ANALYTICAL COMPARISONS OF PESTICIDES ANALYZED BY ISOTOPE DILUTION HRGC/HRMS PBMS METHODOLOGY AND BY TRADITIONAL GC-ECD METHODOLOGY ON SEDIMENT SAMPLES FROM THE CAPE FEAR RIVER

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Introduction

Organochlorine pesticides have long been identified as toxic persistent chemicals. Many of them were manufactured decades ago but are still found in the environment today. These chemicals are routinely analyzed in sediments, tissue and remediation programs. The traditional method used to analyze chlorinated pesticides utilized a Gas Chromatograph equipped with an Electron Capture Detector (GC-ECD). The GC-ECD technique can achieve relatively low detection limits but does not have the ability to discriminate co-extracted halogen containing compounds from the target organochlorine pesticides. Recently an EPA approved isotope dilution method was developed to analyze an extended list of pesticides by HRGC/HRMS called EPA Method 1699. SGS-Analytical Perspectives has developed a true Isotope Dilution method that utilizes all of the concepts of Performance Based Measurement Systems (PBMS). This method uses some of the principles detailed in EPA Method 1699 and includes method modifications for better performance.

The use of the HRGC/HRMS has been a focal point for the ultra trace determination of chlorinated dioxins and furans (PCDDs/PCDFs), polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs) as well as other persistent compounds. Organochlorine pesticides are now on the forefront of many ultra trace studies that surround sediment, tissue, water as well as the benthic organism. The challenges associated with the analysis of pesticides range from compounds being highly susceptible to degradation due to heat and chemical reactions and the lack of aggressive cleanup procedures that can be performed to minimize interferences.

Materials and methods

Sediment samples collected from the Cape Fear river were analyzed for organochlorine pesticides by traditional EPA Method 8081B that utilizes GC-ECD analysis and the data was compared with the same samples analyzed for organochlorine pesticides by HRGC/HRMS using a true performance based approach to give concentrations yielding the greatest accuracy available. The samples analyzed by EPA Method 8081B utilized standard soil cleanup procedures and analysis as well as a portion of the same sample that through the maximum cleanup procedures and analysis. The samples analyzed by HRGC/HRMS utilized the maximum cleanup procedures and was analyzed by isotope dilution with the highest quality and performance possible.

Results and discussion

Results from all of the analyses were compared in this study. The discussion includes the impact from the matrix and how the impact relates to being able to achieve accurate results. The HRGC/HRMS method includes the following compounds:

1234-Tetrachlorobenzene	a-BHC	Endosulfan II	Mirex
1245-Tetrachlorobenzene	b-BHC	Endosulfan Sulfate	Oxychlordane
2,4'-DDD	g-BHC (Lindane)	Endrin	Pentachloroanisole
2,4'-DDE	d-BHC	Endrin Aldehyde	Pentachlorobenzene
2,4'-DDT	Chloropyrifos (Dusrban)	Endrin Ketone	cis-Nonachlor
4,4'-DDD	cis-Chlordane	Heptachlor	trans-Nonachlor
4,4'-DDE	trans-Chlordane	Heptachlor Epoxide (isomer B)	Kepone (Chlordecone)
4,4'-DDT	Dieldrin	Hexachlorobenzene	Isodrin
Aldrin	Endosulfan I	Methoxychlor	

A subset of these analytes was determined by GC-ECD due to method limitations.

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References

EPA Method 8081B: Organochlorine Pesticides by Gas Chromatography, February 2007

EPA Method 1699: Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, December 2007