Multi-Laboratory Validation of Low Resolution GC-MS SIM PCB Congener Method

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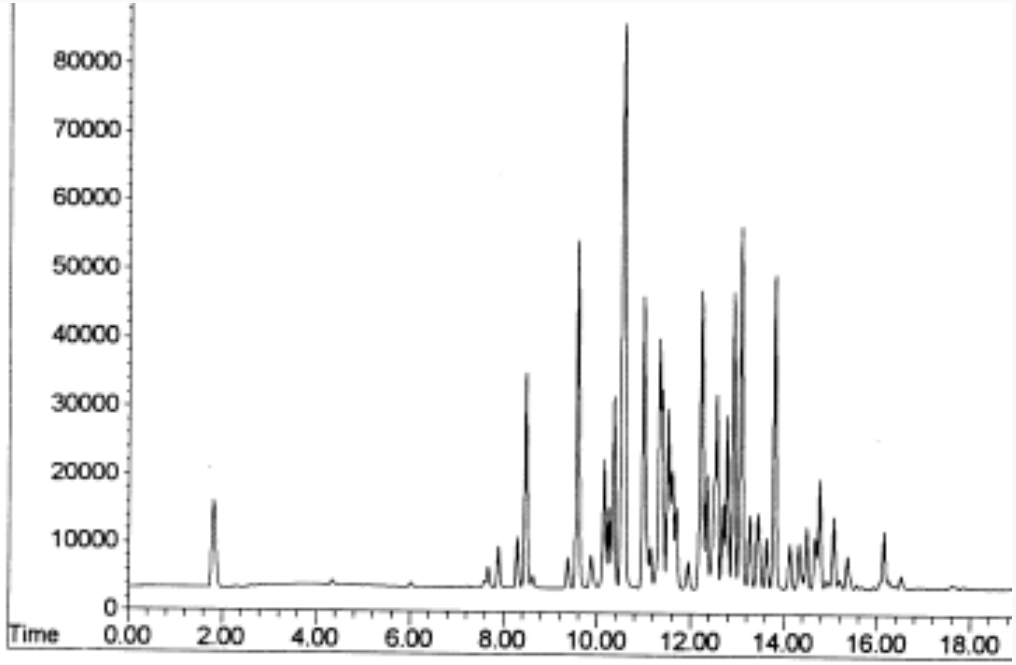
Goal

- Validate a low-resolution GC/MS method for PCB congeners in wastewater and other matrices
- Criteria to meet:
  1. *Identifies and quantifies PCBs using individual congeners, not Aroclors*
  2. *More sensitive than Method 608.3, but not too sensitive (i.e. background contamination issues)*
  3. *Can be implemented at a typical mid-sized full-service environmental laboratory*

Method 608.3



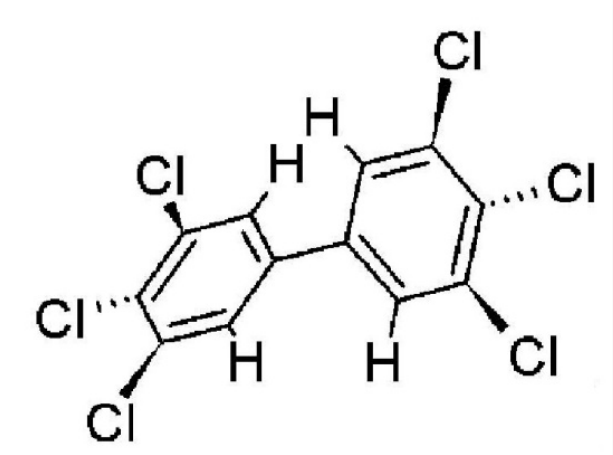
- Only measures the 7 common Aroclor mixtures, not congeners
- Detection Limit: 65 ng/L
- Approximately $80-120 per sample
- Currently the only promulgated method for PCBs at 40 CFR 136; the only NPDES regulations are for Aroclors.

Approach

Method focuses on specific congeners, but detects all

Focus:

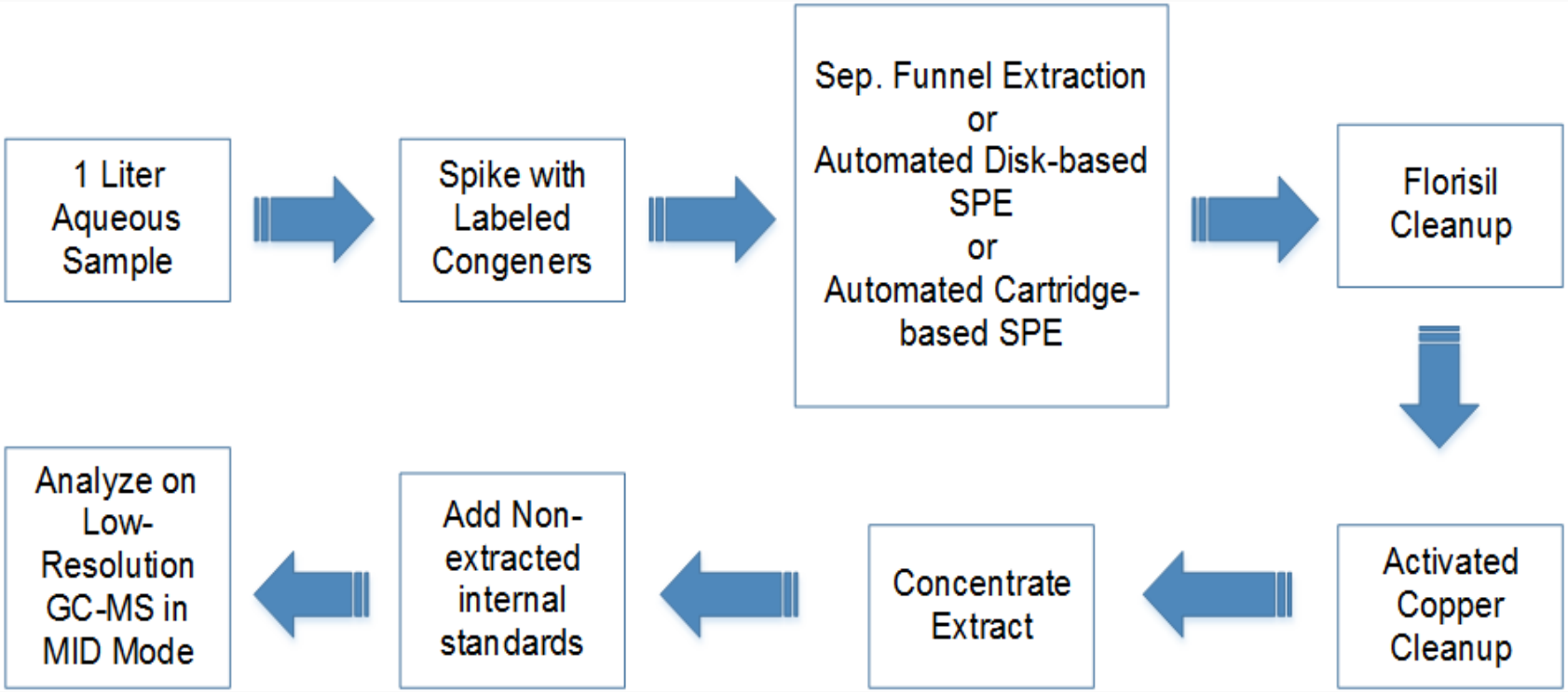
1. First and last eluter of each homolog
2. Most common in environment
3. Prevalent in human tissue
4. Present in Aroclors in large quantities
5. WHO Toxic Congeners



Summary of Method Steps

- Measure sample aliquot
- Spike sample (including QC) with labeled congeners
- Extract
- Cleanup
- Concentrate
- Add non-extracted internal standards
- Analyze by low-resolution GC/MS with SIM

Aqueous Samples



Other Matrices

- Solids samples (soils, sediments, and biosolids) are spiked with the labeled compounds, extracted by Soxhlet, and extracts cleaned with:
  - Silica gel
  - Alumina
  - Florisil
  - Activated copper
- Tissue samples are spiked with the labeled compounds, extracted by Soxhlet, and extracts cleaned with:
  - GPC
  - Florisil

Quantification

Analysis

- DB-5 column
- EI using SIM detection (2 ions per congener)
- 167 peaks for 209 congeners

Quantification

- 48 congeners are calibrated
  - 23 congeners by true isotope dilution
  - 6 congeners by isotope dilution with 8 co-eluters
  - 19 congeners by extracted internal standard (EIS) quantification with 9 coeluters
- Remaining 144 congeners quantified indirectly

Single-Lab Study Summary

Single-laboratory validation study met EPA's goals

1. Method identifies and quantifies PCB contamination using individual congeners, not an estimated quantity based off patterns generated from Aroclor mixtures
2. Method is more sensitive than currently approved Method 608.3, but not so sensitive to be adversely affected by typical laboratory background contamination
3. Can be implemented at a typical mid-sized full-service environmental laboratory

Multi-laboratory Validation Study

- Participants
  - 8 contracted laboratories
  - 4 volunteer laboratories
- Real-world Matrices
  - Wastewater (9)
  - Biosolids (3)
  - Sediment (3)
  - Fish tissue (3)

Custom Standards Provided

- 209 congener mix
  - Commercially available 9-standard set
- Initial calibration standards (6)
  - 48 Natives, 32 ^{13}C labels
- Labeled compound standards
  - 29 ^{13}C labels
- Native standards – 48 congeners
- Internal standards – 3 ^{13}C labels

Required Analyses

- Retention time determination for all 209 congeners
- Initial calibration of 48 congeners
- Method detection limits for all 209 congeners
- Initial precision and recovery for 48 congeners
- Unspiked sample analyses for all 209 congeners
- Matrix spikes and matrix spike duplicates on all samples, using 57 congeners (48 calibrated congeners and 9 additional congeners commonly detected)

Study Progress

- 7 of the initial 12 laboratories completed all aspects of the wastewater portion of the study
  - The labs that dropped out cited time and resource issues, not capability
- Fewer laboratories had agreed to the analyze the other matrices
  - 6 laboratories completed the soil/sediment portion of the study
  - 4 laboratories completed the biosolids portion of the study
  - 4 laboratories completed the fish tissue portion of the study
- Obtained enough data to develop statistically based QC criteria for IPR and OPR analyses, and to develop pooled MDLs
- Obtained enough data to meet the study design for all of the matrices

Report Results

- Wastewater, Sediment, Biosolids, Fish Tissue
  - Pooled MDLs
  - IPRs Recoveries and Reproducibilities
  - MS/MSDs Recoveries and Reproducibilities

Next Steps

- Complete the statistical analyses and QC acceptance criteria development
- Finish report
- Submit draft report for:
  - Workgroup review, internal peer review, management review
- Update draft method with QC acceptance criteria and performance data from the study

Contact Information

For more information or additional feedback contact:

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