

## Organochlorine Pesticides Analysis by Gas Chromatography/ Micro Electron Capture Detector

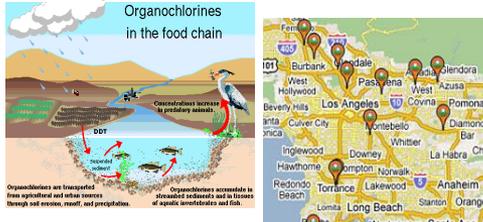
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### OBJECTIVE

- To determine the concentrations of various organochlorine pesticides in extracts from solid matrices using a GC/ $\mu$ ECD.
- To provide credible data from the Performance Evaluation (PE) sample, supported with evidence of the reliability of the data reported. To verify and maintain the high level quality in all analytical analyses using the Quality Assurance (QA) and Quality Control (QC) guidelines established by the Environmental Protection Agency (EPA).

### MOTIVATION

- In 1962, a non-fiction book called "Silent Spring" by Rachel Carson was published. It was widely credited with helping launch the environmental movement.
- It documented detrimental effects of pesticides on the environment, particularly on birds and stated that uncontrolled and unexamined pesticide use was harming and killing both animals and humans.



- Superfund sites allow the EPA to identify, investigate, and clean up uncontrolled or abandoned hazardous waste sites and to compel responsible parties to perform cleanups.
- The mission of the Department of Toxic Substances Control (DTSC) is to protect the people and the environment of the State of California by preventing hazardous wastes into landfill and providing proper disposal of hazardous waste.

### PROCEDURE

#### Method 3540C Soxhlet Extraction

- "Spiking" standards were prepared using serial dilution using hexane as a solvent. The standards contain known amounts of pesticides. All QC samples were prepared using 10g of a certain matrix and 3g of sodium sulfate. They were then placed in the soxhlet extractor.



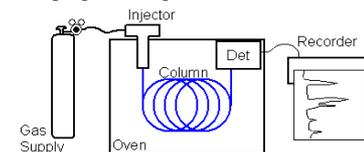
- Methylene chloride solvent is boiled and vapor travels through extraction tube in to condenser tube.
- Cold water condenses the vapor, which drips into thimble, interacting with the compounds.
- Reflux occurs allowing the desired compound to be concentrated in flask.

- The non-soluble portion of the extract remains in the thimble, which is discarded.
- The solvent is put in a hot water bath to remove methylene chloride.
- Extract goes through second solvent exchange with Hexane.
- Placed in Nitrogen evaporator to concentrate to a final volume.



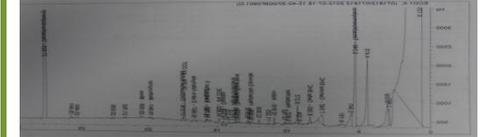
#### Method 8081B Organochlorine Analysis by GC/ $\mu$ ECD

- Prepared six level calibration standards ranging from 2.5ppb to 400ppb. Transferred about 1ml from each sample and standards into 2ml vile and prepared sequence for minimum calibration requirements.
- Sample is volatilized by heat of the oven & carried into column by Helium gas.
- Chemicals with lower boiling points travel quickly, while chemicals with higher boiling points travel slowly.
- Each will elute from column at different time (Retention Time)
- The ECD uses an electron emitter along with makeup gas to ionize the carrier gas and produce a current between a pair of electrons.
- Halogenated molecules, such as organochlorine pesticides, enter detector field and capture electrons, becoming charged.
- Captured electrons are less mobile than free and therefore neutralize with positive ions, current fall in detector measured as the analyte signal.
- Analyte concentration is proportional to degree of electron capture.
- Signal is reported to recorder, and a gas chromatogram is produced.



### RESULTS

#### Method Blank Chromatogram



#### Environmental Sample Chromatogram



#### Performance Evaluation Sample Results

Name	Result (ug/kg)	Acceptance Window	Evaluation
Alpha BHC	126	53.6 to 263	Acceptable
Beta BHC	489	111 to 729	Acceptable
Gamma BHC	<2.50	0.00 to 0.00	Acceptable
Delta BHC	349	114 to 595	Acceptable
Heptachlor	<2.50	0.00 to 0.00	Acceptable
Aldrin	<2.50	0.00 to 0.00	Acceptable
Heptachlor Epoxide	253	117 to 442	Acceptable
Gamma Chlordane	164	85.3 to 278	Acceptable
Endosulfan I	48.7	31.9 to 176	Acceptable
Alpha Chlordane	65.6	27.7 to 121	Acceptable
Dieldrin	156	69.9 to 287	Acceptable
4/4 DDE	308	151 to 572	Acceptable
Endrin	236	93.3 to 310	Acceptable
Endosulfan II	<2.50	0.00 to <5.0	Acceptable
4/4 DDD	82	17.6 to 154	Acceptable
Endrin Aldehyde	23.3	10.4 to 114	Acceptable
Endosulfan Sulfate	175	51.0 to 350	Acceptable
4/4 DDT	106	28.3 to 194	Acceptable
Endrin Ketone	202	74.0 to 405	Acceptable
Methoxychlor	235	28.2 to 458	Acceptable

### CONCLUSION

- Overall, the PE samples passed the acceptable criteria for organochlorine pesticides.
- Some interferences may include: contamination in solvents, reagents, glassware, and other sample processing hardware; inexperience with analytical procedure; time constraint.
- Future work would be to repeat the entire procedure including sample prep, extraction, and sample analysis.

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