Best Practices for PCB Screening of Drinking Water Compliance Samples by EPA 525.3

Introduction & Regulatory Background

PCBs in drinking water are regulated by the Safe Drinking Water Act (SDWA) via a two-step process.

- 1) Water samples are screened for PCBs using one of five available screening methods: EPA 505, 508, 508.1, 525.2, or 525.3.
- 2) If results exceed screening limits, samples are re-analyzed for compliance purposes as decachlorobiphenyl using EPA 508A, relative to the MCL of 0.5 μ g/L as decachlorobiphenyl (DCB).

Screening by EPA 505, 508, 508.1 or 525.2

Screening as Aroclors by 505, 508, 508.1

- Solvent extraction (505, 508) or SPE (508.1), GC/ECD Screening as Aroclors by 525.2
- SPE extraction, GC/MS, scan mode only
- Congeners in 525.2 are not significant components of regulated Aroclors, and may not be used for Aroclor screening

All four Aroclor screening methods require:

- Qualitative pattern recognition and multi-peak quantitation
- Separate multi-level calibration required for each regulated Aroclor (1016, 1221, 1232, 1242, 1248, 1254, 1260). 6 levels = 42 injections.
- Quantitation to the regulatory Aroclor screening MDLs. For 1221 and 1232, 40 CFR screening MDLs exceed the MCL. Screening to the MCL is recommended for these Aroclors.

Aroclor Screening MDLs, MCL equivalent values, and recommended calibration levels and reporting limits for 505, 508, 508A and 525.3						
	Required			Recommended		
	Molar	screening MDL ^b	reporting limit,			
Aroclor	mass ^a	(µg Aroclor/L)	μg Aroclor/L ^c	μg Aroclor/L		
1016	257.5	0.08	0.3	≤0.08		
1221	188.5	20	0.2	≤0.2		
1232	223	0.5	0.2	≤0.2		
1242	257.5	0.3	0.3	≤0.3		
1248	292	0.1	0.3	≤0.1		
1254	326	0.1	0.3	≤0.1		
1260	361	0.2	0.4	≤0.2		
DCB	498.6					
a. EPA 508A and reference therein						
b. 40 CFR 141.24(h)(13)(ii)						
c. (0.50 μg DCB/L) x (Aroclor MWt / DCB MWt). Rounded to 1 sig fig.						

Screening by 525.3 congener method

Screening is based on 14 PCB congeners representative of the major components, by weight %, of the seven SDWA-regulated Aroclors

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Screening as Aroclors by 525.3

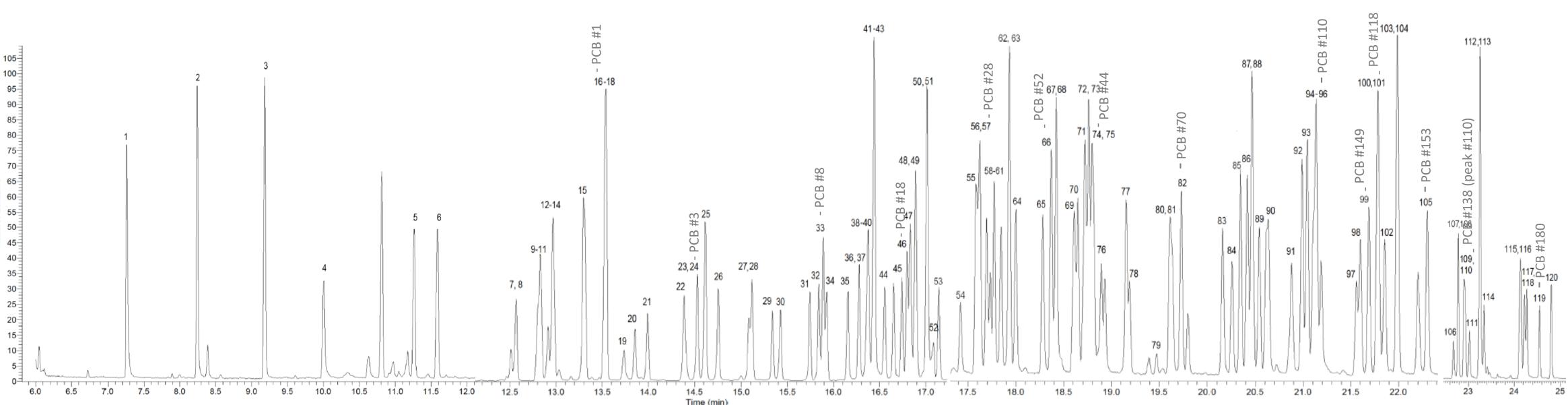
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Laboratory Services & Applied Sciences Division, Analytical Services Branch

Distribution of 525.3-monitored congeners in regulated Aroclors								
PAC#	1221	1232	1016	1242	1248	1254	1260	
1	35.8%	15.5%	0.5%	0.5%	0.04%	0.02%	0.02%	
3	20.4%	10.3%	0.2%	0.2%	0.01%	_	-	
8	12.8%	10.7%	8.3%	7.1%	0.5%	0.1%	0.04%	
18	0.78%	4.9%	10.8%	8.5%	3.8%	0.2%	0.05%	
28	0.62%	3.9%	8.5%	6.9%	4.6%	0.1%	0.03%	
44	0.21%	1.8%	4.5%	3.6%	6.3%	1.5%	0.04%	
52	0.22%	1.8%	4.6%	3.5%	6.9%	3.1%	0.2%	
70	0.24%	1.9%	0.6%	3.7%	7.3%	5.2%	0.04%	
.10	0.05%	0.4%	-	0.8%	2.8%	8.9%	1.3%	
.18	0.08%	0.3%	-	0.7%	2.3%	10.5%	0.5%	
.38	-	0.06%	-	0.1%	0.4%	5.9%	6.5%	
.49	-	0.05%	-	0.06%	0.3%	2.7%	8.8%	
.53	-	0.05%	-	0.06%	0.3%	3.5%	9.4%	
.80	-	0.01%	-	-	0.1%	0.5%	11.4%	
eraged from data in Frame et al IHRC 1996								

Averaged from data in Frame et al, JHRC 1996 Congeners > 5 wt% in bold, lower-abundance congeners in gray

- SPE extraction & GC/MS: scan, SIM or SIM/scan permitted
- No qualitative pattern recognition required
- Multi-level calibration for each congener in a mixed calibration standard. Congeners run with other method analytes. 6 levels = 6 injections.
- Quantitation to at or below the congener equivalent of the DCB MCL



Calibration, Detection Limit and Reporting Requirements

Chromatogram from 525.3 for 5 µg/mL standard. The 14 method congeners can be acquired with other method analytes in <35 minutes at method's chromatographic conditions

• Six-point calibration must include at least one point at or below the MRL

• Minimum Reporting Level (MRL) confirmation study required – 7 replicate BSs at the proposed MRL with a calculated prediction interval of results (PIR) of 50-150% recovery

• Method detection limit (MDL) study not required by method, but may be required by certification authority for drinking water applications. A best practice is to distribute MRL spike preparation and analysis across three days so data can be used to calculate an MDL.

• For each congener, the verified MRL must be \leq the MCL, expressed in μ g congener/L. As shown below, most congeners must be calibrated and reported to $\leq 0.02 \ \mu g/L$ ($\leq 0.02 \ \mu g/mL$ calibration standards, accounting for the 1000x concentration associated with the SPE preparation). Congeners 1, 110, 118, 149 and 153 must be reported to $\leq 0.03 \ \mu g/L$, and congener 180 must achieve $\leq 0.03 \ \mu g/L$.

Congener reporting limits equivalent to the MCL, μ g congener/L							
IUPAC#	1221	1232	1016	1242	1248	1254	1260
1	0.07	0.03					
3	0.04	0.02					
8	0.02	0.02	0.02	0.02			
18			0.03	0.02			
28			0.02	0.02			
44					0.02		
52					0.02		
70					0.02	0.02	
110						0.03	
118						0.03	
138						0.02	0.02
149							0.03
153							0.03
180							0.04

= (0.5 μ g DCB/L) x (MM Aroclor/MM DCB) x (congener wt% / 100) For each congener, the lowest RL associated with a given Aroclor calculation must be achieved. Results rounded to 1 significant figure.

Best Practices & Barriers to Implementation

- Method performance data tables in EPA 525.3 show that required reporting limits are readily achievable in SIM mode, but may not be routinely achievable for all required congeners in scan mode.
- Although reporting is only required to the MCL, it is a best practice to report well below the MCL. For example, reporting to the Aroclor screening MDL would require calibration and a successful MRL study to 0.01 μ g/L for most congeners. This would likely require SIM mode for some or all congeners.
- Since regulatory values do not reference congeners and conversions are not explicitly addressed in 525.3, adopting labs should consider reporting non-detected screening results as $< 0.50 \ \mu g$ DCB/L to facilitate comparison of results to the MCL.
- PCB screening by EPA 525.3 has had limited implementation. One barrier to implementation has been lack of an appropriate routine performance test sample for PCB congeners by 525.3.
 - EPA's Office of Groundwater & Drinking Water Technical Support Center personnel are actively working with vendors to establish an appropriate PT.
 - Until such a PT is established, alternatives include repurposing qualitative and/or quantitative Aroclor PTs for drinking water matrices, quantitative PCB congener PTs for wastewater matrices, analysis of certified reference materials, or interim certification without PT results.

References

- https://www.epa.gov/dwanalyticalmethods/approveddrinking-water-analytical-methods, organic contaminants, April 2019, 815-B-19-003
- PCB MCL: 40 CFR 141.61(c)(15)
- Aroclor screening MDLs: 40 CFR 141.24(h)(13)(ii)
- G.M. Frame, J.W. Cochran and S.S. Bowaldt. J. High Resol. *Chromatography*, 1996, **19**, 657-668
- EPA methods 505, 508, 508.1, 525.2, 525.3 and 508A.

Acknowledgements

The author thanks Dr. Paul Grimmett, U.S. EPA, OGWDW, Technical Support Center, for discussions regarding implementation of EPA 525.3 for PCB screening, and Dr. Carolyn Persoon, US EPA Region 5 Lab Services & Applied Sciences Division for comments.