



## **PCB Laboratory Analysis**



## AIM

The aim is to inform about common procedures for polychlorinated biphenyls (PCBs) analysis in dielectric oils using gas chromatography.



## PERFORMANCE

PCB analysis with gas chromatography should be performed by individuals formally trained in at least the basic principles of chemical analysis and in the use of gas chromatography.



- To separate target compounds from matrix
- Sample Preparation
  - Sample weighing
  - Extraction
  - Clean-up
  - Concentration/dilution
- Instrumental analysis



Cost per sample can vary between 100-400 USD


# Existing Methods on PCB analysis

Examples:

- EPA Method 8082A in association with e.g. EPA Method 3580A (extraction by dilution in organic solvent) and with Method 3620C (Florisil Cleanup)
- UNEP SOP for PCB in human milk, air, blood with GC-ECD
- ASTM D4059 – 00(2010) – PCBs in insulating liquids by GC
- ISO 13876:2013 (Determination of PCB by GC-MS)

Parameters and analytical conditions described in these methods can be changed for your laboratory, while still obtaining the same results.

However, it is important that your method is optimized and validated to ensure the comparability of data.

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# SW-846 Test Method 8082A: Polychlorinated Biphenyls (PCBs) by Gas Chromatography

The following document discusses the procedure used to determine the concentrations of PCBs as Aroclors or as individual PCB congeners in extracts using open-tubular, capillary columns with electron capture detectors (ECDs) or electrolytic conductivity detectors (ELCDs).

You may need a PDF reader to view some of the files on this page. See EPA's [About PDF page](#) to learn more.

- For free online
- For high contaminated samples
- Aroclors or individual PCB congeners
- Detection with electron capture detectors (ECDs) or electrolytic conductivity detectors (ELCDs)
- Extraction techniques for aqueous, solid and tissue sample
- Recommended PCB extraction techniques for dielectric oils are e.g.:
  - Method 3580A (extraction by dilution in organic solvent)
  - Method 3535 (solid-phase extraction)
- Recommended clean-up methods are summarized in Method 3600 e.g. Method 3620C (Florisil Cleanup)
- Offers guide for quality control and assurance

- Available for free online
- Developed for trace analysis in human milk, air and human serum
  - not specifically developed for oil samples
- For the determination of the six indicator PCBs and 14 OCPs
- Detection with electron capture detectors (ECDs)
- Offers guide for quality control and assurance



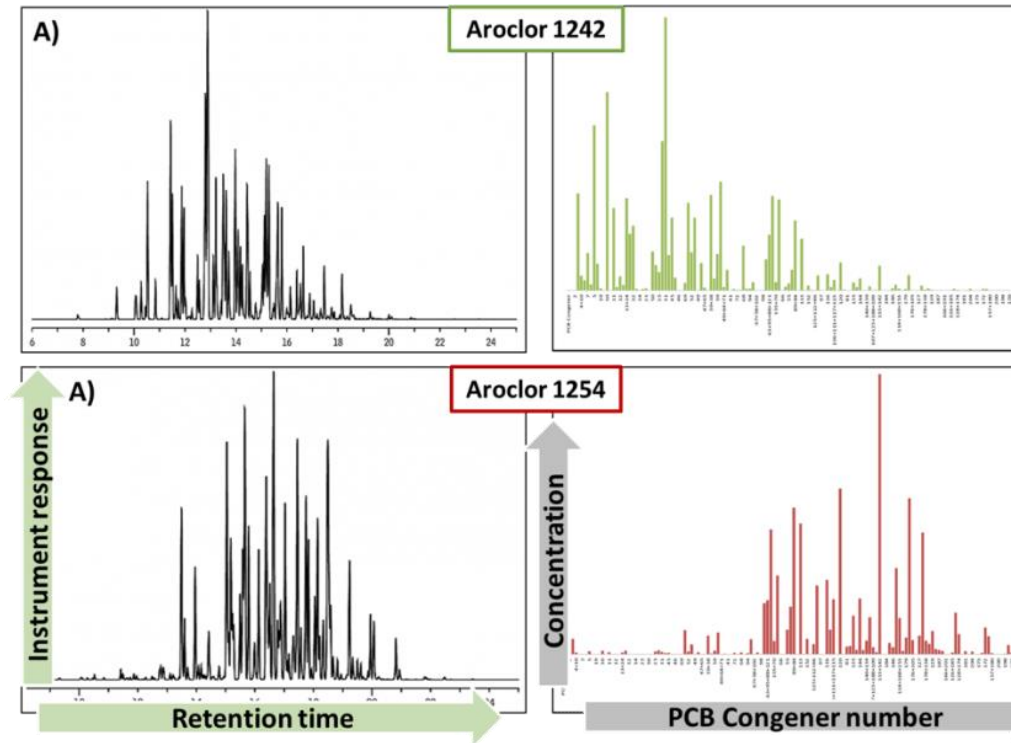
## Global Monitoring Plan on Persistent Organic Pollutants

### Protocol 2: Protocol for the Analysis of Polychlorinated Biphenyls (PCB) and Organochlorine Pesticides (OCP) in Human Milk, Air and Human Serum

# Target PCBs: Arochlors

**Arochlors 1242, 1254, 1260**

Arochlors are multi-component mixtures of PCBs





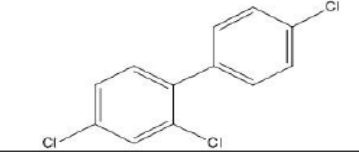
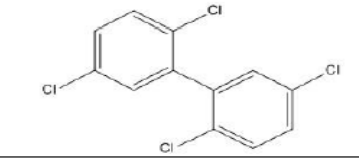
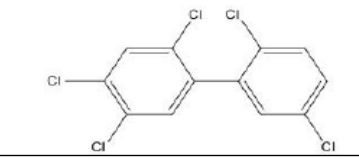
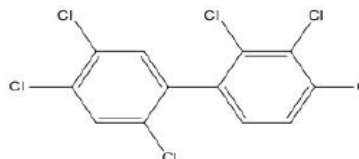
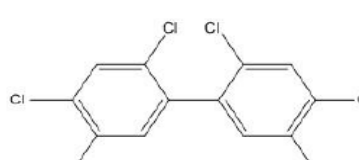
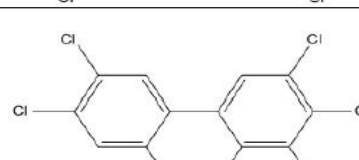
# Target PCBs: Congeners

Six indicator PCBs defined by the EU:

- PCB 28 2,2',4-trichlorobiphenyl
- PCB 52 2,2',5,5'-tetrachlorobiphenyl
- PCB 101 2,2',4,5,5'-pentachlorobiphenyl
- PCB 138 2,2',3,4',5,5'-hexachlorobiphenyl
- PCB 153 2,2',4,4',5,5'-hexachlorobiphenyl
- PCB 180 2,2',3,4,4',5,5'-heptachlorobiphenyl



Congeners are chemical compounds that have similar structures and similar properties.

PCB 28	
PCB 52	
PCB 101	
PCB 138	
PCB 153	
PCB 180	

- Reagent-grade or pesticide-grade chemicals must be used in all tests
  - Common solvents: n-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane
- Use appropriate glassware or plastic containers

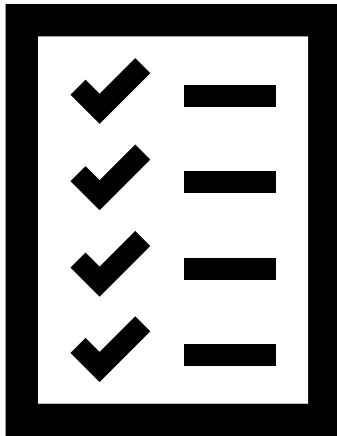
Solvents and materials used during the analysis must be tested to prove they do not contain any PCBs!

# Precautions

- Laboratory coat
- Nitrile gloves
- Safety glasses
- Organic solvents mask

Handle samples only under an extractor hood!





## Internal standard

- When PCB congeners are to be determined, the use of an internal standard is highly recommended
- For example: Decachlorobiphenyl may be used as an internal standard, added to each sample extract prior to analysis, and included in each of the initial calibration standards

## Surrogate standard

- The performance of the method should be monitored using surrogate compounds. The choice of surrogate compounds will depend on analysis mode chosen, e.g., Aroclors or congeners.

# Suitable Instruments

- Gas chromatography coupled to:
  - An electron capture detectors (ECD)
  - An electrolytic conductivity detectors (ELCD)
  - A mass spectrometers (MS)
- Columns: capillary columns with silica/siloxane
- Each laboratory must determine retention times and retention time windows for their specific application of the method.
- The instrument must be calibrated for the target analytes when it is used for quantitative analysis.
- Ideally when target PCB are analyzed on GC/ECD, then the analyst must demonstrate that those PCBs identified can be confirmed by GC/MS and vice versa.



# Calibration

- Prepare calibration standards
- For analysis of PCB **congeners**, the use of **internal standard calibration** is highly recommended
- For analysis of PCB as **Aroclors**, **external standard calibration** is generally used
- Multi-point calibration that includes all target PCBs should be used
- Verify calibration at least once each 12-hr shift prior to sample analyses.
- A calibration standard must also be injected at intervals of not less than once every twenty samples

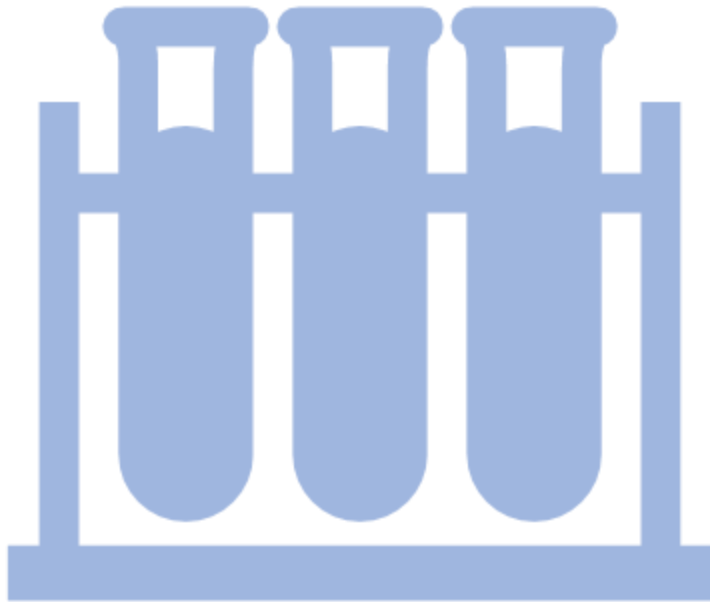


All reagents, disposals and sample residues that have been in contact with PCBs during analysis are considered as **hazardous waste**.

Laboratories require good waste management practices that are consistent with all applicable rules and regulations.



# Quality control and Quality assurance



The laboratory must also have procedures for method performance on precision, accuracy, method sensitivity.

At a minimum, this should include the analysis of :

- QC samples
- method blanks
- a matrix spike
- a duplicate sample
- laboratory control sample (LCS)

All in each analytical batch and the addition of surrogates to each field sample and QC sample is recommended.

All should be subjected to the same analytical procedures as the actual samples.



# Method performance



The laboratory should:

- regularly perform analyses on certified reference materials (CRM)
- participate in proficiency testing (e.g. interlaboratory testing, ILS)
- ideally, get an international certification such as ISO to demonstrate its competence

A photograph of three business professionals in a light blue office setting. In the foreground, three hands are raised, each giving a thumbs-up gesture. The hands are of different skin tones, representing diversity. The background shows the lower faces and upper bodies of three people: a man on the left, a woman in the center, and a man on the right, all smiling.

**It is important that your method is approved by national authorities.**



**Thank you for your  
attention !**

<https://www.pcb.unitar.org/>

