have used or are currently using this methodology within the Spokane Watershed (WRIAs 54, 55, 56 or 57).

10 References

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