

# PCB Disposal Rulemaking: Updates to Extraction and Analytical Methods for PCBs

April 2024

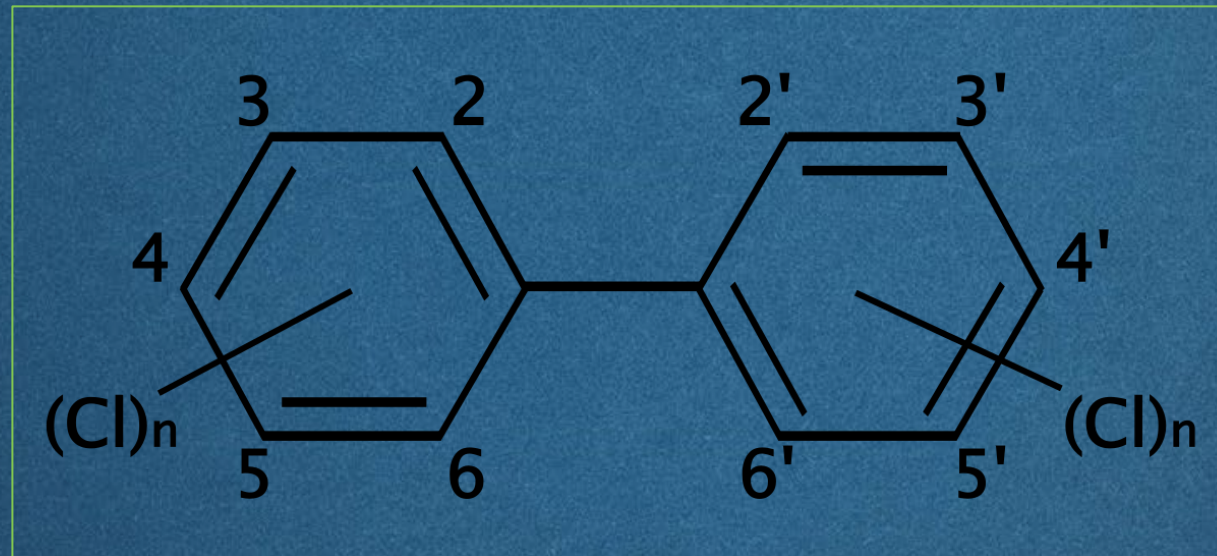


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Our *Not So Emerging*  
Contaminant.....Polychlorinated  
Biphenyls (PCBs)



# Agenda

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- Introduction
- Why Change the Methods?
- PCB Cleanup and Disposal Program
- PCB Extraction Methods – Solid Matrices
- PCB Extraction Methods – Aqueous Matrices
- PCB Determinative Methods – Solid Matrices



# Introduction

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- David Blye – Environmental Chemist
  - 40 years experience
  - SME on PCB, Dioxin/Furan and PFAS analytical chemistry
  - Broad background on various regulatory programs and contaminant types.
  - Forensics
- Retained by USWAG to evaluate USEPA's proposed changes in TSCA Part 761 to the PCB extraction and analytical methods and to comment to the Docket
- Public comments are available in the Federal Register.  
<https://www.regulations.gov/docket/EPA-HQ-OLEM-2021-0556>





# Why Change the Methods?

The PCB methods were limited and outdated

- Did not provide for use of newer, current methods
- Specific method versions were codified in the regulations
- The regulations were not updated with newer methods since 1979!!

SW-846 METHOD STATUS TABLE (Cont.)

(Date in parenthesis is found at bottom right-hand corner of method).								METHOD TITLE
THIRD ED (9/86)	FIN. UP. I (7/92)	FIN. UP. II (9/94) IIA (8/93) IIB (1/95)	FIN. UP. III (12/96) IIIA (4/98) IIIB (11/04)	FIN. UP. IV (2/07)	UPDATE V (07/14)	UPDATE VI	UPDATE VII	
--	8070	--	8070A (Up. III)	--	--	--	--	Nitrosamines by Gas Chromatography
--	--	8081 (Up. II)	8081A (Up. III)	8081B	--	--	--	Organochlorine Pesticides by Gas Chromatography
--	--	--	8082 (Up. III)	8082A	--	--	--	Polychlorinated Biphenyls (PCBs) by Gas Chromatography
--	--	--	--	8085	--	--	--	Compound-independent Elemental Quantitation of Pesticides by Gas Chromatography with Atomic Emission Detection (GC/AED)
--	--	--	8091 (Up. III)	--	--	--	--	Nitroaromatics and Cyclic Ketones by Gas Chromatography
--	--	--	--	8095	--	--	--	Explosives by Gas Chromatography
8100	--	--	--	--	--	--	--	Polynuclear Aromatic Hydrocarbons
--	--	--	8111 (Up. III)	--	8111	--	--	Haloethers by Gas Chromatography
--	--	8121 (Up. II)	--	--	--	--	--	Chlorinated Hydrocarbons by Gas Chromatography: Capillary Column Technique
--	--	--	8131 (Up. III)	--	--	--	--	Aniline and Selected Derivatives by Gas Chromatography
8140	--	--	Deleted (Up. III)	--	--	--	--	Organophosphorus Pesticides
--	8141	8141 (Up. II)	--	8141B	--	--	--	Organophosphorus Compounds by Gas Chromatography
--	--	8151 (Up. II)	8151A (Up. III)	--	--	--	--	Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization

# PCB Cleanup and Disposal Program

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“The PCB Cleanup and Disposal Program benefits communities by ensuring that sites contaminated with PCBs are cleaned up to reduce risks and by ensuring that materials contaminated with PCBs are safely managed and disposed of in landfills or destroyed in other types of waste management units.”



# PCB Cleanup and Disposal Program

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Sooooooooo ,

- It only makes sense to use current methods over outdated and antiquated methods!!
- The addition of the new methods was a good thing!!
- The proposed addition of two determinative methods was not appropriate, and USWAG comments helped prevent their inclusion in the final rule.



# PCB Extraction Methods – Solid Matrices

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For extraction of PCBs from solid matrices, EPA proposed to add (*and DID*):

- SW-846 Method 3541 (Automated Soxhlet Extraction),
- SW-846 Method 3545A (Pressurized Fluid Extraction), and
- SW-846 Method 3546 (Microwave Extraction).

For extraction of PCBs from solid matrices, EPA proposed to remove:

- SW-846 Method 3550B (Ultrasonic Extraction).
- Comments were made concerning the inconsistent performance and robustness of Ultrasonic Extraction
- EPA is allowing Method 3550C for wipe samples only.





# PCB Extraction Methods – Aqueous Matrices

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For extraction of PCBs from aqueous matrices, EPA proposed to add (*and DID*):

- SW-846 Method 3510C (Separatory Funnel Liquid-Liquid Extraction),
- SW-846 Method 3520C (Continuous Liquid-Liquid Extraction), and
- SW-846 Method 3535 (Solid-Phase Extraction).



# PCB Determinative Methods

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EPA proposed to add three determinative methods to the PCB regulations:

- SW-846 Method **8082A** (Polychlorinated Biphenyls (PCBs) By Gas Chromatography),
- SW-846 Method 8275A (Semivolatile Organic Compounds (PAHs And PCBs) In Soils/Sludges and Solid Wastes Using Thermal Extraction/Gas Chromatography/Mass Spectrometry (TE/GC/MS)), and
- Clean Water Act (CWA) Method 1668C (Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS).



# PCB Determinative Methods

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- SW-846 Method **8082A** – Aroclor Method was added.
- SW-846 Method 8275A – not added.
- Method 1668C – 209 PCB Congeners – not added
- Although EPA did not add Method 8275A and Method 1668C, “..the agency notes that these methods, as well as other methods that have been published since the proposed rule, such as CWA Method **1628**, may be appropriate and useful in certain situations.”



TABLE 1—TABLE OF EPA METHODS DISCUSSED IN THIS RULEMAKING

Source	Method ID	Publication year	Method type	Method name	Final change
SW-846	Method 3510C <sup>6</sup>	1996	Extraction	Separatory Funnel Liquid-Liquid Extraction.	Added to Regulations.
SW-846	Method 3520C <sup>7</sup>	1996	Extraction	Continuous Liquid-Liquid Extraction.	Added to Regulations.
SW-846	Method 3535A <sup>8</sup>	2007	Extraction	Solid-Phase Extraction (SPE).	Added to Regulations.
SW-846	Method 3500B	2007	Extraction	Organic Extraction and Sample Preparation.	Removed from Regulations.
SW-846	Method 3540C	1996	Extraction	Soxhlet Extraction	Remains in Regulations.
SW-846	Method 3541 <sup>9</sup>	1994	Extraction	Automated Soxhlet Extraction.	Added to Regulations.
SW-846	Method 3545A <sup>10</sup>	2007	Extraction	Pressurized Fluid Extraction.	Added to Regulations.
SW-846	Method 3546 <sup>11</sup>	2007	Extraction	Microwave Extraction	Added to Regulations.
SW-846	Method 3550B	1996	Extraction	Ultrasonic Extraction	Updated to Method 3550C and Limited to Wipe Samples Only.
SW-846	Method 3550C	2007	Extraction	Ultrasonic Extraction	Replaces Method 3550B and Limited to Wipe Samples Only.
SW-846	Method 8082	1996	Determinative	Polychlorinated Biphenyls (PCBs) by Gas Chromatography.	Removed from Regulations.
SW-846	Method 8082A	2007	Determinative	Polychlorinated Biphenyls (PCBs) by Gas Chromatography.	Added to Regulations.
SW-846	Method 8275A	1996	Extraction and Determinative.	Semivolatile Organic Compounds (PAHs and PCBs) in Soils/Sludges and Solid Wastes Using Thermal Extraction/Gas Chromatography/Mass Spectrometry (TE/GC/MS).	Not Added to Regulations.
CWA	Method 1668C	2010	Extraction and Determinative.	Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS.	Not Added to Regulations.
CWA	608	2006	Extraction and Determinative.	Organochlorine Pesticides and PCBs.	Updated to CWA Method 608.3.
CWA	608.3	2016	Extraction and Determinative.	Organochlorine Pesticides and PCBs by GC/HSD.	Replaces CWA Method 608.



# PCB Determinative Methods

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- SW-846 Method 8275A – not added.
- Don't ever use.....
  - NO commercial labs even do it!!!
  - Select – 19 – PCB congeners – CAN'T GET TOTAL PCBs
  - Only solid matrices, no aqueous
  - Miniscule sample size - 0.003-0.250 grams
    - Poor representativeness and poor method precision
  - Sample introduction prone to contamination
  - Wet samples are dried and sieved – LOSS of PCBs
  - Requires use of surrogates, none specified



# PCB Determinative Methods

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- Method 1668C – 209 PCB Congeners – not added
- USWAG comments kept it out of the final rule!!
  - OVERLY sensitive. ppq-water; ppt-solids
  - Never promulgated under CWA
  - No defined, uniform way to get Total PCB.
  - Blank Contamination
  - No soil or sediment in the interlab validation study
  - Calibration technique leads to estimated results for some congeners. Isotope dilution for some, not all!
  - Lacks chromatographic resolution criteria
  - Mass interference from higher CBs to lesser chlorination level CBs.
- Environmental Standards full report critiquing M1668 submitted.
- No improvements by EPA since 2010.



# PCB Determinative Methods

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- Two other PCB methods worth quickly mentioning.
- CWA Method 608.3 –PCB Homologs
  - Aqueous matrices only
- CWA Method 1628 – PCB Congeners
  - Not yet promulgated, but EPA will likely attempt to do so in 2024??



# Thank you

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# QUESTIONS?



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