Analytical Methods

Below are links to the analytical methods used by the current and previous contract laboratories used by the Analytical Control Facility (ACF) to do sample analyses.

To view all the methods for a given lab, or a specific method, click one the lab names below.

Organic and Inorganic laboratories currently under contract with ACF.		
Lab ID	Lab Name	Analyses
GERG	Geochemical and Environmental Research Group	Organic
LET	Laboratory and Environmental Testing	Inorganic
MSCL	Mississippi State Chemical Lab	Organic
ACF	Analytical Control Facility	Organic & Inorganic
RTI	Research Triangle Institute	Inorganic
TDI	TDI - Brooks International, Inc.	Organic
TERL	Trace Element Research Laboratory	Inorganic

Lab Methods for laboratories no longer under contract with ACF.		
Lab ID	Lab Name	Analyses
AXYS	Analytical Services, Ltd.	Organic
BRND	Brooks - Rand	Inorganic
ETSL	Environmental Trace Substance Laboratory	Organic & Inorganic
GERG	Geochemical and Environmental Research Group	Inorganic
HES	Hazleton Environmental Services, Inc	Organic & Inorganic
MRI	Midwest Research Institute	Inorganic

The following labs were used in the past by ACF, but their method descriptions are not available on our website.

Lab ID	Lab Name
BION	Bionetics
BRCE	PWRC Contaminant Ecology Group
BRRA	PWRC Risk Assessment Group
BRWT	PWRC Research Wildlife Toxicology Group

CBL	Chesapeake Biological Laboratory
CNFR	Columbia National Fisheries Contaminants Research Center
EPA	Environmental Protection Agency
FGS	Frontier Geo Sciences
ITC	International Technology Corporation
PWRC	Patuxent Analytical Chemistry Group
TLI	Triangle Laboratories
UMI	University of Miami
VERS	Versar, Inc.
WSU	Wright State University

Analytical Control Facility (ACF) Laboratory Methods

Select one of the links below to display the method descriptions associated with ACF or <HERE> to go to the Start Page.

Method Code	Method Title
001	Organophosphate/Carbamate Scanning for Tissue and Sediment
002	Organophosphate/Carbamate Scanning for Water
003	Aromatic Hydrocarbons for Sediment
004	Organochlorines for Tissue
005	Organochlorines for Sediment
006	ICP Scan for Water
007	ICP Scan for Tissue
008	ICP Scan for Soil/Sediment
009	Arsenic and Selenium for Tissue
010	Arsenic and Selenium for Water
011	Arsenic and Selenium for Sediment
012	Mercury for Sediment and Tissue
013	Total Suspended Solids (TSS)
014	Aluminum In Water
015	HGA: Sediment
016	Plant and Animal Tissues For Organochlorine Pesticides, Co-Planar and Other PCBs.
017	Mercury
018	Quantifying PCB Aroclors
020	Percent Moisture
021	Temephos in Water
022	Temephos in Soil/Sediment
023	Temephos in Tissue
024	Total Organic Carbon

025 Grain Size Analysis

026 White Phosphorus In Tissue

027 ICP Scan-Tissue

028 HGA Analytes in Tissue

029 Mercury in Tissue

030 Percent Lipid Determinations

031 Moisture by Freeze Drying

032 Total Petroleum Hydrocarbon in Soils/Sediments

033 Total Petroleum Hydrocarbon in Tissue

034 Percent Food Content in Digestive Tract

035 Temephos (Abate) in Tissue, Plants, Water and Sediment.

036 ICP-MS Scan Tissue

Lab Name: *Analytical Control Facility* Method Code: *001*

Analytical Methodology for Organophosphate/Carbamate Scanning for Tissue and Sediment

This method involves homogenization of the sample followed by mixing with acetone and methylene chloride to separate the pesticides from the tissue. The organic extract is filtered and adjusted to volume prior to gas chromatography using a flame photometric detector for organophosphate determinations and a nitrogenphosphorus detector for carbamate determinations. Magabore capillary columns are used for the GC separations.

Reference:

1. Patuxent Wildlife Research Center, Analytical Chemistry Group SOP Organophosphate/Carbamate Scanning Method (0-25.00). April 28, 1989.

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Lab Name: *Analytical Control Facility* Method Code: *002*

Analytical Methodology for Organophosphate/Carbamate Scanning for Water

The extraction procedure was a modified version of the one described in the EPA test Method #608, Section 10 (1). The water samples were extracted three times with methylene chloride (all containers were rinsed three times as well). The methylene chloride extracts were combined, dried with sodium sulfate and concentrated on a rotary evaporator. Sample extracts were refrigerated prior to analysis. Residues were quantified by gas chromatography using either an instrument selective for organophosphate pesticides (Flame Photometric Detector) or one selective for carbamate pesticides (Nitrogen Phosphorus Detector) similar to Belisle et al. (1988) (2). Megabore capillary columns were used for the GC separation.

References

1. EPA test Method #608, Section 10. (sample extraction) 1982.

2. Belisle, A.A., and D.M. Swineford. 1988. Simple, specific analysis of organophosphorous and carbamate pesticides in sediments using column extraction and gas chromatography. Environ. Toxicol. Chem. 7(9):749-752.

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Lab Name: *Analytical Control Facility* Method Code: *003*

Analysis for Aromatic Hydrocarbons for Sediment

Ten gram homogenized aliquots of sediment were mixed with sodium sulfate and placed into a glass Soxhlet extraction thimble. Prior to extraction 1.6 ug of each of the following deuterated internal (surrogate) aromatic hydrocarbon standards were added to sample: d8 napthalene, d10 acenapthylene, d10 phenanthrene, d12 chrysene, and d12 perylene. These internal standards wereused to quantitate the aromatic hydrocarbon residues. Samples were extracted in the same manner as for pesticides (Cromartie et. al., 1975) except that petroleum ether was used instead of hexane as an extracting solvent. After extraction samples were concentrated to 10 mL using an air stream and treated with gel permeation chromatography (GPC) for cleanup. Samples were then concentrated to 1 mL and 1 mL of 1 ppm pyrene-d10 was added as a GC internal standard in order to monitor the recoveries of deuterated PAHs. Samples were quantified by GC/MS using Selective Ion Monitoring (Wade et al., 1988).

The GC/MS used was a Hewlett Packard 5970 MSD and 5890 gas chromatograph equipped with a 59970 ChemStation computer data system. The GC column used was a 50m bonded methyl phenyl (5%) silicone column, 0.32 ID, 0.25 micron film thickness. The GC conditions were: injection port - 300oC, transfer line 280oC. Temperature program: Initial temperature 100oC for 1 min followed by 7oC/min to 290oC and hold for 40 min.

Calibration is accomplished by integration of single ions characteristic of each compound using the five deuterated internal standards. The compounds, ions used, and standards used are shown below:

Compound	Ion	Standard	Ion
Naphthalene	128	Naphthalene-d8	136
Acenaphthylene	152	Acenaphthylene-d10	164
Acenaphthene	154	Acenaphthylene-d10	164
Fluorene	166	Acenaphthylene-d10	164
Phenanthrene	178	Phenanthrene-d10	188
Anthracene	178	Phenanthrene-d10	188
Fluoranthene	202	Phenanthrene-d10	188
Pyrene	202	Chrysene-d12	240
Benzo(a)anthr.	228	Chrysene-d12	240

Compound	Ion	Standard	Ion
Chrysene	228	Chrysene-d12	240
Benzo(b)Fluor.	252	Perylene-d12	264
Benzo(k)Fluor.	252	Perylene-d12	264
Benzo(e)pyrene	252	Perylene-d12	264
Benzo(a)pyrene	252	Perylene-d12	264
Perylene	252	Perylene-d12	264
Indeno(123cd)per.	276	Perylene-d12	264
Benzo(ghi)per.	276	Perylene-d12	264
Dibenzo(ah)anth.	278	Perylene-d12	264

The lower detection limit is 0.01 ppm based on a 10 g sample wet weight.

References

1. Cromartie, E.W., W.L. Reichel, L.N. Locke, A.A. Belisle, T.E. Kaiser, T.G. Lamont, B.M. Mulhern, R.M. Prouty, and D.M. Swineford. 1975. Residues of organochlorine pesticides and polychlorinated biphenyls and autopsy data for Bald Eagles, 1971-72. Pestic. Monit. J. 9:11-14.

2. Wade, T.L., Atlas, E.L., Brooks, J.M., Kennicutt, M.C. II, Fox, R.G., Sericano, J., Garcia, B. and Defreitas, D. 1988. NOAA Gulf of Mexico Status and Trends Program: Trace organic contaminant distribution in sediments and oysters. Estuaries 11, 171-179.

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Lab Name: *Analytical Control Facility* Method Code: *004*

Analytical Methodology for Organochlorines for Tissue

The analytical methods, including preparation, Soxhlet extraction, and lipid removal are described by Cromartie et al., 1975. Glass extraction thimbles were used. The silica gel separation of the pesticides from PCBs was different from the above reference in that four fractions were used instead of three to enable the separation of dieldrin and endrin from the rest of the pesticides. The pesticides in each fraction were quantified with a gas-liquid chromatograph (GLC), equipped with a 63Ni electron capture detector. The GLC column used was a 30m MEGABORE coated with a 1.0 micron film of 7% cyanopropyl 7% phenyl polysiloxane. Residues in 10%

of the samples were confirmed by gas chromatography/mass spectrometry (GC/MS). The nominal lower limit of detection is 0.01 ppm for pesticides and 0.05 ppm for PCBs based on a 10 g aliquot wet weight.

Reference

1. Cromartie, E.W., W.L. Reichel, L.N. Locke, A.A. Belisle, T.E. Kaiser, T.G. Lamont, B.M. Mulhern, R.M. Prouty, and D.M. Swineford. 1975. Residues of organochlorine pesticides and polychlorinated biphenyls and autopsy data for Bald Eagles, 1971-72. Pestic. Monit. J. 9:11-14.

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Lab Name: *Analytical Control Facility* Method Code: *005*

Analytical Methodology for Organochlorines for Sediment

The Soxhlet extraction procedure was a modified version of the one described by Cromartie et al. (1975). Ten gram aliquots of moist soil were mixed together in activated florisil and ignited sea sand. The samples were then placed into a glass extraction thimble and extracted eight hours via Soxhlet extraction using an 8/1/1 hexane - acetone - methanol solution. The extracts were then extracted three times with water and the organic layers were then combined and cleaned up with florisil (Cromartie et. al.). The pesticides were separated from the PCBs using silica gel column chromatography as described by the above reference except that two fractions were used instead of three (Pesticide and PCB). Samples were quantified by a gas-liquid chromatograph (GLC), equipped with a 63Ni electron capture detector. The GLC column used was a 30m MEGABORE coated with a 1.0 micron film of 7% cyanopropyl 7% phenyl polysiloxane. Residues were confirmed in one of the samples by gas chromatography/mass spectrometry (GC/MS).

Reference

1. Cromartie, E.W., W.L. Reichel, L.N. Locke, A.A. Belisle, T.E. Kaiser, T.G. Lamont, B.M. Mulhern, R.M. Prouty, and D.M. Swineford. 1975. Residues of organochlorine pesticides and polychlorinated biphenyls and autopsy data for Bald Eagles, 1971-72. Pestic. Monit. J. 9:11-14.

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Lab Name: *Analytical Control Facility* Method Code: *006*

Analytical Methodology for ICP Scan for Water

ICP analyses were performed using the instrumental conditions recommended by the manufacturer (1). 100 grams of water was combined with 10 mL of concentrated nitric acid and evaporated gently in a teflon beaker to approximately 10 mL. Contents were then transferred to a 20 mL polypropylene centrifuge tube. 100 uL of 10,000 ppm scandium solution was added. The final volume of the tube was brought up to 20 mL with reverse phase osmosis deionized water. The analyses were performed using a Perkin Elmer Plasma II sequential inductively coupled plasma emission spectrometer.

Reference

1. Perkin Elmer Corporation. 1985. Plasma II Emission Spectrometer. Vol. 1-3.

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Lab Name: *Analytical Control Facility* Method Code: *007*

Analytical Methodology for ICP Scan for Tissue

ICP analyses were performed using the dry ash procedure described by Haseltine et al., 1981 (1) and the instrumental conditions recommended by the manufacturer (2). Up to 5 grams of tissue were weighed into a Vycor crucible (for other metals except boron) or a Coors crucible (boron only). The crucible was placed in a drying oven overnight at 105oC for moisture determination. After weighing the dried material, the crucible was then placed in a muffle furnace. The temperature was set at 200oC and was raised at a rate of 50oC per hour until the temperature was at 550oC. After ashing overnight the residues were dissolved in nitric and hydrochloric acids and transferred to a 15 mL polypropylene centrifuge tube. 500 micrograms of a scandium internal standard was added and the sample diluted to 10 mL with reverse osmosis deionized water. The analyses were performed using a Perkin Elmer Plasma II sequential inductively coupled plasma emission spectrometer.

Reference

1. Haseltine, S.D., G.H. Heinz, W.L. Reichel, and J.F. Moore. 1981. Organochlorine and Metal Residues in Eggs of Waterfowl Nesting on Islands in Lake Michigan Off Door County, Wisconsin. Pestic. Monit. Jour. 15(2):90-97.

2. Perkin Elmer Corporation. 1985. Plasma II Emission Spectrometer. Vol. 1-3.

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Lab Name: *Analytical Control Facility* Method Code: *008*

Analytical Methodology for ICP Scan for Soil/Sediment

ICP analyses were performed using a 3:1 hydrochloric acid:nitric acid digestion procedure and the instrumental conditions recommended by the manufacturer (1). One gram aliquots were placed into 300 mL teflon beakers and 20 mL of 3:1 hydrochloric acid:nitric acid were added. Approximately 11 mL of acid was slowly evaporated off via hot plate. The sample was then transferred into a 50 mL centrifuge tube and 100 uL of 10,000 ppm scandium solution was added. The final volume of the tube was brought up to 20 mL with reverse phase osmosis deionized water. The analyses were performed using Perking Elmer Plasma II sequential inductively coupled plasma emission spectrometer.

Reference

1. Perkin Elmer Corporation. 1985. Plasma II Emission Spectrometer. Vol. 1-3.

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Lab Name: *Analytical Control Facility* Method Code: *009*

Analytical Methodology for Arsenic and Selenium for Tissue

Samples were analyzed for selenium and arsenic according to the method described by Krynitsky (1987). A 0.5 gram fresh weight aliquot of tissue was used for digestion in 5 ml of nitric acid. Determination was by Stabilized Temperature Platform Graphite Furnace Atomic Absorption Spectroscopy using Zeeman effect background correction. The nominal detection limit was 0. 1 ppm on a wet weight basis.

Reference

1. Krynitsky, A.J. 1987. Preparation of biological tissue for determination of arsenic and selenium by graphite furnace atomic absorption spectrometry. Analytical Chemistry 59(14):1884-1886.

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Lab Name: *Analytical Control Facility* Method Code: *010*

Analytical Methodology for Arsenic and Selenium for Water

Water samples were analyzed for selenium and arsenic in a similar way as described by Krynitsky (1987). However, 100 grams of water was combined with 10 mL of concentrated nitric acid and evaporated gently in a teflon beaker to a final volume of 20 mL. Determination was by Stabilized Temperature Platform Graphite

Furnace Atomic Absorption Spectroscopy using a Perkin-Elmer Zeeman Model 3030 Graphite Furnace.

Reference

1. Krynitsky, A.J. 1987. Preparation of biological tissue for determination of arsenic and selenium by graphite furnace atomic absorption spectrometry. Analytical Chemistry 59(14):1884-1886.

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Lab Name: *Analytical Control Facility* Method Code: *011*

Analytical Methodology for Arsenic and Selenium for Sediment

Sediment samples were analyzed for selenium and arsenic according to the method described by Krynitsky (1987). A 0.5 gram fresh weight aliquot of sediment was used for digestion in 5 mL nitric acid and 0.5 mL of 30% hydrogen peroxide. However, the sediment samples needed further treatment with 5 mL hydrofluoric acid in order to free the analyte from the silicates. Determination was by Stabilized Temperature Platform Graphite Furnace Atomic Absorption Spectroscopy using a Perkin-Elmer Zeeman Model 3030 Graphite Furnace. The nominal detection limit was 0.1 ppm based on a 0.5 g wet weight basis.

Reference

1. Krynitsky, A.J. 1987. Preparation of biological tissue for determination of arsenic and selenium by graphite furnace atomic absorption spectrometry. Analytical Chemistry 59(14):1884-1886.

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Lab Name: *Analytical Control Facility* Method Code: *012*

Analytical Methodology for Mercury for Sediment and Tissue

One gram aliquots were digested under reflux in sulfuric and nitric acids as described by Monk. The determination was performed by cold vapor atomic absorption spectrophotometry using a Spectro Products mercury analyzer equipped with a Varian VGA-76 vapor generation accessory. The nominal lower limit of detection is 0.05 ppm on a wet weight basis.

Reference

1. Monk, H.E. 1961. Recommended methods of analysis of pesticide residues in food stuffs. Report by the Joint Mercury Residue Panel. Anal. 82:608-614.

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Lab Name: *Analytical Control Facility* Method Code: *013*

Analytical Methodology for Total Suspended Solids (TSS)

Preweigh empty beaker. Add 20gm of the sample to a tared beaker. Allow the aliquot to go to dryness.

TSS = weight of beaker + aliquot weight - weight of empty beaker.

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Lab Name: *Analytical Control Facility* Method Code: *014*

Aluminum In Water

Summary: Water samples were analyzed for aluminum. 100 grams of water was combined with 10 ml of concentrated nitric acid and evaporated gently in a teflon beaker to a final volume of 20 ml. Determination was by Stabilized Temperature Platform Graphite Furnace Atomic Absorption Spectroscopy using a Perkin-Elmer Zeeman Model 3030 Graphite Furnace. The nominal detection limit was 0.0005 ppm based on a 100 gram sample of water.

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Lab Name: *Analytical Control Facility* Method Code: *015*

HGA: Sediment

Sediment samples were prepared according to the method described by Krynitsky (1987). A 0.5-1.0 gram fresh weight aliquot was used for sample digestion in 5 ml of nitric acid and approximately 500 ul of 30% hydrogen peroxide was added to remove excess organic matter. The sample tubes and graphite furnace cups were prewashed in dilute nitric and hydrochloric acid prior to use. Determination was by Stabilized Temperature Platform Graphite Furnace Atomic Absorption Spectroscopy.

Reference:

1. Krynitsky, A.J. 1987. Preparation of biological tissue for determination of arsenic and selenium by graphite furnace atomic absorption spectrometry. Analytical Chemistry 59(14):1884-1886.

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Lab Name: *Analytical Control Facility* Method Code: *016*

Procedures for Analyzing Plant and Animal Tissues For Organochlorine Pesticides, Co-Planar and Other PCBs.

The preparation methods include those described by Cromartie et al., 1975. The co-planar method was described by Chia-Swee Hong and Brian Bush at the Wadsworth Center for Laboratories and Research. Homogenized animal or plant tissue is extracted with a 6% ethyl ether/hexane mixture. The extract is cleaned up on a florisil column by eluting with the 6% ethyl/hexane mixture. The PCB fraction is separated from the other compounds by silica gel column chromatography. Non-ortho and mono-ortho PCB co-planars are separated from other PCB congeners by HPLC using a column of 1:12 mixture of activiated carbon and silica gel. The pesticides and PCBs in each fraction are quantified with a gas liquid chromatography (GLC), equipped with a 63 Ni electron capture detector. The GLC column used with a 30m Megabore, coated with 1.0 micron film of 7% cyano propyl, 7% phenyl polysiloxane. Co-planars and other PCBs are analyzed on Ultra-2, 50m, I.D. 0.20 mm, film thickness 0.33 microns. Residues in 10% of the samples were confirmed by gas chromatography/mass spectrometry (GC/MS). The nominal lower limit of detection is 0.01 ppm for pesticides 0.05 ppm for PCBs and 0.005 ppm for co-planar PCBs on a 10g aliquot wet weight.

Reference

1. Cromartie, E.W., W.L. Reichel, L.N. Locke, A.A. Belisle, T.E. Kaiser, T.G. Lamont, B.M. Mulhern, R.M. Prouty, and D.W. Swineford. 1975. Residues of Organochlorine Pesticides and Polychlorinated Biphenyls and Autopsy Data for Bald Eagles, 1971-72. Pest. Mont. J. 9:11-14.

2. Chemosphere, Vol. 21, Nos. 1-2, pp. 173-181, 1990 (Printed in Great Britain).

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Lab Name: *Analytical Control Facility* Method Code: *017*

Analytical Methodology For Mercury

One half to one gram of tissue or sediment homogenate or 10 grams for water was digested under reflux in sulfuric and nitric acids as described by Monk. The mercury determination was by cold vapor atomic absorption spectroscopy as described by Hatch and Ott modified for use with a Perkin Elmer Atomic Absorption Spectrophotometer 3100 equipped with a Perkin Elmer FIAS 200.

Reference

1. Monk, H.E. 1961. Recommended methods of analysis of pesticide residues in food stuffs. Report by the Joint Mercury Residue Panel. Anal., 86:608-614.

2. Hatch, W.R., and W.L. Ott. 1968. Determination of submicrogram quantities of mercury by atomic absorption spectrophotometry. Perkin Elmer Corporation Manual. April 1991. Flow Injection Analysis System.

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Lab Name: *Analytical Control Facility* Method Code: *018*

Quantifying PCB Aroclors

Samples were extracted by Soxhlet extraction with hexane, cleaned up by Florisil and silica gel fractionation per Kaiser et al. (1980). Limits of detection were 0.01 ppm for OC pesticides and 0.05 ppm for PCBs. Four sectors of the chromatogram were used to estimate the total amount of Aroclors 1242, 1248, 1254, and 1260 in the sample. The amount of 1242, e.g., was estimated by comparison with Aroclor 1242 using decachlorobiphenyl as an internal standard. The amount of 1242 standard represented by its selected area e.g., was calibrated by quantifying the percent of the total area of 1242 that was located in its selected sector and multiplying this value by the total amount of 1242 injected.

Reference

1. Kaiser, T.E., W.L. Reichel, L.N. Locke, E. Cromartie, A.J. Krynitsky, T.G. Lamont, B.M. Mulhern, R.M. Prouty, and D.M. Swineford. 1980. Organochlorine pesticides, PCB, and PBB residues and necropsy data for bald eagles from 29 states -- 1975-1977. Pesticides Monitoring Journal 13:145-149.

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Lab Name: *Analytical Control Facility* Method Code: *020*

Analytical Methodology for Percent Moisture

Preweigh pan. Add aliquot to tared pan. Allow sample to dry 24 hours in an oven at 200 degrees Fahrenheit. Samples are then placed in a desiccator to cool. Record pan + dry weight.

Percent Moisture = 1 - (pan + dry - pan weight / original aliquot) * 100.

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Lab Name: *Analytical Control Facility* Method Code: *021*

Temephos in Water

The entire sample, including the container, was extracted by shake-out with 3x100 ml portions of methylene chloride. The extracts were pooled, concentrated, cleaned up by Florisil column chromatography, and analyzed by gas chromatography on a DB-1 megabore column using a flame photometric detector.

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Lab Name: *Analytical Control Facility* Method Code: *022*

Temephos in Soil/Sediment

A 10 gram sample is mixed with 10 or more grams of sand and 20 or more grams of Sodium Sulfate. The mixture is added to a chromatography column over a 40 gram bed of Sodium Sulfate. The column is eluted with a 200ml mixture of 1:1 Acetone/Methylene Chloride at a rate of 5ml/min. The 200ml mixture is captured in round bottom flask, fitted to a Rota- Evap and concentrated to approximately25ml. The 25ml extract is transferred to a beaker with several rinses of Methylene Chloride and further concentrated to approximately 5ml in a warm water bath held at 45oC. The extract is transferred to a mills tube and diluted to a final volume of 10 ml. The extract is analyzed by Gas Chromatography/Flame Photometric Detection.

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Lab Name: *Analytical Control Facility* Method Code: *023*

Temephos in Tissue

This method involves homogenization of the sample followed by mixing with acetone and methylene chloride to separate the pesticides from the tissue. The organic

extract is filtered, cleaned up by Florisil column chromatography, and adjusted to volume prior to gas chromatography using a flame photometric detector for organophosphate determinations. DB-1 megabore capillary columns are used for the GC separations.

Reference

1. Patuxent Wildlife Research Center, Analytical Chemistry Group SOP Organophosphate/Carbamate Scanning Method (0-25.00). April 28, 1989.

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Lab Name: *Analytical Control Facility* Method Code: *024*

Total Organic Carbon

Total Organic Carbon (TOC) is determined by chemical oxidation of the organic carbon of a sample in an ampule. The sample is acidified, persulfate is added and the sample is purged of Total Inorganic Carbon (TIC). The ampule is sealed and placed in a oven or water bath. The strong oxidant quickly reacts with organic carbon at 100 C to CO2. When the oxidation is complete, the ampule is broken and the CO2 is purged, concentrated by trapping, desorbed, and carried into a non- dispersive infrared detector (NDIR). The measured mass of CO2 is equal to the mass of TOC in the sample.

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Lab Name: *Analytical Control Facility* Method Code: *025*

Grain Size Analysis

A 15 to 20 gram homogenized sample is placed in a large glass jar. The sample is treated with 50 to 100 ml of Hydrogen Peroxide to oxidize organic matter. The sample is washed with distilled water to remove soluble salts. 400 ml of Sodium Hexametaphosphate (5.5g/L) is added to disperse the sample. The sample is shaken for 24 hours. After shaking, the sample is filtered through a 62.5 micron screen into a 1000 ml graduated cylinder. The sample is rinsed with dispersant in order that all fine-grain sediment is washed into the cylinder. This separates the gravel sand fraction (on the screen) from the silt/clay fraction (in the cylinder). The coarse fraction is washed into a pre-weighed beaker with distilled water and placed in a 100 degree oven for 24 hours. The sample is allowed to cool. The sample is weighed and weights recorded on the data sheets.

The silt/clay size material is determined by settling. A graduated cylinder is filled to exactly 1 liter. The sample is stirred and left to stand for one day. The sample is stirred vigorously. After removing the stirring rod, a 20 ml aliquot is taken from a depth of 20 cm after 20 seconds. This is the 4 phi aliquot and the total weight of the silt + clay fraction. The aliquot is placed in a pre-weighed beaker. At the 2 hour and 3 minute time another 20 ml aliquot is taken at the 10 cm depth. This is the 8 phi aliquot. Pipette the suspension into a different pre-weighed beaker. The beakers are dried overnight and cooled. The beakers are weighed, and the percent sand, silt, and clay fractions are determined.

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Lab Name: *Analytical Control Facility* Method Code: *026*

White Phosphorus In Tissue

A 10-g sample of tissue is placed into a 120-ml vial or jar containing 10.0 ml of isooctane and 10.0 ml of degassed water. The sample is vortex-mixed for 1 min., then placed horizontally on a platform shaker for 18 hr. or overnight. The sample is then allowed to stand vertically for 15 min. to allow phase separation. A 3.0-ul aliquot of the isooctane layer is analyzed on gas chromatograph equipped with a nitrogen-phosphorus detector.

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Lab Name: *Analytical Control Facility* Method Code: *027*

Analytical Methodology for ICP Scan-Tissue

Summary: Samples were lyophilized using a Labconco, Freeze Dry System, Model 7756. A one half to one gram aliquot of freeze dried aliquot was digested in 10 ml ultrapure nitric acid and 4 ml of 30% hydrogen peroxide using an OI Model 7301 Microwave Digestion System. After digestion a scandium internal standard was added. The analyses were performed using a Perkin Elmer Plasma II sequential inductively coupled plasma emission spectrometer.

Reference

1. Labconco, Freeze Dry System Model 77560 Instruction Manual 1993.

2. OI Analytical, Microwave Model 7301, Instruction Manual 1992.

3. Perkin Elmer Corporation Manual. 1985. Plasma II Emission Spectrometer. Vol. 1-3.

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Lab Name: *Analytical Control Facility* Method Code: *028*

Analytical Methodology For HGA Analytes in Tissue

Summary: Samples were lyophilized using a Labconco, Freeze Dry System Model 77560. A one half to one gram freeze dried aliquot was digested in 10 ml of ultrapurenitric acid and 4 ml of 30% hydrogen peroxide using an OI Analytical Model 7301 Microwave digestion System. Determination was by Stabilized Temperature Platform Graphite Furnace Atomic Absorption Spectroscopy using a Perkin Elmer Zeeman 3030 Atomic Absorption Spectrophotometer.

Reference

1. Labconco, Freeze Dry System Model 77560 Instruction Manual 1993.

2. OI Analytical, Microwave Model 7301, Instruction Manual 1992.

3. Atomic Spectroscopy. Vol. 4 #3, "Recent Experiences with the Stabilized Temperature Platform Furnace and Zeeman Background Correction ." Slavin, W., G.R. Carnrick, D. C. Manning, and E. Pruszkowska.

4. Perkin Elmer Corporation Manual. Atomic Absorption Spectrophotometer, Zeeman 3030.

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Lab Name: *Analytical Control Facility* Method Code: *029*

Analytical Methodology for Mercury in Tissue

Summary: Samples were lyophilized using a Labconco, Freeze Dry System Model 77560. A one half to one gram aliquot of freeze dried tissue was digested in 10 ml ultrapure nitric acid and 4 ml of 30% hydrogen peroxide using an OI Analytical 7301 Microwave digestion System. The mercury determination was by cold vapor atomic absorption spectroscopy as described by Hatch and Ott modified for use with a Perkin Elmer Atomic Absorption Spectrophotometer 3100 equipped with a Perkin Elmer FIAS 200.

Reference

1. Labconco, Freeze Dry System Model 77560 Instruction Manual 1993.

2. OI Analytical, Microwave Model 7301, Instruction Manual 1992.

3. Hatch, W.R., and W.L. Ott, 1968. Determination of submicrogram quantities of mercury by atomic absorption spectrophotometry.

4. Perkin Elmer Corporation Manual. April 1991. Flow Injection Analysis System.

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Lab Name: *Analytical Control Facility* Method Code: *030*

Percent Lipid Determinations

Summary: A portion of the extracted lipid solution is placed on a pre- weighed aluminum pan. The solvent is evaporated from pan and the pan is reweighed. The percent lipid is calculated accordingly as described in detail below.

Calculations:

PE = Pan Empty

PF = Pan Full after solvent has been evaporated

AL = Sample Aliquot

% Lipid = [(PF - PE) * 10 / AL) * 100

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Lab Name: *Analytical Control Facility* Method Code: *031*

Analytical Methodology Moisture by Freeze Drying

Summary: Empty sample jars and their lids were weighed(WEIGHT1). Up to 20 G of sample homogenate were weighed(WEIGHT2) into each jar. Samples were lyophilized using a Labconco, Freeze Dry System Model 77560. After drying, the samples, their jars and lids were weighed(WEIGHT3).

Percent Moisture = (1 - (WEIGHT3 - WEIGHT1) / WEIGHT2) * 100.

Reference

1. Labconco, Freeze Dry System Model 77560 Instruction Manual 1993.

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Lab Name: *Analytical Control Facility* Method Code: *032*

Total Petroleum Hydrocarbon in Soils/Sediments

A 10-g sample of soil/sediment is mixed with anhydrous sodium sulfate placed in an extraction thimble and extracted with methylene chloride using a soxhlet extractor. The extract is dried and concentrated for analysis. The extract is analyzed by Gas Chromatography/Flame Ionization Detection. The resulting capillary column profile is compared against hydrocarbon standards used to identify and calculate the TPH concentrations.

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Lab Name: *Analytical Control Facility* Method Code: *033*

Total Petroleum Hydrocarbon in Tissue

A 1-10g sample of tissue (depending on lipid content) is mixed with anhydrous sodium sulfate, placed in an extraction thimble and extracted with Methylene Chloride using a soxhlet extractor. The extract is concentrated and put on a florisil column. The column is eluted with 200 ml of Methylene Chloride and concentrated for analysis. The extract is analyzed by Gas Chromatography/Flame Ionization Detection. The resulting capillary column profile is compared against hydrocarbon standards using to identify and calculate the TPH concentrations.

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Lab Name: *Analytical Control Facility* Method Code: *034*

Percent Food Content in Digestive Tract

Digestive tract samples are laid on a bench for thawing. After thawing, the digestive tract is carefully observed and a visual estimate is made as to what volume may be food.

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Lab Name: *Analytical Control Facility* Method Code: *035*

Temephos (Abate) in Tissue, Plants, Water and Sediment.

Summary: A sample aliquot is extracted, dried with sodium sulfate, concentrated , cleaned up on Florisil with methylene chloride, and concentrated again for GC/FPD analysis as follows:. Water (about 800-1000 mls) is extracted with 3x 100 mls methylene chloride, filtered and dried with Na2SO4. For tissue, sediment, and plant material, a 10 g aliquot of tissue or sediment or 5 g of (less dense) homogenized plant material is placed in a 50 ml centrifuge tube and extracted by sonication with 3 x 20 mls of a 1:1 mixture of acetone: methylene chloride, filtered and dried with Na2SO4. Tissue, plant extracts, and "dirty" water and sediment extracts showing dark-colored coextracted material (e.g. from pond water) are cleaned up on Florisil using methylene chloride. GC analysis using flame photometric detection is performed on a megabore (0.53 mm I.D.) column of DB-1 or equivalent programmed from 150 C to 235 C.

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Lab Name: *Analytical Control Facility* Method Code: *036*

Analytical Methodology for ICP-MS Scan Tissue

Samples were analyzed using a microwave digestion procedure with ICP-MS determination. Tissues were lyophilized using a Labconco Freeze Dry System Model 7730. A one half to one gram sample of the freeze dried tissue was digested in 10 ml ultrapure nitric acid and 4 ml of 30% hydrogen peroxide using an IO Model 7301 Microwave Digestion System. The final volume was brought to 25 ml with reverse osmosis deionized water. The analyses were performed using a Perkin Elmer Elan 6000 Inductively Coupled Plasma Mass Spectrometer with conditions recommended by the manufacturer.

Reference

1. Labconco Freeze Dry System Model 7756 Instruction Manual. 1993.

2. OI Analytical Microdigest Model 7301 Instruction Manual. 1992.

3. Perkin Elmer Corporation Manual. 1998. Elan 6000 Inductively Coupled Plasma Mass Spectrometer Software Guide and Update Kit.

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Analytical Services, Ltd. (AXYS) Laboratory Methods

Select one of the links below to display the method descriptions associated with AXYS or <HERE> to go to the Start Page.

Method Code	Method Title
001	Dissection of Tissue Samples
002	Homogenization of Tissue Samples
003	Homogenization of Vegetation
004	Decantation of Water from Sediments
005	Homogenization of Soil and Sediment
006	Homogenization by Stirring
007	*Subsampling* of Aqueous Samples
008	Homogenization of Blood Samples
011	*Soxhlet* Extraction of Tissue, Sediment and Soil Samples
012	Solvent Extraction of Water Samples
013	Solvent Extraction of Blood Samples
014	*Soxhlet* Extraction of XAD Columns
015	*Soxhlet* Extraction of Particulate Filters
021	Gel Permeation Column Chromatography
022	Chromatographic Cleanup of PCBs, Pesticides and *Toxaphene* on *Florisil*
023	Chromatographic Cleanup of Non-*ortho*-substituted PCBs on Carbon/*Celite* and Alumina
024	Chromatographic Cleanup of PCB Congeners on *Florisil*
025	Chromatographic Separation and Cleanup of Dioxins, Furans, Non-*ortho*-substituted PCBs, Ortho-substituted PCBs, Pesticides and *Toxaphene* on Carbon/*Celite*
026	Chromatographic Cleanup of Dioxins, Furans and Non-*ortho*-substituted PCBs on Alumina

027 Chromatographic Cleanup on Layered Silica

028 Chromatographic Cleanup on PCB Congeners on *Florisil*, Alumina and Layered Silica

029 Chromatographic Cleanup of *PAHs* on Layered Silica

030 Chromatographic Cleanup of PCDD/F on *Florisil* and Alumina Columns

031 HRGC/MS Analysis of PCB Congeners

032 HRGC/MS Analysis of Non-*ortho*-substituted PCB Congeners

033 HRGC/MS Analysis of Chlorinated Pesticides

034 LRGC/MS Analysis of Chlorinated Pesticides, PCBs (*Aroclors*), *Toxaphene*, and PCB Congeners

035 GC/MS/ECNI Analysis of *Toxaphene*

036 GC/ECD Analysis of Polar Chlorinated Pesticides

037 HRGC/MS Analysis of Dioxins, Furans and Non-*ortho*-substituted PCBs

038 HRGC/MS/ECNI Analysis of *Toxaphene*

039 HRGC/MS analysis of PCB Congeners (EPA Method 1668)

040 LRGC/MS Analysis of *PAHs*

041 HRGC/HRMS Analysis of Polybrominated Diphenylethers (PBDE)

051 Determination of Percent Moisture

052 Determination of Percent Lipid

053 Determination of Grain Size

054 Determination of Total Organic Carbon

055 Determination of Percent Lipid

Lab Name: *Analytical Services, Ltd.* Method Code: 001

Dissection of Tissue Samples

The animal is dissected frozen. Muscle tissue is carefully removed from the bones and skin of the animal with a clean, solvent-rinsed scalpel. Organs are excised from the animal using a solvent rinsed scalpel. Dissected tissue and/or organ is stored frozen.

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Lab Name: *Analytical Services, Ltd.* Method Code: 002

Homogenization of Tissue Samples

Tissue samples are thawed then homogenized using clean, solvent rinsed homogenization apparatus suitable to the size of the sample. Small quantities of sample, including organs and bivalves, are homogenized with a *Virtis* blender. Muscle tissue is homogenized with an *Oster* blender. Whole samples are ground by passing the sample three times through a commercial meat grinder. Homogenized samples are stored frozen.

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Lab Name: *Analytical Services, Ltd.* Method Code: 003

Homogenization of Vegetation

Vegetation samples are homogenized frozen. Grass, leaves, and stalks are cut into small pieces with solvent rinsed scissors and homogenized with a clean *Waring* Blender. Large quantities of vegetation are homogenized using a solvent rinsed meat grinder. Homogenized samples are stored frozen.

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Lab Name: *Analytical Services, Ltd.* Method Code: 004

Decantation of Water from Sediments

Samples are thawed. Free standing water is carefully decanted without disturbing the sediment. If fine particles are decanted with the free water, they are separated by centrifugation and added back to the sample. Decanted water is discarded.

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Lab Name: *Analytical Services, Ltd.* Method Code: 005

Homogenization of Soil and Sediment

Samples Soil and sediment samples are thawed. The sample is transferred to a clean solvent rinsed stainless steel bowl and manually broken up. Rocks larger than 0.5 cm are removed and discarded. If dry, the sample is passed through a 0.4 cm sieve to remove rocks. The sample is stirred with a clean spatula until homogeneous. The homogenized sample is stored frozen.

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Lab Name: *Analytical Services, Ltd.* Method Code: 006

Homogenization by Stirring

Samples which are received as homogenates are homogenized by stirring with a clean, solvent rinsed spatula just prior to *subsampling*.

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Lab Name: *Analytical Services, Ltd.* Method Code: 007

***Subsampling* of Aqueous Samples**

Aqueous samples which need to be *subsampled* for analysis are homogenized by shaking vigorously and immediately poured into a graduated cylinder.

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Lab Name: *Analytical Services, Ltd.* Method Code: 008

Homogenization of Blood Samples

Blood samples are thawed and mixed thoroughly by shaking prior to *subsampling*.

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Lab Name: *Analytical Services, Ltd.* Method Code: 011

Soxhlet Extraction of Tissue, Sediment and Soil Samples

Animal and plant tissue, soil and sediment samples are extracted by grinding the sample with anhydrous sodium *sulphate*, spiking with surrogate standards, and refluxing in a *soxhlet* apparatus for 16 to 20 hours. The cooled extract is concentrated by rotary evaporation.

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Lab Name: *Analytical Services, Ltd.* Method Code: 012

Solvent Extraction of Water Samples

Aqueous samples are spiked with surrogate standards and extracted by shaking three times with dichloromethane. The extract is dried over anhydrous sodium *sulphate* and concentrated in a *Kuderna*-Danish concentrator.

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Lab Name: *Analytical Services, Ltd.* Method Code: 013

Solvent Extraction of Blood Samples

Blood samples, to which surrogate standards have been added are extracted by shaking with an ethanol/ammonium *sulphate*/hexane mixture, followed by shaking with hexane. The combined hexane extracts are washed with water and dried over anhydrous sodium *sulphate*. The extract is filtered and the solvent removed by rotary evaporation.

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Lab Name: *Analytical Services, Ltd.* Method Code: 014

Soxhlet Extraction of XAD Columns

XAD resin, from XAD water sampling columns, is dried by filtration through a Millipore filtration apparatus. The resin is placed in a *soxhlet* thimble and spiked with

surrogate standards. The sample is *soxhlet* extracted with dichloromethane. The extract is concentrated by rotary evaporation.

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Lab Name: *Analytical Services, Ltd.* Method Code: 015

Soxhlet Extraction of Particulate Filters

Particulate filters from XAD water sampling columns are air-dried. The filters are placed in a *soxhlet* thimble and spiked with surrogate standards. The sample is *soxhlet* extracted with dichloromethane. The extract is concentrated by rotary *evporation*.

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Lab Name: *Analytical Services, Ltd.* Method Code: 021

Gel Permeation Column Chromatography

Concentrated extracts are loaded onto a calibrated gel permeation column (*Biobeads* SX-3) to remove high molecular weight interferences. The column is eluted with 1:1 dichloromethane and the second fraction collected. This fraction is concentrated by rotary evaporation, prior to additional chromatographic cleanup procedures.

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Lab Name: *Analytical Services, Ltd.* Method Code: 022

Chromatographic Cleanup of PCBs, Pesticides and *Toxaphene* on *Florisil*

The extract is loaded onto a *Florisil* column (2.1% deactivated) which is eluted with hexane followed by 15:85 *dichloromethane*:hexane. The *eluates* are collected together (F1). This fraction contains chlorinated pesticides, *toxaphene*, and PCB congeners. The fraction is concentrated, an aliquot of recovery standard added and the extract transferred to an *autosampler* vial in preparation for instrumental analysis. However, for the analysis of non-*ortho*-substituted congeners, the fraction is first split, and one half is subject to additional cleanup on carbon/*Celite* to isolate the non-*ortho*-substituted PCBs. The *Florisil* column is then eluted with 1:1 *dichloromethane*:hexane and the *eluate* collected (F2). This fraction contains polar chlorinated pesticides. The fraction is concentrated, an aliquot of recovery standard

added and the extract transferred to an *autosampler* vial in preparation for instrumental analysis.

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Lab Name: *Analytical Services, Ltd.* Method Code: 023

Chromatographic Cleanup of Non-*ortho*-substituted PCBs on Carbon/*Celite* and Alumina

The first fraction from the *Florisil* cleanup procedure is eluted through a 4.75% carbon on *Celite* chromatography column. The column is eluted with 1:1 *cyclohexane*:dichloromethane (discard) followed by *ethylacetate* (discard). The non-*ortho*-substituted PCB congeners are eluted with 1:1 *toluene*:ethylacetate. The concentrated extract is loaded onto an alumina column (1% deactivated) which is eluted with hexane (discard) followed by 1:1 *dichloromethane*:hexane (retain). The *eluate* is concentrated and an aliquot of recovery standard added in preparation for instrumental analysis.

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Lab Name: *Analytical Services, Ltd.* Method Code: 024

Chromatographic Cleanup of PCB Congeners on *Florisil*

The extract is loaded onto a *Florisil* column (2.1% deactivated) which is eluted with hexane followed by 15:85 *dichloromethane*:hexane. The *eluates* are collected together (F1). This fraction contains PCB congeners. The fraction is concentrated, an aliquot of recovery standard added and the extract transferred to an *autosampler* vial in preparation for instrumental analysis.

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Lab Name: *Analytical Services, Ltd.* Method Code: 025

Chromatographic Separation and Cleanup of Dioxins, Furans, Non-*ortho*-substituted PCBs, Ortho-substituted PCBs, Pesticides and *Toxaphene* on Carbon/*Celite*

The extract is loaded onto a pre-eluted 4.5% carbon on *Celite* chromatography column which is eluted with 1:1 *cyclohexane*:dichloromethane followed by 10:1 *ethylacetate:toluene*. The *eluates* are collected together and concentrated (Fraction

E1). This fraction contains *ortho*-substituted PCBs, pesticides and *toxaphene* and requires additional cleanup on *Florisil*. The column is inverted, eluted with toluene and the *eluate* collected (Fraction E2). This fraction contains dioxins, furans and non-*ortho*-substituted PCBs and requires further cleanup on alumina.

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Lab Name: *Analytical Services, Ltd.* Method Code: 026

Chromatographic Cleanup of Dioxins, Furans and Non-*ortho*-substituted PCBs on Alumina

The concentrated extract is loaded onto an alumina column (1% deactivated) which is eluted with hexane. This fraction is added to the F1/F2 fraction from a *Florisil* column, if pesticides are part of the analysis. Otherwise, this fraction is discarded. The column is then eluted with 1:1 *dichloromethane*:hexane (retain). The *eluate* is concentrated and an aliquot of recovery standard added in preparation for instrumental analysis.

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Lab Name: *Analytical Services, Ltd.* Method Code: 027

Chromatographic Cleanup on Layered Silica

The concentrated extract is loaded onto a layered silica column (layers: neutral, basis, neutral, acidic, acidic). The column is eluted with hexane. The *eluate* is collected and concentrated in preparation for additional chromatographic cleanup procedures.

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Lab Name: *Analytical Services, Ltd.* Method Code: 028

Chromatographic Cleanup on PCB Congeners on *Florisil*, Alumina and Layered Silica

The extract is loaded onto a *Florisil* column (2.1% deactivated) which is eluted with hexane followed by 15:85 *dichloromethane*:hexane. The *eluates* are collected together (F1). The concentrated fraction is loaded onto an alumina column (1% deactivated) which is eluted with hexane (discard) followed by 1:1 *dichloromethane*:hexane (retain). The concentrated fraction is loaded onto a layered

silica column (layers: neutral, basic, neutral, acidic, acidic). The column is eluted with hexane. The *eluate* is concentrated and an aliquot of recovery standard added in preparation for instrumental analysis.

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Lab Name: *Analytical Services, Ltd.* Method Code: 029

Chromatographic Cleanup of *PAHs* on Layered Silica

The concentrated extract is loaded onto a layered *silican* column (layers: neutral, basic, neutral, acidic, acidic). The column is eluted with pentane, and the *eluate* discarded. The column is eluted with dichloromethane. The *eluate* is collected and concentrated in preparation for additional chromatographic cleanup procedures.

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Lab Name: *Analytical Services, Ltd.* Method Code: 030

Chromatographic Cleanup of PCDD/F on *Florisil* and Alumina Columns

The extract is loaded onto a *Florisil* column (2.1% deactivated) which is eluted with hexane followed by 15:85 *dichloromethane*:hexane. The *eluates* are collected together (F1). The concentrated fraction is loaded onto an alumina column (1% deactivated) which is eluted with hexane (discard) followed by 1:1 *dichloromethane*:hexane (retain). The column is eluted with hexane. The *eluate* is concentrated and an aliquot of recovery standard added in preparation for instrumental analysis.

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Lab Name: *Analytical Services, Ltd.* Method Code: 031

HRGC/MS Analysis of PCB Congeners

High resolution analysis of PCB congeners is carried out using a either a VG 70SE or VG *Ultima* mass spectrometer. Each instrument is equipped with a Hewlett Packard 5890 GC, a CTC *autosampler* and a VAX 3100 data system. Data are acquired in the Selected Ion Monitoring mode to enhance sensitivity. The MS is operated at mass resolution 10000. Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 mm *i.d*., 0.1 *æm* film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response

factors of PCB congeners are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 032

HRGC/MS Analysis of Non-*ortho*-substituted PCB Congeners

High resolution analysis of non-*ortho*-substituted PCBs is carried out using a VG 70SE mass spectrometer equipped with a Hewlett Packard 5890 GC, a CTC *autosampler* and a VAX 3100 data system. Data are acquired in the Selected Ion Monitoring mode to enhance sensitivity. The MS is operated at mass resolution 10000. Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 mm *i.d*., 0.25 *æm* film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors of PCBs are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 033

HRGC/MS Analysis of Chlorinated Pesticides

High resolution analysis of pesticides is carried out using a VG 70SE mass spectrometer equipped with a Hewlett Packard 5890 GC, a CTC *autosampler* and a VAX 3100 data system. Data are acquired in the Selected Ion Monitoring mode to enhance sensitivity. The MS is operated at mass resolution 10000. Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 mm *i.d*., 0.25 *æm* film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors of pesticides are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 034

LRGC/MS Analysis of Chlorinated Pesticides, PCBs (*Aroclors*), *Toxaphene*, and PCB Congeners

Analysis of pesticides, PCBs (*Aroclors*), Total PCBs, *toxaphene* and individual PCB congeners is carried out using a *Finnigan* INCOS 50 mass spectrometer equipped with a Varian 3400 GC, a CTC A200S *autosampler* and a DG10 data system running *Incos* 50 (Rev 11) software. The MS is operated at unit mass resolution in the Multiple Ion detection mode. Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 mm *i.d*., 0.10 *æm* film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 035

GC/MS/ECNI Analysis of *Toxaphene*

Toxaphene is analyzed using a *Finnigan* INCOS 50 mass spectrometer equipped with a Varian 3400 GC, a CTC A200S *autosampler* and a DG10 data system running *Incos* 50 (Rev 11) software. The MS is operated in the electron capture negative ionization mode. Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 mm *i.d*., 0.10 *æm* film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 036

GC/ECD Analysis of Polar Chlorinated Pesticides

The most polar chlorinated pesticides are analyzed using a Hewlett Packard 5890A gas chromatograph equipped with a 63Ni electron capture detector. Chromatographic separation is achieved using a DB-5 capillary column (60 m, 0.25 mm *i.d*., 0.10 *æm* film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors of pesticides are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 037

HRGC/MS Analysis of Dioxins, Furans and Non-*ortho*-substituted PCBs

High resolution analysis of polychlorinated dioxins and furans and non-*ortho*-substituted PCBs is carried out using a VG *Ultima* mass spectrometer equipped with a Hewlett Packard 5890 GC, a CTC *autosampler* and a VAX 3100 data system. Data are acquired in the Selected Ion Monitoring mode to enhance sensitivity. The MS is operated at mass resolution 10000. Chromatographic separation is achieved with a DB-5 capillary column (60 m, 0.25 mm *i.d*., 0.1 *æm* film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors of authentic dioxins, furans and non-*ortho*-substituted PCBs are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 038

HRGC/MS/ECNI Analysis of *Toxaphene*

Toxaphene is analyzed using a VG *Autospec* mass spectrometer equipped with a Hewlett Packard 6890 GC, a A200S CTC *autosampler* and an Alpha data station. Data are acquired in the Selected Ion Monitoring mode to enhance sensitivity. The MS is operated at mass resolution 10000 and in the electron capture negative ionization mode. Chromatographic separation is achieved with a BC-5 capillary column (60 m, 0.25 mm *i.d*., 0.10 (m film thickness). A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 039

HRGC/MS analysis of PCB Congeners (EPA Method 1668)

High resolution analysis of PCB congeners is carried out using a VG 70SE mass spectrometer equipped with a Hewlett Packard 5890 GC, a CTC *autosampler* and an Alpha workstation. Data are acquired in the Selected Ion Monitoring mode to enhance sensitivity. The MS is operated at mass resolution 10000. Chromatographic separation is achieved with a SPB-*Octyl* capillary column (30 m, 0.25 mm *i.d*., 0.25 um film thickness). A *splitless*/split injection sequence is used. Initial calibration is established using a series of calibration solutions encompassing the working concentration range. Calibration is verified every 12 hours by analysis of a mid-range calibration solution. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 040

LRGC/MS Analysis of *PAHs*

Analysis of *PAHs* is carried out using a *Finnigan* INCOS 50 mass spectrometer equipped with a Varian 3400 GC, a CTC A200S *autosampler* and a Pentium workstation running manufacturer software. The MS is operated at unit mass resolution in the Multiple Ion detection mode. Chromatographic separation is achieved on a *Restek* Rtx-5 chromatography column (30 m, 0.25 mm *i.d*., 0.25m film thickness), coupled directly to the MS source. A *splitless*/split injection sequence is used. A calibration solution is run every 12 hours and response factors are determined. *Analytes* are quantified using the internal standard method of quantification, comparing the area of *analyte* peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 041

HRGC/HRMS Analysis of Polybrominated Diphenylethers (PBDE)

Polybrominated diphenylethers are analyzed by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) using a Micromass Ultima or VG70 mass spectrometer (MS) equipped with a Hewlett Packard 5890 or 6890 gas chromatograph, running Micromass software. A DB-5HT capillary chromatography column (30 m, 0.25 mm i.d. x 0.1 um film thickness) is coupled to the MS source. The mass spectrometer is tuned to have a static mass resolution of 5,000. Data are acquired in the Selected Ion Monitoring mode to enhance sensitivity. Initial calibration is established using a series of calibration solutions encompassing the working concentration range. Calibration is verified

every 12 hours by analysis of a mid-range calibration solution. Analytes are quantified using the internal standard method of quantification, comparing the area of analyte peak to that of the corresponding surrogate standard and correcting for response factors.

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Lab Name: *Analytical Services, Ltd.* Method Code: 051

Determination of Percent Moisture

A weighed *subsample* is dried for at least 16 hours at 105øC and then reweighed. Percent moisture is calculated as the percent difference between the dry weight and the wet weight.

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Lab Name: *Analytical Services, Ltd.* Method Code: 052

Determination of Percent Lipid

Percent lipid determination is carried out on extracts by drying two weighed *subsamples* of extract at 105øC for 30 minutes. The extracts are re-weighed. The percent lipid is calculated as the percent lipid of wet sample weight.

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Lab Name: *Analytical Services, Ltd.* Method Code: 053

Determination of Grain Size

To determine particle size distribution, samples are pre-treated to remove organic matter, carbonates, soluble salts, and iron oxides. The percent gravel, sand, silt and clay is determined by a combination of dry sieving, wet sieving and *pipetting* techniques.

Reference: "Methods of Soil Analysis, Part 1 - Physical and Mineralogical Methods" (Gee and *Bauder*, 1986) Method 15-4.

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Lab Name: *Analytical Services, Ltd.* Method Code: 054

Determination of Total Organic Carbon

Total organic carbon is determined by high temperature oxidation of carbon to carbon dioxide which is then measured by means of a *nondispersive* infrared analyzer. Total organic carbon is calculated as the percent difference between total and inorganic carbon.

Reference: USEPA Method 9060A

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Lab Name: *Analytical Services, Ltd.* Method Code: 055

Determination of Percent Lipid

The concentration of triglycerides, *cholestrol* and phospholipids are determined *enzymatically* using *Technicon* RA-500 *Analyser*.

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Brooks - Rand (BRND) Labartory Methods

Select one of the links below to display the method descriptions associated with BRND or <HERE> to go to the Start Page.

Method Code	Method Title
001	Total Mercury in Hair and Certified Standard Tissues
002	Methylmercury in Hair and Certified Tissues

Lab Name: Brooks - Rand (BRND) Labartory Method Code 001

Total Mercury in Hair and Certified Standard Tissues

For total mercury in hair and certified standard tissues, approximately 30 mg of hair was weighed to the nearest milligram into 18.2 mL teflon vials. 5 mL of 7:3 HNO_3 + H_2SO_4 was added, and the samples digested in sealed containers for 3 h at 70 C. Upon cooling, samples were diluted to the 18.2 mL mark with a 5% solution of 0.2N CrCl in 12 N HC1. For total mercury in blood, 100 uL of homogenized blood was placed into a 7 mL conical teflon vial. 0.9 mL of the acid mixture was added, and the sample digested as above. The final volume was taken to be 1.0 mL. Total mercury was analysed on 25-100 uL aliquots of the digestate, by $SnCl_2$ reduction, purging onto gold coated sand, and quantification by Cold Vapour Atomic Fluorescence Spectrometry (CVAFS). The instrumental Detection limit is about 1 pg Hg.

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Lab Name: Brooks - Rand (BRND) Labartory Method Code 002

Methylmercury in Hair and Certified Tissues

For methylmercury in hair and certified tissues, approximately 30 mg was weighed to the nearest mg into 18.2 mL teflon vials. 10 mL of 25% KOH/methanol was added, and the samples digested in sealed containers for 3 h at 70 C. Upon cooling, the samples were diluted to the 18.2 mL mark with methanol. For blood samples, 50 uL of blood was placed into a 7 mL conical teflon vial, 950 uL of KOH/methanol added., and the samples digested as above. The methylmercury was analysed on 25-100 uL aliquots by aqueous phase ethylation, cryogenic GC separation, and CVAFS detection.

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Environmental Trace Substance Laboratory (ETSL) Labratory Methods

Select one of the links below to display the method descriptions associated with ETSL or <HERE> to go to the Start Page.

Method Code	Method Title
001	PRECONCENTRATION OF ICP - PH6
002	PRECONCENTRATION OF ICP - PH3
003	NITRIC - PERCHOLORIC DIGESTION - (I.C.P.)
004	INDUCTIVELY COUPLED PLASMA (ICP)
005	% MOISTURE
006	HOMOGENIZATION
007	NITRIC - PERCHOLORIC DIGESTION - (SELENIUM)
008	NITRIC - PERCHOLORIC DIGESTION - (ARSENIC)
009	ARSENIC AND SELENIUM BY HYDRIDE
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030 Total Cyanide Detection from Water Samples

Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 001

PRECONCENTRATION OF ICP - PH6

A 30 g. sample of the digestate for I.C.P. was weighed into a 50 ml. screw top centrifuge tube. One ml. of 2000 ppm Indium and 1 ml. of 10% ammonium acetate buffer were added and the pH adjusted to 6.5 with high purity NH4OH from Seastar. One ml. of a 10% DDTC was added and the caps screwed on and mixed by turning end over end 6 times slowly. After mixing, the tubes were centrifuged in an I.E.C. refrigerated centrifuge at 20 C for 15 minutes at 15,000 RPM. The liquid was then decanted from the precipitate and 0.3 ml. of high purity HNO3 from Seastar was added. The tubes were heated in a water bath at 95oC to dissolve the precipitate and diluted to 3 ml. with deionized water.

For samples high in Calcium and Phosphate a pH of 6.0 was used to reduce the precipitation of Ca3(PO4)2.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 002

PRECONCENTRATION OF ICP - PH3

A 30 g. sample of the digestate for I.C.P. was weighed into a 50 ml. screw top centrifuge tube. One ml. of 2000 ppm Indium and 1 ml. of 10% ammonium acetate buffer were added and the pH adjusted to 3.0 with high purity NH4OH from Seastar. One ml. of 10% APCD was added and the caps screwed on and mixed by turning end over end 6 time slowly. After mixing, the tubes were centrifuged in an I.E.C. refrigerated centrifuge at 20oC for 15 minutes at 15,000 RPM. The liquid was then decanted from the precipitate and 0.3 ml. of high purity HNO3 from Seastar was added. The tubes were heated in a water bath at 95 C to dissolve the precipitate and diluted to 3 ml. with deionized water.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 003

NITRIC - PERCHOLORIC DIGESTION - (I.C.P.)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. quartz Kjeldahl flask. (Samples containing a high percent of silica and sediment samples were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample was measured into a teflon beaker. Slowly 15 ml. of concentrated sub- boiled HNO3 and 2.5 ml. of concentrated sub- boiled HC104 were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes has ceased. Gradually, the heat was increased until the HNO3 began refluxing; samples were allowed to reflux overnight. (This decreased the chance for charring during the reaction with HC104.) After the refluxing, the heat was gradually increased until the HNO3 had been driven off, and the reaction with HC104 had occurred. When dense white fumes from the HC104 were evident, the samples were removed from the heat and allowed to cool. Two ml. of concentrated sub-boiled HCl was added. The flasks were replaced on the heat and warmed until the containers were hot to the touch or started to boil. They were removed from the heat, and 5-10 ml. of deionized water was added. Samples were allowed to cool. They were then diluted using deionized water in a 50 ml. volumetric flask and transferred to a clean, labeled, 2 oz. polyethylene bottle.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 004

INDUCTIVELY COUPLED PLASMA (ICP)

The instrument used for ICP analysis was a Jarrell-Ash Model 1100 Mark III with 40 analytical channels, controlled by a Digital Equipment Company (DEC) 11/23+ computer with two RLO2 disk drives, DEC VT100 terminal, and DEC LA120 Decwriter III. The instrument was standardized with a series of seven standards containing 36 elements. After the standardization, the detection limit was determined by taking ten integrations of the zero standard; three times the standard deviation of the mean was used as the detection limit. Instrumental quality control samples were then analyzed to check the ICP operation. If the values were acceptable, the samples were then analyzed. Standards were run every 10-15 samples to check for drift. If the drift was more than 5%, the instrument was restandardized. After the analysis was completed, the data was transferred to the Perkin-Elmer LIMS 2000 computer for calculation. The final detection limit for each element was further increased by 4% of the magnitude of the spectral interferences from the other elements. The data was checked before calculation to correct for possible errors in sample number, weight, volumes and dilution. The data was calculated using the ICP calculation program written by ETSRC computer staff, which corrected for blanks, standard drift, spectral interferences, sample weight, sample volume, and dilution. After quality control was reviewed, a final report was generated using a Hewlett Packard laser jet printer.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 005

% MOISTURE

For animal tissue and sediments of sufficient size, moisture was determined by placing a weighed aliquot of the sample in a Fisher Isotemp oven and drying at 103 - 105 C. The dried sample was then weighed and the data entered into a computer program to generate the % moisture and final report.

Plants, and samples to small for oven dried moisture determination had the % moisture calculated from the moisture lost during the freeze-drying in the Labcono Freeze-Dryer 8. The data was entered into a computer program to generate a % moisture and final report.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 006

HOMOGENIZATION

Large tissue samples, such as whole fish, were first run through a meat grinder one or more times depending on the size of the sample. An aliquot of the ground sample was weighed and frozen. For smaller tissue samples and plant samples the entire sample was weighed and then frozen. For sediments, the sample was mixed and an aliquot weighed and frozen. The frozen samples were placed in a Labcono Freeze Dryer 8 until the moisture had been removed. The dry samples were then weighed and further homogenized using a blender, or Spex Industries, Inc. Model 8000 mixer/mill with tungsten-carbide vial and balls.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 007

NITRIC - PERCHOLORIC DIGESTION - (SELENIUM)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. quartz Kjeldahl flask. (Samples containing a high percent of silica and sediment samples were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample was measured into a teflon beaker. Slowly 15 ml. of concentrated sub- boiled HNO3 and 2.5 ml. of concentrated sub- boiled HC104 were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the HNO3 began refluxing; samples were allowed to reflux overnight. (This decreased the chance for charring during the reaction with HC104.) After the refluxing, the heat was gradually increased until the HNO3 had been driven off, and the reaction with HC104 had occurred. When dense white fumes from the HC104 were evident, the samples were removed from the heat and allowed to cool. Two ml. of concentrated sub-boiled HCl was added. The flasks were replaced on the heat and warmed until the containers were hot to the touch or started to boil. They were removed from the heat, and 5-10 ml. of deionized water was added. Samples were allowed to cool. They were then diluted using deionized water in a 50 ml. volumetric flask and transferred to a clean, labeled, 2 oz. polyethylene bottle.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 008

NITRIC - PERCHOLORIC DIGESTION - (ARSENIC)

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. Kjeldahl flask. (Samples containing a high percent of silica and sediment samples were digested in 100 ml. teflon beakers.) For water samples, 50 ml. of sample was measured into a teflon beaker. Slowly 15 ml. of concentrated sub-boiled HNO3 and 2.5 ml. of concentrated sub-boiled HC104 were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the HNO3 had been driven off, and the reaction with HC104 were evident. The samples were removed from the heat and allowed to cool. Samples were diluted using deionized water in 50 ml. volumetric flasks and transferred to clean, labeled, 2 oz. polyethylene bottles.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 009

ARSENIC AND SELENIUM BY HYDRIDE

The Varian VGA-76 hydride generation accessory was mounted on either a Perkin-Elmer Model 603 AA or Model 3030 (B) AA. Electrodele ss Discharge lamps (EDL) were used. The instrument and EDL settings were taken from the instrument manuals. The burner mount for a Perkin-Elmer Model 10 Hydride generator was modified slightly to hold the Varian quartz cell. The cell was aligned in the light path of the burner chamber and a very lean flame was used for heating the cell. The two stoc solutions were 50% v/v sub-boiled HCl and 0.6% NaBH4 in 0.5% NaOH for Selenium and concentrated sub-boiled HCl and 1% NaBH4 in 0.5% NaOH for Arsenic. Samples were diluted in 10% v/v sub-boiled HCl. Standards were prepared by dilution of Fisher 1000 ppm stock in 10% v/v sub-boiled HCl in the range of 0 to 20 PPB. The instrument was standardized to read directly in PPB using S1 = 5.00 and S2 = 20.00. After standardization, the standardization was checked by reading other standards such as 2.00,10.00 and 15.00 PPB and an instrumental quality control sample with a known value. If the standards and quality control were acceptable, the detection limit was determined by reading the zero standard 10 times and twice the standard deviation of the mean was used as the detection limit. Samples were analyzed by taking an integrated reading for 3 seconds after the plateau was reached for the sample. This occurred approximately 45 seconds after the sample tube was placed in the sample. Standardization was checked every 8-15 samples and approximately 10% of the samples were checked by the method of additions to monitor matrix effects. Matrix effects were usually not significant with the VGA-76. The data was corrected for drift of the standard curve and entered into the AA calculation program. This program corrected for blank, dilution, sample weight, sample volume and recorded the data in the LIMS database for report generation.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 010

ARSENIC IN FISH AND MARINE SAMPLES BY HYDRIDE

The Perkin-Elmer MHS-1 hydride generation accessory was mounted on either a Perkin-Elmer Model 603 AA or Model 3030 (B) AA. An Electrodeless Discharge Lamp (EDL) was used. The instrument and EDL settings were taken from the instrument manuals. The cell was aligned in the light path of the burner chamber and a very lean flame used for heating the cell. The standard curve was run and a quality control sample of known concentration to check the standard curve. Blanks and samples are run by diluting an aliquot of the digested sample to 10 ml. with 4% v/v HC104. The amount of sample used varied with the Arsenic concentration. Samples were analyzed using the Method of Standard Additions. The peaks from the recorder tracing were measured with a ruler and the slope and intercept calculated on a calculator. The data was entered into the AA calculation program. This program

corrected for the blank, dilution factors, sample weight, sample volume and recorded the data in the LIMS database for report generation.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 011

NITRIC REFLUX DIGESTION FOR MERCURY

Approximately 0.5 g. of sample was weighed into a freshly cleaned 50 ml. round bottom flask with 24/40 ground glass neck. For waters, 10 ml. of sample was measured into the flask. Five ml. of concentrated sub-boiled HNO3 was added and the flask was placed under a 12 inch water cooled condenser with water running through the condenser. The heat was turned up to allow the HNO3 to reflux no more than 1/3 the height of the columns. Samples were allowed to reflux for two hours. Then the heat was turned off and the sample allowed to cool. The condensers were rinsed with 1% v/v HCl and the flasks removed. The samples were diluted with 1% v/v HCl in a 50 ml. volumetric flask and then transferred to a clean, labeled, 2 oz. flint glass bottle.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 012

MERCURY - COLD VAPOR ATOMIC ABSORPTION

Equipment used for Cold Vapor Atomic Absorption include: Perkin-Elmer Model 403 AA; Perkin- Elmer Model 056 Recorder; Technicon Sampler I; Technicon Pump II; a glass cell with quartz windows and capillary tube for entry and exit of the mercury vapor; and a liquid-gas separator. The samples were placed in 4 ml. sample cups at least 3/4 full. The samples were mixed with hydroxylamine for preliminary reduction, then stannous chloride for reduction to the mercury vapor. The vapor was separated from the liquid and passed through the cell mounted in the light path of the burner compartment. The peaks were recorded and the peak heights measured. The standardization was done with at least 5 standards in the range of 0 to 10 ppb. The correlation coefficient was usually 0.9999 or better and must have been at least 0.999 to have been acceptable. A standard was run every 8-10 samples to check for drift in the standardization. This was usually less than 5%. Standards were preserved with 10% v/v HNO3, 1% v/v HCl and 0.05% w/v K2Cr207. The solution concentrations were calculated and the data entered into the AA calculation program which corrected for blank, dilution, sample weight, sample volume and entered the data into the LIMS system for report generation.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 013

NITRIC DIGESTION FOR GRAPHITE FURNACE

Approximately 0.5 g. of sample was weighed into a freshly cleaned 100 ml. Kjeldahl flask (samples containing a high percent of silica and sediment samples were digested in 100 ml. teflon beakers). For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly, 15 ml. of sub-broiled HN03 were added. Foaming may occur with some samples. If the foaming started to become excessive, the flask was cooled in a beaker of cold water. The samples were placed on low heat until the initial reaction and evolution of dark red fumes had ceased. Gradually, the heat was increased until the volume was reduced to approximately 2 ml. The sample was removed from the heat and allowed to cool. Samples were diluted using deionized water in a 50 ml. volumetric flask and transferred to a clean, labeled, 2 oz. polyethylene bottle.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 014

GRAPHITE FURNACE AA

The instruments used for graphite furnace AA, were either a Perkin-Elmer Model 3030B with Model HGA-500 graphite furnace, Model AS-40 autosampler and Model 056 recorder, or the Perkin-Elmer Model 5100 with Model HGA-600 graphite furnace, Model AS- 60 autosampler and Model 7300 computer. The conditions for a particular element were set up according to the instruction manual. The L'vov platform and appropriate matrix modifier were used. A standard curve and known quality control sample were run to check the instrument operation. The Method of Standard Additions was used on a minimum of 1 out of 5 samples. If the average slopes for the standard additions gave a %RSD of 5% or less then the average slope was used to calculate the sample concentrations. If the average slope was not acceptable then the sample all had to be run using the Method of Standard Additions. After calculating the solution concentration, the data were entered into a computer program that corrected for blank, dilution, sample weight and volume, and entered the data into the LIMS data base for report generation.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 015

TOTAL VOLATILE SOLIDS (TVS)

Aliquots of oven-dried samples were weighed into previously fired crucibles. The crucibles with samples were then placed in an oven and fired at 550øC for a minimum of 4 hours. The crucibles were allowed to partially cool and then were placed in a desiccator until they cooled to room temperature and were weighed. The oven-dried weights and the weights of the samples after firing were entered into the computer and TVS was calculated. The formula used to calculate TVS follows.

$$\frac{\text{Oven-dry Sample Weight - Fired Sample Weight}}{\text{Oven-dry Sample Weight}} = \text{TVS}$$

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 016

ANTIMONY BY HYDRIDE

The Varian VGA-76 hydride generation accessory was mounted on either a Perkin-Elmer Model 603 AA or Model 3030 (B) AA. Electrodeless Discharge Lamps (EDL) were used. The instrument and EDL settings were taken from the instrument manuals. The burner mount for a Perkin-Elmer Model 10 Hydride generator was modified slightly to hold the Varian quartz cell. The cell was aligned in the light path of the burner chamber and a very lean flame was used for heating the cell. The two stock solutions were concentrated sub-boiled HC1 and 0.6% NaBH4 in 0.5% NaOH. Samples were diluted with 50% v/v sub-boiled HC1, 1% w/v KI. Standards were prepared by dilution of Fisher 1000 ppm stock with 50% v/v sub-boiled HC1, 1% w/v KI, in the range of 0 to 20 PPB. The instrument was standardized to read directly in PPB using S1 = 5.00 and S2 = 20. After standardization, the standardization was checked by reading other standards such as 2.00, 10.00, and 15.00 PPB and an instrumental quality control sample with a known value. If the standards and quality control were acceptable, the detection limit was determined by reading the zero standard 10 times, and twice the standard deviation of the mean was used as the detection limit. Samples were analyzed by taking an integrated reading for 3 seconds after the plateau was reached for the sample. This occurred approximately 45 seconds after the sample tube was placed in the sample. Standardization was checked every 8-15 samples and approximately 10% of the samples were checked by the method of additions to monitor matrix effects. Matrix effects were usually not significant with the VGA-76. The data was corrected for drift of the standard curve and entered into the AA calculation program. This program corrected for blank,

dilution, sample volume and recorded the data in the LIMS database for report generation.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 017

NITRIC - PERCHOLORIC DIGESTION - (ANTIMONY)

Approximately 0.5 g. of sample wa weighed into a freshly cleaned 100 ml. quartz Kjeldahl flask. (Sediment samples and samples containing a high percent of silica were digested in 100 ml. teflon breakers). For water samples, 50 ml. of sample were measured into a teflon beaker. Slowly, 15 ml. of concentrated sub-boiled HNO3 and 2.5 ml. of concentrated sub-boiled HC104 were added. Foaming may occur with some samples. If the foaming started to become excessive, the container was cooled in a beaker of cold water. After the initial reaction had subsided, the sample was placed on low heat until the evolution of dark red fumes had ceased. Gradually, the heat was increased until the HNO3 began refluxing, samples were allowed to reflux overnight. (This decreased the chance of charring during the reaction with HC104). After the refluxing, the heat was gradually increased until the HNO3 had been driven off, and the reaction with HC104 had occurred. When dense white fumes from the HC104 were evident, the samples were removed from the heat and allowed to cool. Fifteen ml. of concentrated sub-boiled HC1 were added. The flasks were replaced on the heat and warmed until the containers were hot to the touch or started to boil. They were removed from the heat, and 5-10 ml. of deionized water were added. Samples were allowed to cool. They were then diluted using deionized water in a 50 ml. volumetric flask and transferred to clean, labeled, 2 oz. polyethylene bottles.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 018

TOTAL ORGANIC CARBON ANALYSIS (SOIL AND SEDIMENT) DRY OXIDATION METHOD

The analyst will need to make three (3) replicates of each blank and each sample.

Label all of the ampules with a high temperature grease pencil before weighing. Break all ampules open, trying to get as little glass inside the ampules as possible. When not using, cover the ampules with aluminum foil to keep additional contaminants out of the ampules.

SAMPLE PREPARATION

Place ampule on pan of 5 place balance, tare to zero, add the desired mass of sample (usually 3-5mg), record the mass of the sample, put the ampule in the holding rack. Cover the ampule with aluminum foil. Start the same procedure with the next ampule.

Add 100 ul of 10% HCl solution to all of the sample and blank ampules. The fizzing that may be observed is the reaction between the HCl and any inorganic carbon in the sample.

Put the uncovered ampules into an oven and bake at 100 C for at least one (1) hour or however long it takes for sample to reach dryness.

Prepare a set of KHP stds. ranging from 25 ug to 250 ug C following the KHP std. prep method. These will be used to determine a std. curve for TOC values.

KHP STD. PREP.

2.126 grams of Potassium Hydrogen Phthalate is weighed then transferred to a 100 ml volumetric flask. The dilution to 100 mls is done with boiled D.I. H20. (The D.I. H20 is boiled to d rive off as much carbon as possible).

This reagent (which equals 10,000 ug/ml C) is further diluted to 500, 400, 300, 250, 200, 100 and 50 ug/ml C levels. One half ml (500 ul) pipet is then used to make the 250 thru 25 ug C stds.

Spikes are also made from the same reagent.

At least 4 ampules should be made of each std., and more depending on the total amount of analyses.

The stds. are dried at 100 C but usually take as long as 4 hrs. to dry because of their 1/2 ml liquid volume.

After all visible liquid has evaporated from samples and stds. remove the ampules from the drying oven and allow to cool.

When the ampules are cool, add one (1) dipper (200 mg) of the combusted cupric oxide to each ampule.

Purge each ampule with oxygen from the Purge & Seal Unit for 4 to 6 minutes and then seal.

The ampules can then be carefully placed in the muffle furnace and baked at 550 C for 4-5 hours.

Cool and then analyze using a non-dispersive infrared analyzer. (Oceanographic International Corp.)

ANALYSIS

Set up standard curve using the KHP stds. Three or four ampules are broken of each std. to get a good representative curve.

After the curve is established, begin analyzing the samples and determine their ugC according to this KHP curve.

Detection limit for a 4 mg (0.004 gm) sample is 0.1% or 1000 mcg/g dw.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 019

GRAIN SIZE DETERMINATION

Sieve an air dried, crushed sample. Place approximately 10 g of sample in a 500 ml Fleaker. Those samples with greater than 1.0% organic carbon (wet weight) need to be treated to remove organic matter. Add 10 mls deionized water, 10 mls 30% H_2O_2, and 1 drop acetic acid. Stir, cover and heat to approximately 60 C. Then add additional H_2O_2 in 5 ml increments at 15 minute intervals until organic matter is oxidized (no bubbling). If the reaction is too vigorous, slow with the addition of Dl water. After organic matter is removed, oven dry the samples (still in Fleakers) at 105 C. Weigh samples.

Add 10 mls sodium hexametaphosphate to each sample, and bring volume to approximately 150 mls with Dl water. A blank consisting of 10 mls sodium hexametaphosphate and 150 mls Dl water should also be made at this time. Put stoppers in the Fleakers and shake either for 4 hours of overnight (minimum 12 hours) on a horizontal reciprocating shaker (120 oscillations/minute).

Sieve samples through a 50 um sieve to remove sand fraction. Transfer the sand fraction that remains in the sieve to a weighing pan, oven-dry, cool in a desiccator and weigh. Arrange the fleakers for pipetting and bring volume to 400 mls with Dl water. Determine average temperature of the suspensions, and cover each Fleaker with a watch glass.

Remove watch glass from the first Fleaker, cap with a rubber stopper, and shake vigorously for approximately 10 seconds. Then shake end-over-end for 15 seconds. Remove rubber stopper and replace watch glass. Shake samples at approximately 2 minute intervals.

Pipet an aliquot at 5.0 cm below the surface within a 15- to 20-second interval, discharge into a weighing pan, rinse the pipet and add the rinse to the pan. Oven dry the aliquots, cool in a desiccator, then weigh. The correction factor for the weight of the sodium hexametaphosphate must be subtracted to obtain the weight of the clay.

Percentages of sand, silt and clay are then determined according to the following equations:

% sand = 100 (total sand weight/total soil weight)
% clay = (clay weight) (400/pipette volume) (100/total soil weight)
% silt = 100 x (% sand + % clay)

Table 1. Setting times at corresponding temperature for pipetting <2-um fraction at a 5-cm depth.

Temperature Setting Times

C	h: mm
17	4:22
18	4:15
19	4:09
20	4:03
21	3:57
22	3:51
23	3:46
24	3:41
25	3:36
26	3:31
27	3:26
28	3:22
29	3:17
30	3:13

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 020

Microwave Digestion

This procedure is used for samples of limited size. Preferably 0.5 g of dry sample is weighed directly into a clean tared teflon bomb. Then 5 mls of concentrated sub-boiled nitric acid (HNO3) and 3 mls of hydrogen peroxide (H202, 30% ACS Reagent) are added.

The bomb is capped and tightened at the capping station to 12 ft. lbs./in2. The bomb is then set overnight. After
12 or more hours, the accumulated gases are vented. The bombs are then placed in the teflon rack. Tubing is inserted into each bomb with the other end of the tubing going into the waste container in the center of the rack. The rack with bombs is then placed on the turntable in the microwave oven, and the door is closed. The fan and turntable are turned on. The pressure limit is set to 100. Then the microwave is programmed and started.

When samples are finished heating, they are allowed to cool to room temperature. The samples are diluted to 50 ml using deionized water or 1% HCl, depending on what elements were to be analyzed from this digestion. If samples were small, the volume of acid and hydrogen peroxide were adjusted, and the final volume was diluted to 25 ml.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 021

Magnesium Nitrate Prep.

Approximately 0.5 g of sample was weighed into an acid cleaned 100 ml glass beaker. The samples were wet with 3 mls of methanol. 5 drops of anti-foam agent, 10 mls of 40% (w/v) magnesium nitrate hexahydrate and 10 mls of nitric acid were then added.

Beakers were covered with watch glasses and refluxed on low (70-80) heat overnight (8-12 hrs.). The temperature was then increased to 200 C and the watch glasses slid to one side of beaker to speed evaporation and cook sample to complete dryness. This may take 8-12 hrs. Watch glasses were then repositioned and the samples allowed to cool.

Samples were then transferred to a cold muffle furnace. The temperature was started at 250 and ramped to 500 in 50 increments. Samples were left at each

temperature for 15 minutes. Once temperature reached 500, the samples remained at that temperature for 3 hours.

Samples were cooled to room temperature. The 20 mls of 50% HCl were added. The watch glasses were replaced and samples were gently boiled on a hot plate for 1 hour. Samples are not allowed to go to dryness. If needed, more than 50% HCl was added to continue the boiling for 1 hour.

The sample was again cooled. The volume was readjusted to 20 mls with 50% HCl, then diluted to 100 mls with deionized water.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 022

HCl04-HF Digestion for Sediments

Approximately 1 gram of sample is weighed into a clean 100 ml teflon beaker and is then wet with 5 ml of distilled water.

Two ml of HCl04 (70%) and 12 ml of HF (40%) are added, and the sample is heated to near dryness.

An additional 8 ml of HF are then added and the sample heated to dryness.

Two ml of HCl04 and several ml of distilled water are again added to the sample and evaporated to dryness.

The remaining residue is dissolved in 8 ml of 1:1 HCl and 20 ml of water. The sample is then diluted to a final volume of 100 ml and stored in a 4 oz. polyethylene bottle.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 023

Microwave Digestion Which Is Aliquoted and Has HCl Added To The Aliquot For Mercury Analysis.

This procedure is used for samples of limited size. Preferably 0.5 g of dry sample is weighed directly into a clean tared teflon bomb. Then 5 mls of concentrated sub-

boiled nitric acid (HNO3) and 3 mls of hydrogen peroxide (H202, 30% ACS Reagent) are added.

The bomb is capped and tightened at the capping station to 12 ft. lbs./in2. The bomb is then set overnight. After 12 or more hours, the accumulated gases are vented. The bombs are then placed in the teflon rack. Tubing is inserted into each bomb with the other end of the tubing going into the waste container in the center of the rack. The rack with bombs is then placed on the turntable in the microwave oven, and the door is closed. The fan and turntable are turned on. The pressure limit is set to 100. Then the microwave is programmed and started.

When samples are finished heating, they are allowed to cool to room temperature. The samples are diluted to 50 ml using deionized water. If samples were small, the volume of acid and hydrogen peroxide were adjusted, and the final volume was diluted to 25 ml.

Certain elements are adversely effected by HCl in the dilution. However, HCl is required when mercury is an analyte. In these cases, the microwave digestion is diluted with deionized water and no HCl. Then an aliquot is taken immediately, placed in a separate bottle and has concentrated HCl added. The size of the aliquot and the amount of HCl will vary depending on sample size and number of analytes. Sample weights and volumes are corrected for the
aliquoting and addition of HCl.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 024

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

The instrument used for ICP-MS analysis was a Perkin-Elmer model ELAN 5000. The instrument was standardized with t
wo multi-element standards. After the standardization, the detection limit was determined by taking ten measurements of the zero standard. Three times the standard deviation of the mean was used as the detection limit. An instrument quality control sample was then analyzed to check the ICP-MS operation. If the value was acceptable the samples were then analyzed. Standards were run every 10-20 samples to check for drift. If the drift was more than 10%, the instrument was restandardized. After the analysis was completed, the data was transferred to the Concurrent LIMS system for calculation. The data was checked before calculation to correct for possible errors in sample number, weight, volume and dilution. The data was calculated using the ICP-MS calculation program written by ETSRC computer staff, which corrected for blanks, instrument drift, sample weight, sample volume and dilution (1). After quality control was reviewed, a final report was generated.

(1) A spectral correction on Cr was performed for samples prepared in hydrochloric or perchloric acid. Due to the fact that V can not be measured in chlorine matrices with quadrupole based ICP-MS, the V spectral interference on Cr was corrected with the V value from ICP- OES. The reported Cr value was the Cr value from ICP-MS minus 5.6% of the V value from ICP-OES.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 025

GRAPHITE FURNACE AND ICP SOIL DIGESTION

SCOPE: Metals from sand, soil, or sediment.

PRINCIPLE: Dissolve sample in liquid so that it may be injected into analyses instrument.

EQUIPMENT: Face shield, teflon beakers, hot plate, 10 ml and 50 ml poly propylene centrifuge tubes.

REAGENTS: Concentrated nitric acid (HN03) (Baker instra-analyzed or better) concentrate hydrochloric acid (HCL) (Baker instra-analyzed or better) scandium 10.000 ppm

PROCEDURE:

1. Weigh 1.0 g of subsample into a teflon beaker
2. Add 20 ml of 3:1 hydrochloric:nitric
3. Place on hot plate and heat for 4 hours at a setting 1-2
4. After 4 hours, raise setting to 4-5 until approximately 11 mls of acid has evaporated (if the sample goes dry or starts to scorch it will need to be redone)
5. The sample is then filtered and transferred into a 50 ml tube. The total volume of sample is increased to 20 mls using deionized water.
6. Add 100 microliters of scandium if samples are to be analyzed by ICP.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 026

Residue, Filterable (Gravimetric, Dried at 180-C)

INSTRUMENTATION: Drying Oven

SCOPE AND APPLICATION -- This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination is 10 mg/L to 20,000 mg/L.

SUMMARY OF METHOD -- A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180-C. If Residue, Non-Filterable is being determined, the filtrate from that method may be used for Residue, Filterable.

DEFINITIONS -- Filterable residue is defined as those solids capable of passing through a glass fiber filter a
nd dried to constant weight at 180-C.

SAMPLE HANDLING AND PRESERVATION -- Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4-C, to minimize micro-biological decomposition of solids, is recommended.

INTERFERENCES -- Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing. Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180-C to insure that all the bicarbonate is converted to carbonate. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

APPARATUS -- Glass fiber filter discs, 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E, or equivalent. Filter holder, membrane filter funnel or Gooch crucible adapter. Suction flask, 500 mL. Gooch crucibles, 25 mL (if 2.1 cm filter is used). Evaporating dishes, porcelain, 100 mL volume. (Vycor or platinum dishes may be substituted). Steam bath. Drying oven, 180- C +/- 2-C. Desiccator. Analytical balance, capable of weighing to 0.1 mg.

PROCEDURE -- Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is app lied, wash the disc with three successive 20 mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings. Preparation of evaporating dishes: If Volatile Residue is also to be measured heat the clean dish to 550 +/- 50-C for one hour in a muffle furnace. If only Filterable Residue is to be measured heat the clean dish to 180 +/- 2-C for one hour. Cool in desiccator and store until needed. Weigh immediately before use. Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 mL to the funnel by means of a 100 mL graduated cylinder. If total filterable residue is low, a larger volume may be filtered. Filter the sample through the glass fiber filter, rinse with three 10 mL portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible. Transfer 100 mL (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath. Dry the evaporated sample for at least one hour at 180 plus or minus 2-

C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

CALCULATE FILTERABLE RESIDUE AS FOLLOWS:

$$\text{Filterable residue, mg/ml} = \frac{(A - B) * 1{,}000}{C}$$

where:

A = weight of dried residue + dish in mg
B = weight of dish in mg
C = volume of sample used in mL

Precision and Accuracy are not available at this time.

BIBLIOGRAPHY

Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 92, Method 208B, (1975).

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 027

Residue, Non-Filterable (Gravimetric, Dried at 103 - 105-C)

INSTRUMENTATION: Drying Oven

SCOPE AND APPLICATION -- This method is applicable to drinking, surface, and saline water, domestic and industrial wastes. The practical range of the determination is 4 mg/L to 20,000 mg/L.

SUMMARY OF METHOD -- A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103 - 105-C. The filtrate from this method may be used for Residue, Filterable.

DEFINITIONS -- Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103 - 105-C.

SAMPLE HANDLING AND PRESERVATION -- Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4-C, to minimize microbiological decomposition of solids, is recommended.

INTERFERENCES -- Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results. Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.

APPARATUS -- Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeves Angel 934-AH, Gelman type A/E, or equivalent. NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above. Filter support: filtering apparatus with reservoir and a coarse (40-6- microns) fritted disc as a filter support.

NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

Suction flask. Drying oven, 103 - 105-C. Desiccator. Analytical balance, capable of weighing to 0.1 mg.

PROCEDURE -- Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103 - 105-C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only. Selection of Sample Volume for a 4.7 cm diameter filter, filter 100 mL of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. If other filter diameters are used, start with a sample volume equal to 7 mL/cm2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.

NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiver filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty- five mL increments for timing are suggested. Continue to record the time and volume increments until filtration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration rate occurred.

Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support. Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through. With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through. NOTE: Total volume of wash water used should equal approximately 2 mL per cm2 for a 4.7 cm filter the total volume is 30 mL. Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103 - 105 C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

CALCULATE NON-FILTERABLE RESIDUE AS FOLLOWS:

$$\text{Non-filterable residue, mg/ml} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = mL of sample filtered

Precision and Accuracy are not available at this time. Accuracy data on actual samples cannot be obtained.

BIBLIOGRAPHY

NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 028

This method code is used when liquid samples, such as waters, are analyzed as they were received at our laboratory. Samples did not have any further preparation prior to being analyzed for specific analytes.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 029

Determination of Acid Volatile Sulfide in Sediments

Determination of AVS (acid volatile sulfide) in a sediment is conducted by treatment of the sample for one hour with dilute HC1 in an oxygen- free atmosphere. The resultant hydrogen sulfide which forms is purged by nitrogen into a sodium hydroxide trapping solution. Free sulfide ion is rapidly formed in the trap which is then measured by a sulfide- specific electrode.

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Lab Name: Environmental Trace Substance Laboratory (ETSL) Labratory
Method Code: 030

Total Cyanide Detection from Water Samples

This method comprises of two parts: distillation and colorimetry. The QC standard, with certified value of 59.2 mg/L total cyanide, was purchased from Environmental Resource Associates, 5540 Marshall St. Arvada, Colorado 80002. Cat. #502, Lot # 99110.

A. Distillation:

1. Add 250 ml of water sample to the 500-mL round bottom flask.
2. Add 10 ml of 1N NaOH solution to the gas scrubber and dilute with DI water to about 150 ml.
3. Connect the cyanide distillation apparatus. Turn on the cold water for the condenser. Adjust gas flow rate.
4. Add 2 g sulfamic acid through the air inlet tube and wash down with DI water.
5. Add 50 ml 1:1 H2SO4 through the air inlet tube. Let air mix flask contents for 3 min.
6. Add 20 ml MgCl2 reagent and wash down with DI water.
7. Heat the sample to boil and reflux for one hour.
8. Discontinue heating but continue air flow for 15 min. Cool and quantitatively transfer absorption solution to a 250-ml volumetric flask. Dilute to volume with DI water.
9. Determine cyanide concentration in absorption solution by colorimetric method.

B. Colorimetry:

1. Standard curve: Pipet a series of standards containing 0, 1, 2, 4, 6, 8, 10 mg CN- into 50-ml volumetric flask
s. Dilute to 40 ml with NaOH dilution solution.
2. Sample: Pipet a portion of absorption solution from step A into a 50-ml volumetric

flask and dilute to 40 ml with NaOH dilution solution.
3. Add 1 ml acetate buffer and 2 ml chloramines-T solution to each flask, mix well and let stand for 2 min.
4. Add 5 ml pyridine-barbituric acid reagent to each flask. Dilute to volume with DI water. Mix well and let stand for 8 min.
5. Measure absorbance against DI water at 578 nm.
6. Make standard curve and measure the cyanide concentration of the samples.

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Geochemical & Environmental Research Group (GERG) Laboratory Methods

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003	% Dry Weight
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005	Aromatic Hydrocarbon Metabolites in Bile
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009	Total Organic Carbon-Leco
010	Total Organic Carbon-Coulometrics
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012	Total Petroleum Hydrocarbons
013	Acid Volume Sulfide (AVS)
014	Trace Metal-Tissue
015	Trace Metal (except Hg) - Water
016	Determination of Benzene, Toluene, Ethylbenzene and M,P,O - Xylene By Gas Chromatography/Mass Spectrometry (Btex)
017	Water Organic
018	TBT
019	Percent Total Volatile Solids
020	Trace Method Total Sediment BOMB Digest
021	Trace Metal Total Tissue BOMB Digest
022	Total Cyanide (Sediment, Tissues or Water)

023 Sediment Trace Metals by INAA
024 Tissue Trace Metals by INAA
025 Tissue Organics
026 Dioxin/Furan Analysis
027 Analysis of Tetra Ethyl Lead
028 Total Petroleum Hydrocarbons
029 Organophosphate Pesticides - Tissues
030 Organophosphate Pesticides - Sediments
031 EPA Method for Semivolatiles
035 Simultaneously Extracted Metals (SEM) -- Sediment
036 Methyl Mercury in Tissue
037 Chlorophylls and Other Plant Pigments
038 Biomass Determination
039 Hydrazine Analysis in Sediments
040 Determination of Pesticides and PCBs by GC/MS
041 Determination of PBDE's by GC/MS
042 Determination of Triazines by GC/MS

Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 001

Tissue Organics

The tissue samples were extracted by the NOAA Status and Trends Method (MacLeod et al., 1985) with minor revisions (Brooks et al., 1989; Wade et al., 1988). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and methylene chloride in a centrifuge tube. The tissue extracts were purified by silica/alumina column chromatography to isolate the aliphatic and PAH/pesticide/PCB fractions. The PAH/pesticide/PCB fraction was further purified by HPLC in order to remove interfering lipids.

The quantitative analyses were performed by capillary gas chromatography (CGC) with a flame ionization detector for aliphatic hydrocarbons, CGC with electron capture detector for pesticides and PCB's, and a mass spectrometer detector in the SIM mode for aromatic hydrocarbons (Wade et al., 1988).

There are specific cases where analytes requested for the pesticide and PCB analyses and are known to co-elute with other analytes in the normal CGC with electron capture. These include the pesticide Endosulfan I and the PCB congeners 114 and 157. In these cases, the samples will be analyzed by CGC with a mass spectrometer detector in the SIM mode.

References

1. Brooks, J.M., T.L. Wade, E.L. Atlas, M.C. Kennicutt II, B.J. Presley, R.R. Fay, E.N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

2. MacLeod, W.D., D.W. Brown, A.J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R.G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Compounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS, NOAA Tech. Memo. NMFS F/NWRC-92.

3. Wade, T.L., E.L. Atlas, J.M. Brooks, M.C. Kennicutt II, R.G. Fox, J. Sericano, B. Garcia, and D. DeFreitas (1988) NOAA Gulf of Mexico Status and Trends Program: Trace Organic Contaminant Distribution in Sediments and Oyster. Estuaries 11, 171-179.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 002

Tissue, Sediment and Water Mercury

Mercury was determined by EPA method 245.5 with minor revisions. Sediment samples can be analyzed either freeze dried or on a wet basis. Sediment samples are homogenized by mixing before subsampling. The tissue samples were homogenized in the original sample containers with a Tekar Tissumizer and subsampled. Water samples are acidified (0.5% v/v with high purity nitric acid, HNO3) in the original sample bottle. For sediments a 0.5 to 1.0 gram sample (dry weight) was used. For tissues a 1.5 to 2.0 gram sample (wet weight) was used. For water the sample size is 20 ml.

For tissue and sediment, the sample is weighed into a 50 ml polypropylene centrifuge tube. 2.5 ml of concentrated sulfuric acid (H2SO4) and 1.5 ml of concentrated nitric acid (HNO3) were added and the samples heated in a water bath at 90 C for 15 min. After cooling 10 ml of distilled water and 15 ml of mixture of 3.3% (w/w) potassium permanganate (KMnO4), and 1.7% (w/w) potassium persulfate (K2S2O8) were added to each tube and the samples heated in a water bath at 90 C for 30 min. After cooling 5 ml of 10% (w/w) hydroxylamine hydrochloride (NH2OH HC1) was added to reduce excess permanganate and the volume brought to 35 ml with distilled water.

For water samples, the sample is weighed into a 50 ml polypropylene centrifuge tube, 1 ml of concentrated H2SO4 is added and the solution mixed vigorously with a vortex stirrer. Then 4.5 ml of the KMnOr/K2S2O8 is added and the resulting mixture heated in a 90 C water bath for 2 hours. After cooling, 1.5 ml of a 10% (w/w) hydroxylamine hydrochloride (NH2OH HC1) solution is added, sample volume adjusted to a constant volume with distilled water and the resulting solution mixed vigorously.

Mercury is determined by a modification of the method of Hatch and Ott (1968). A portion of the digest solution is placed in a sealed container. To this is added 0.4 ml of 10% (w/w) stannous chloride (SnCl2). Mercury is reduced to the elemental state and aerated from solution into an atomic absorption spectrophotometer where its concentration is measured.

References

1. "USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis." Document Number ILM01.0 USEPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

2. "Interim Method for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," USEPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, Aug. 1977, Revised Oct. 1980.

3. Hatch, W.R. and Ott, W.L., "Determination of Sub-Microgram Quantities of Mercury in Solution by a Flameles Atomic Absorption Technique", Analytical Chemistry 40,14 p 2085-2087 (1968).

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 003

% Dry Weight

Approximately 1 gram of wet sample is weighed into a clean, labeled, preweighed 10 ml beaker. The beaker is placed in a forced air oven at approximately 75 degrees Celsius for 24 hours. The beaker with the dry sample is then weighed and the % dry weight is calculated by the formula:

$$\frac{(\text{wt. dry sample and beaker}) - (\text{wt. beaker})\ (100)}{(\text{wt. wet sample and beaker}) - (\text{wt. beaker})}$$

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 004

Sediments Organic/Pesticide

The sediment samples were freeze-dried and extracted in a Soxhlet extraction apparatus. Briefly, the freeze-dried sediment samples were homogenized and a 10-gram sample was weighed into the extraction thimble. Surrogate standards and methylene chloride were added and the samples extracted for 12 hrs. The extracts were treated with copper to remove sulfur and were purified by silica/alumina column chromatography (MacLeod et al., 1985; Brooks et al., 1989) to isolate the aliphatic and aromatic/pesticide/PCB fractions.

The quantitative analyses were performed by capillary gas chromatography (CGC) with a flame ionization detector for aliphatic hydrocarbons, CGC with electron capture detector for pesticides and PCB's, and a mass spectrometer detector in the SIM mode for aromatic hydrocarbons (Wade et al., 1988).

There are specific cases where analytes requested for the pesticide and PCB analyses and are known to co-elute with other analytes in the normal CGC with electron capture. These include the pesticide Endosulfan I and the PCB congeners

114 and 157. In these cases, the samples will be analyzed by CGC with a mass spectrometer detector in the SIM mode.

References:

1. Brooks, J.M., T.L. Wade, E.L. Atlas, M.C. Kennicutt II, B.J. Presley, R.R. Fay, E.N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 005

Aromatic Hydrocarbon Metabolites in Bile

SAMPLE PRESERVATION AND STORAGE

Upon arrival at the laboratory sample vials are inspected, logged in, and unique laboratory identification numbers are assigned. Chain-of- custody protocols are followed.

INSTRUMENT ANALYSIS

Five ul of the sample is injected onto the IIPLC system using an autosampler. Instrument settings are:

Column Oven Temperature 50 plus/minus 1 C
HPLC Pump Run Time 35 minutes
Gradient Elution Linear, methanol/water

SAMPLE ANALYSIS

Once optimal HPLC operating parameters have been established and the instrument is functioning according to specifications (see above), 5 ul of each sample/standard are injected directly onto the HPLC system using an autosampler. The response of the fluorescence detector is recorded with a HP-1000 computer for 35 minutes at naphthalene, phenanthrene and benzo[a]pyrene excitation/emission wavelength paris. Peak areas are integrated for those peaks eluting between 5 and 28 minutes for naphthalene and between 5 and 28 minutes. These times are approximate and are based on times reported in the NOAA Technical Memorandum, NMFS F/NWC-102 and are verified for each system. Chromatographic conditions are selected so that no aromatic hydrocarbon metabolites elute before 5 minutes and solvent contaminants elute after 28 minutes for naphthalene, phenanthrene and benzo[a]pyrene metabolites.

CALCULATIONS

Phenanthrene and naphthalene peaks are identified from the calibration standards. The retention times are recorded and the areas of the reference standards are integrated. The mean response factor (ng/integration unit) is used to calculate sample analyte concentrations. The approximate retention times for naphthalene, phenanthrene, and benzo[a]pyrene are approximately 15, 17, and 21 minutes, respectively under the given analytical conditions.

STANDARDS

Standard concentrations are approximately: naphthalene 4 ng/ul, phenanthrene 2 ng/ul, and benzo[a]pyrene 0.3 ng/ul. Standards are prepared in methanol under yellow lights and stored at 20 C in amber vials.

CALIBRATION

Three calibration standards are analyzed at the beginning of each sample run and must agree within an RSD of plus/minus 10% before proceeding. During the sample run standards will comprise at least 10% of the run and must agree within an RSD of 10% of the mean calibration value.

REFERENCE BILE

A reference bile (provided by Dr. Margaret Krahn) is analyzed and reported for each sample run and must have an RSD of no more than plus/minus 15% of the previous value and not exceed an RSD of plus/minus 25% for each batch.

REFERENCES

1. Krahn, M.M., M.S. Meyers, D.G. Burrows, and D.C. Malins. 1984. Determination of metabolites in zenobiotics in bile of fish from polluted water ways. Zenobiotica 14:633-646.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 006

Grain Size

A small aliquot of sediment is treated with 30% hydrogen peroxide to remove organic coating from grains. A dispersing agent is then added to the sample. The sand/mud fractions are then separated using a 63 micron sieve. The sand fraction (>63 microns) is retained on the screen and the mud fraction (silt and clay <63 microns) is washed into a 1 (one) liter volumetric cylinder. The sand fraction is dried, sieved on a 63 micron screen and weighed. The sediment which passes through the screen a second time is added to the 1 liter cylinder. The mud fraction is analyzed by stirring the cylinder and sampling 20 ml aliquots at 4 and 8 phi intervals. The 4 and 8 phi samples are dried and weighed. The % sand, silt, and clay fractions are determined on a dry weight basis.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 007

Trace Metal-Sediment

Sediments are digested with aqua regia (3:1 HCl:HNO3) in glass beakers on a hotplate and diluted to volume with distilled water. Metals in the digestate are determined by 3 techniques, depending upon concentration and element. Mercury is determined by cold vapor atomic absorption spectrometry (AAS), in which Sn2+ is used to reduce Hg0. Arsenic, selenium, cadmium, and lead are determined by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud. Remaining elements (and Cd or Pb when in high concentration) are determined by atomic emission using an argon plasma.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 008

Trace Metal SEM-Sediment

Sediments are digested with 1N HC1 in glass beakers on a hotplate and diluted to volume with distilled water. Metals in the digestate are determined by 3 techniques, depending upon concentration and element. Mercury is determined by cold vapor atomic absorption spectrometry (AAS), in which Sn2+ is used to reduce Hg0. Arsenic, selenium, cadmium, and lead are determined by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud. Remaining elements (and Cd or Pb when in high concentration) are determined by atomic emission using an argon plasma.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 009

Total Organic Carbon-Leco

TOC contents are analyzed by two methods. The first method (Leco method) burns acidified freeze dried sediment using a LECO Model 523-300 induction furnace under an oxygen environment. The resultant carbon dioxide gas is detected and quantified with a Horiba PIR-2000 infrared detector. The output signal from the Horiba is sent to an HP 3396A integrator which reports the quantity of carbon dioxide as a peak area. If it is necessary to calculate inorganic carbon, an unacidified sample is also analyzed and the difference between the acidified sample (Total Organic Carbon) and the nonacidified sample (Total Carbon) is the inorganic carbon.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 010

Total Organic Carbon-Coulometrics

The second method (Coulometrics) also burns sediment under an oxygen atmosphere to produce carbon dioxide gas. This gas is bubbled through an electrochemical cell, where a reaction between the CO2 and the cell solution

produces a color change. This solution is then electrically backtitrated to the endpoint. The amount of electricity (coulombs) used to reach this endpoint is directly proportional to the amount of organic carbon in the sample. The concentration of inorganic carbon is determined using the same detector, but the CO2 is produced by acidifying the sample in a closed environment.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 011

Oil and Grease

Follow Method number 001 or Method number 004 for the extraction of sediments or tissues. Before the extract is concentrated for silica gel/alumina column, mark the level of extract on the flask then remove 20 ml and rotovap to dryness. Bring the volume of the dry aliquot to 1 ml with methylene chloride, and weigh a 100 microliter aliquot. Measure the volume of the original extract. The concentration of Oil and Grease is calculated by the formula:

$$\frac{\text{(wt. 100 ul aliquot) (total volume)}}{\text{(sample wt.) (20)}}$$

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 012

Total Petroleum Hydrocarbons

Follow Method number 001 or Method number 004 for the extraction of sediment or tissues. After the extract has passed through an alumina column remove a known volume and rotovap to dryness. Bring the volume of the dried aliquot to 1 ml with methylene chloride and weigh a 100 microliter aliquot. The concentration of Total Petroleum Hydrocarbons is calculated by the formula:

$$\frac{\text{(wt. / 100 ul aliquot) (total volume)}}{\text{(sample wt.) (\% removed for aliquot)}}$$

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 013

Acid Volume Sulfide (AVS)

Acid volatile sulfide (AVS) is defined as sulfides that are converted to H2S upon exposure to 1N HCl at room temperature for 1 hour. Approximately 10 grams of wet sediment are acidified with 1N HCl, and reactive sulfides that are converted to gaseous H2S are trapped as Ag2S and measured gravimetrically.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 014

Trace Metal-Tissue

Tissues are either digested with nitric acid or dry ashed in a muffle furnace. Metals are determined by 3 techniques, depending upon concentration and element. Mercury is determined by cold vapor atomic absorption spectrometry (AAS), in which Sn2+ is used to reduce Hg0. Arsenic, selenium, cadmium, and lead are determined by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud. Remaining elements (and Cd or Pb when in high concentration) are determined by atomic emission using an argon plasma.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 015

Trace Metal (except Hg) - Water

The water sample is kept in the bottle in which originally received for the entire procedure to minimize the possibility of contamination. If not acidified prior to receipt, the sample is acidified with high purity nitric acid to achieve a 0.5% (v/v) acid concentration. After acidification the sample is stored at room temperature for at least 24 hours prior to analysis. Metals are determined by 3 techniques to achieve maximum sensitivity for all elements of interest. Mercury is determined by cold vapor atomic absorption spectrophotometry (AAS, see method code 002). Typically B, Ba, Be, Mg, Mo and Sr are determined by argon plasma atomic emission spectroscopy using undiluted acidified sample. Remaining elements are determined, also using

undiluted sample, by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 016

Determination of Benzene, Toluene, Ethylbenzene and M,P,O - Xylene By Gas Chromatography/Mass Spectrometry (Btex)

INTRODUCTION

The method described quantitatively determines Benzene, Toluene, Ethyl-benzene, and M,P,O Xylene (BTEX) in a variety of matrices. This method is applicable to nearly all types of samples including water, sediments, soils, waste solvents, sludges, and industrial wastes. Quantitation is performed by gas chromatography/mass spectrometry (GC/MS) in the full scan mode. The BTEX compounds are introduced into the gas chromatograph by a purge and trap technique and detected using a mass spectrometer, which provides both qualitative and quantitative information. The GC/MS system is operated to obtain separation of the analytes of interest from any interferences.

SAMPLE PREPARATION

Water samples: Water is carefully drawn into a syringe and the volume adjusted to either 5 or 25 ml. Once the volume has been adjusted the surrogate and internal standards are added. A second aliquot is removed at this time to protect the integrity of the sample. The sample is then introduced into the purging device. The second sample is maintained only until such time when the first sample has been properly analyzed. If necessary the second sample must be analyzed within 24 hours.

After the sample and appropriate standards have been transferred to the purging device the purging program is initiated. The samples are purged with purified helium at a flow of 40 ml.min for 11 min at a temperature of 75 C. The purged analytes are trapped on a 0.31 x 25 cm stainless steel column packed with 8 cm each of Tenax-GC, Silican gel, and Charcoal. The trap is held at ambient temperatures (25 C). After purging is completed the analytes are backflushed for two minutes from the trap to the head of the analytical capillary column which has been cooled to -160 C.

After the desorb step is completed the analytes are analyzed by GC/MS.

Units are reported in mg/L for water. Units are reported in ng/g for sediments. Results are reported to three (3) significant figures.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 017

Water Organic

Water samples are extracted after acidified to a pH of 2 or less with HCl. Surrogate standards are added and the water is extracted with methylene chloride in a separatory funnel. The water is extracted three times and the extracts are combine. The organic phase is concentrated to -10-15 ml in a round bottom flask equipped with a three-ball Snyder condenser. The extract is concentrated further and exchanged into 2 ml of hexane in a 25 ml Kuderna-Danish (KD) receiver in a water bath (60 C). Extracts are now ready for purification by column chromatography or analyses directly by GC.

Separation of aliphatic hydrocarbons from aromatic and chlorinated hydrocarbons is accomplished by alumina/silica gel chromatography. Silica gel (20 g, 170 /12 hrs, deactivated 5% with water) is slurry packed in CH2Cl2 over alumina (10 g 400 C/4 hrs, deactivated 1% with water). The extract in 2 ml of hexane is transferred to the column. The column is then eluted with 50 ml of pentane (f1), and 200 ml of 1:1 CH2Cl2:pentane (f2). This recovers the aliphatic (f1) and aromatic hydrocarbon/chlorinated hydrocarbon (f2) fractions.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 018

TBT

The concentration of butyltins including tetrabutyltin, tributyltin, dibutyltin, and monobutyltin were determined as described by Wade et al. (1990) for sediments and Garcia-Romero et al. (1993) for tissues.

Sediments are freeze-dried and a 10-15 gram aliquots of dry sediments are placed into a 50 ml centrifuge tube. Tripropyltin (TPT), is added as surrogate. The sample is extracted with 40 ml of 0.2% tropolone in methylene chloride by vigorously shaking the capped tube with a wrist action shaker. The tube is centrifuged and the supernatant collected. This procedure is repeated two more times. The extract is then concentrated on a rotary evaporator to approx. 20 ml. The samples are transferred into a 50 ml centrifuge tubes and the methylene chloride is replaced with hexane.

Tissue samples are homogenized, an aliquot (2-10g) wet is weighed into a centrifuge tube and surrogate (TPT, approximately 300 ng as Sn) added. The

sample is extracted using a Tissumizer after addition of 100 ml of 0.2% tropolone in methylene chloride and sodium sulfate as a drying agent. The sample and reagents are tissumized for 3 min. The tube is centrifuged and the solvent removed. The extraction is repeated two more times after adding 100 ml of 0.2% tropolone in methylene chloride. The extracts are combined and concentrated on a rotary evaporator to approx. 20 ml. The samples are transferred into a 50 ml centrifuge tubes and the methylene chloride is replaced with hexane.

Sediment or tissue samples are hexylated in the centrifuge tubes by adding 2 ml of hexylmagnesium bromide under a nitrogen atmosphere and heating at 60 C in a water bath for six hours. The excess hexylmagnesium bromide is neutralized by adding 5 ml of 6 M hydrochloric acid. The organic fraction is removed and saved and the aqueous phase is extracted three more times with 10 ml of pentane each time. Sodium sulfate is added to the combined extracts to remove water. The samples are then concentrated to 2 ml, which is transferred to a silica (13.5 g)/alumina (17.0g) column and eluted with 50 ml of pentane. Samples are concentrated to 0.5 ml and tetrapropyltin (4PT), approximately 300 ng as Sn, is added as a recovery standard. Samples are quantitatively analyzed by gas chromatography using a flame photometric detector equipped with a 610 nm filter. The sample concentrations are reported as ng of Sn per gram of dry or wet sediment or tissue.

REFERENCE:

Wade, Terry L., Bernardo Garcia-Romero, and James M. Brooks (1990) Butyltins in Sediments and Bivalves from U.S. Coastal Areas. Chemosphere, Vol. 20, No. 6, pp. 647-662.

Garcia-Romero, B., Terry L. Wade, Gregory G. Salata, and James M. Brooks (1993) Butyltin Concentration in Oysters From The Gulf of Mexico From 1989 to 1991. Environmental Pollution, Vol. 81, pp. 103-

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 019

Percent Total Volatile Solids

Samples are dried in the same manner as for percent moisture (method code 3) except that the procedure is done using high temperature quartz or Vycor crucibles. After the percent moisture has been determined, the dried samples are heated in an electric muffle furnace at 550 degrees centigrade for 4 hours. Samples are cooled in a desiccator and reweighed. The percent total volatile solids (TVS) are then calculated as follows:

Percent TVS = (A-C) * 100/A

A = Weight of dry solids prior to muffle furnace heating
C = Weight of ash (fished matter) after muffle furnace heating

REFERENCE

APHA, AWWA and WPCF. 1976. Standard Methods for the examination of water and wastewater. Method 208 G. pp. 96-98.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 020

Trace Method Total Sediment BOMB Digest

Sediments are digested in heavy-walled, screw-cap Teflon Bombs with concentrated nitric acid, concentrated hydrofluoric acid and 4.5% boric acid added sequentially with separate 6-12 hour oven heating (120 deg. C) after each addition. This procedure results in a total digestion with all trace elements present in the sediment sample being solubilized. Most metals in the digestate are determined by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud. Some elements are typically in high enough concentration (e.g. Mn, Zn) to be determined by flame AAS. Mercury is determined by cold vapor atomic absorption spectrometry (AAS), in which Sn2+ is used to reduce Hg0.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 021

Trace Metal Total Tissue BOMB Digest

Tissues are digested in heavy-walled, screw-cap Teflon Bombs with concentrated high purity nitric acid. Bombs are heated (for 2-8 hours) and opened three times to release CO2 build-up. Oven temperature is 129 deg. C. This procedure results in a total digestion with all trace elements present in the tissue samples being solubilized. Most metals in the digestate are determined by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud. Some elements are typically in high enough concentration (e.g. Zn) to be determined by flame AAS. Mercury is determined by cold vapor atomic absorption spectrometry (AAS), in which Sn2+ is used to reduce Hg0.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 022

Total Cyanide (sediment, Tissues or Water)

Total cyanide was determined according to EPA method for water and waste (Method # 335.2). Sediment samples are h
omogenized by mixing with a metal spoon. Tissue samples are homogenized with a Tekmar Tissumizer. A 1.5 to 2.0 gm sample (wet weight) is used for sediments or tissues and 400 ml for water samples. The samples are refluxed with hot dilute sulfuric acid containing magnesium to convert the free and complexed cyanide to hydrocyanic acid gas (HCN). Sulphamic acid (2 gm) is added to avoid possible interference from nitrate and nitrite. The expelled HCN gas is scrubbed free of accompanying hydrogen sulfide by bubbling through lead acetate solution, then the HCN gas is absorbed into a sodium hydroxide solution. For samples high in cyanide, the sodium hydroxide/cyanide solution is titrated with a standard silver nitrate solution in the presence of Rhodanine a silver sensitive indicator. Alternatively, for samples low in cyanide, the cyanide content in the sodium hydroxide solution is determined colorimetrically by reaction with chloramine T at a pH less than 8. After the reaction is complete, color is formed by the addition of pyridine-barbituric acid reagent and cyanide concentration is determined spectrophotometrically from the absorbance of the solution at 578 nm.

REFERENCES

Bark, L.S. and Higson, H.G. (1964) Investigation of Reagents for the Colorimetric Determination of Small Amounts of Cyanide. Talanta, 2:471-479.

Elly, C.T. (1968) Recovery of Cyanides by Modified Serfass Distillation. Journal Water Pollution Control Federation, 40:848-856.

Annual Book of ASTM Standards, Part 31 (1976) Water. Standard D2036- 75, Method A, p. 503.

Standard Methods for the Examination of Water and Wastewater (1975) 14th Edition, p. 367 and 370, Method 413B and D.

Egekeze, J.O. and Ochne, F.W. (1979) Direct Potentiometric Determination of Cyanide in Biological Materials. J. Analytical Toxicology, Vol. 3, p. 119, May/June.

Casey, J.P., Bright, J.W. and Helms, B.D. Nitrosation Interference in Distillation Tests for Cyanide. Gulf Coast Waste Disposal Authority, Houston, Texas.

EPA, Methods for Chemical Analyses of Water and Wastes (1983). U.S. Environmental Monitoring and Support Lab-Cincinnati, Ohio. pp. 302-310.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 023

Sediment Trace Metals by INAA

This method determines the total concentration of antimony (Sb), arsenic (As), barium (Ba), cerium (Ce), cesium (Cs), cobalt (Co), chromium (Cr), europium (Eu), iron (Fe), hafnium (Hf), manganese (Mn), neodymium (Nd), rubidium (Rb), scandium (Sc), selenium (Se), tantalum (Ta), titanium (Ti), thorium (Th) and vanadium (V) in sediment. Sediment samples are freeze-dried and mechanically powdered with a mortar and pestle prior to loading. Approximately 0.50g of dry powdered sediment is weighed into 2/5 dra m polyethylene polyvials and heat sealed. Samples are arranged in groups of 13 including 2 certified reference materials (CRM as comparator standards), 1 quality control CRM, 8 samples (1 in duplicate) and 1 procedural blank. Other quality control samples are included within the 9 sample slots per can as needed including matrix spikes and blank spikes. Each group of samples is placed in an aluminum can and up to 6 aluminum cans (72 samples and 6 blanks) are irradiated at one time. Samples are irradiated for 14 hours at the TAMU NSC TRIGA reactor using a continuously rotating, long-tube rotisserie device. The nominal thermal neutron fluence for the irradiation is 1xE14 neutron s/cm2. Irradiated samples are allowed to decay for 10 days and then analyzed using gamma-ray spectroscopy. Gamma-ray spectra are acquired for each sample using high resolution pure germanium detectors linked to multi-channel analyzers used in the pulse height analysis mode. Detector efficiencies are typically 20-25% with an observed resolution of 1.8-1.9 KEV full-width half-max at 1332.5 KEV. Analog to digital convertor (instrumental) dead time is kept at 10%. Peak extraction and NAA calculations are accomplished using Nuclear Data proprietary PEAK and NAA programs. Analytical determinations are made by direct comparison with comparator standards (CRM's) having known concentrations of the desired elements.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 024

Tissue Trace Metals by INAA

This method determines the total concentration of antimony (Sb), arsenic (As), barium (Ba), cerium (Ce), cesium (Cs), cobalt (Co), chromium (Cr), europium (Eu), iron (Fe), hafnium (Hf), manganese (Mn), neodymium (Nd), rubidium (Rb), scandium (Sc), selenium (Se), silver (Ag), tantalum (Ta), titanium (Ti), thorium (Th), vanadium (V) and zinc (Zn) in tissue. Tissue samples are freeze-dried and mechanically

powdered with metal-free equipment prior to loading. Approximately 0.30g of dry powdered tissue is weighed into 2/5 dram polyethylene polyvials and heat sealed. Samples are arranged in groups of 13 including 2 certified reference materials (CRM as comparator standards), 1 quality control CRM, 8 samples (1 in duplicate) and 1 procedural blank. Other quality control samples are included within the 9 sample slots per can as needed including matrix spikes and blank spikes. Each group of samples is placed in an aluminum can and up to 6 aluminum cans (72 samples and 6 blanks) are irradiated at one time. Samples are irradiated for 14 hours at the TAMU NSC TRIGA reactor using a continuously rotating, long-tube rotisserie device. The nominal thermal neutron fluence for the irradiation is 1xE14 neutrons/cm2. Irradiated samples are allowed to decay for 10 days and then analyzed using gamma-ray spectroscopy. Gamma-ray spectra are acquired for each sample using high resolution pure germanium detectors linked to multi-channel analyzers used in the pulse height analysis mode. Detector efficiencies are typically 20-25% with an observed resolution of 1.8-1.9 KEV full-width half-max at 1332.5 KEV.

Analog to digital convertor (instrumental) dead time is kept at 10%. Peak extraction and NAA calculations are accomplished using Nuclear Data proprietary PEAK and NAA programs. Analytical determinations are made by direct comparison with comparator standards (CRM's) having known concentrations of the desired elements.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 025

Tissue Organics

The tissue samples were extracted by the NOAA Status and Trends Method (MacLeod et al., 1985) with minor revisions (Brooks et al., 1989; Wade et al., 1988). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and methylene chloride in a centrifuge tube. The tissue extracts were purified by silica/alumina column chromatography to isolate the aliphatic and PAH/pesticide/PCB fractions. The PAH/pesticide/PCB fraction was further purified by HPLC in order to remove interfering lipids.

The quantitative analyses were performed by capillary gas chromatography (CGC) with a flame ionization detector for aliphatic hydrocarbons, CGC with electron capture detector for pesticides and PCB's, and a mass spectrometer detector in the SIM mode for aromatic hydrocarbons (Wade et al., 1988). The pesticides and PCBs are initially analyzed on a DB-5 capillary column. The analyte identity and concentrations are confirmed on a DB-17 capillary column.

REFERENCES

Brooks, J.M., T.L. Wade, E.L. Atlas, M.C. Kennicutt II, B.J. Presley, R.R. Fay, E.N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

MacLeod, W.D., D.W. Brown, A.J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R.G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Com pounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS. NOAA Tech. Memo. NMFS F/NWC-92.

Wade, T.L., E.L. Atlas, J.M. Brooks, M.C. Kennicutt II, R.G. Fox, J. Sericano, B. Garcia, and D. DeFreitas (1988) NOAA Gulf of Mexico Status and Trends Program: Trace Organic Contaminant Distribution in Sediments and Oysters. Estuaries 11:171-179.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 026

Dioxin/Furan Analysis

The procedure uses matrix specific extraction, analyte specific cleanup, and HRGC/HRMS analysis techniques. If interferences are encountered, the method provides selected cleanup procedures to aid in their elimination.

A specified amount of the sample matrix is spiked with a solution containing each of fifteen isotopically (13C12) labeled PCDDs/PCDFs. The sample is then extracted according to a matrix specific extraction procedure. Aqueous samples that are judged to contain 1% or more solids, and solid samples that show an aqueous phase, are filtered, the solid phase and the aqueous phase extracted separately, and the extracts combined before cleanup. Following a solvent exchange step, the extracts are cleaned up by column chromatography on alumina, silica gel, and AX-21 activated carbon on silica. The preparation of the final extract for HRGA/HRMS analysis is accomplished by adding two isotopically (13C12) labeled recovery standards.

Two uL of the concentrated extracts are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10% valley definition). The identification of the sixteen 2378-substituted isomers for which a 13C-labeled standard is available is based on their elution at their exact retention time and the simultaneous detection of the two most abundant ions in the molecular ion region. The identification of OCDF is based on its retention time relative to 13C-OCDD. Confirmation is based on a comparison of the ratios of the integrated ion abundance of the molecular ion species to their theoretical abundance

ratios. Quantitation of the individual congeners is achieved in conjunction with the establishment of a multipoint calibration curve for each homologue, during which each calibration solution is analyzed once.

REFERENCES

Tondeur, Yves, "Method 8290: Analytical Procedures and Quality Assurance for Multimedia Analysis of Polychlorinated Dibenzo-p-dioxins and Dibenzofurans by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry", USEPA EMSL, Las Vegas, Nevada, June 1987. (Revision 0, November 1990.)

USEPA Office of Water Regulation and Standards, Industrial Technology Division, "Method 1613: Tetra- through Octa- Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS", Revision A, April 1990.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 027

Analysis of Tetra Ethyl Lead

Tetra ethyl lead was analyzed by graphite furnace atomic absorption spectrometry (GFAAS, SW-846 7421) following extraction with toluene (ASTM D 3341-80). The methods were performed as follows:

Extraction:

Liquids (FWS samples 001, 002) - 25 ml sample were extracted with 50 ml of an iodine monochloride reagent and 25 ml of petroleum either. Solids (FWS samples 003, 004) - 10 g samples were extracted with 30 ml toluene for 1 hour, and then filtered. A 20 ml aliquot of the extract was then put through the above extraction with iodine monochloride and petroleum ether. The extract from both liquid and solid samples was then digested with nitric acid and brought to a volume of 100 ml for analysis.

Analysis:

Digested extracts, in an aqueous/dilute nitric acid matrix, were analyzed for lead by GFAAS.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 028

Total Petroleum Hydrocarbons

The sediment samples were freeze-dried and extracted in a Soxhlet extraction apparatus. The freeze-dried sediment samples were homogenized and a 15-gram sample was weighed into the extraction thimble. Surrogate standards and methylene chloride were added and the samples extracted for 12 hrs. The extracts were treated with copper to remove sulfur. Extract is rotovaped to 5 mL and then brought to dryness under a clean nitrogen stream. GC internal standards are added and the extract is run on gas chromatograph with flame ionization detector. TPH is determined by summing the total unresolved complex mixture (UCM) and the total resolved (all peaks in the chromatogram). The concentration is based on an average of the response factors for alkanes from n-C10 through n-C34. Chromatograms are also provided.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 029

Organophosphate Pesticides - Tissues

The tissue samples were extracted by EPA SW-846 method 3540 using a Soxhlet extraction apparatus. The tissue samples were homogenized and aliquots sample weighed, ground with 30 grams of anhydrous sodium sulfate and transferred into the extraction thimbles. Surrogate standards and methylene chloride were added and the samples extracted for 16-24 hrs. Lipids were removed from the extracts using gel permeation chromatography.

The quantitative analyses were performed using EPA SW-846 method 8141 by capillary gas chromatography (CGC) with a flame photometric detector for organophosphate pesticides.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 030

Organophosphate Pesticides - Sediments

The sediment samples were extracted by EPA SW-846 method 3550 extracted using sonication. The sediment samples were homogenized and an aliquot weighed

and ground with anhydrous sodium sulfate. Surrogate standards are added to the sample and the sample was extracted three times with 1:1 methylene chloride/acetone by sonication. Lipids were removed from the extracts using gel permeation chromatography.

The quantitative analyses were performed using EPA SW-846 method 8141 by capillary gas chromatography (CGC) with a flame photometric detector for organophosphate pesticides.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 031

EPA Method for Semivolatiles

The sediment samples are dried with sodium sulfate, surrogate standards are added and the sediment sample extracted with methylene chloride in a Accelarated Solvent Extraction (ASE) apparatus. Copper is added to remove elemental sulfur. The tissue samples are extracted by the NOAA Status and Trends Method (Qian et al., 1998). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and methylene chloride in a centrifuge tube. The extracts were purified by gel permeation chromatography as outlined by the EPA methods.

The quantitative analyses were performed by capillary gas chromatography (GC) with a mass spectrometer detector in the SCAN mode for semivolatile hydrocarbons (EPA) Contract Laboratory for Organic Analyses OLM04.2 and EPA 8270). The compound list is based on the EPA CLP protocol but analytes from SW846 Method 8270 have been added.

References:

Qian, Y., J. L. Sericano and T. L. Wade. (1998) Extraction Tissues for Trace Organic Analysis. In Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update (G. G. Lauenstein and A. Y. Cantillo, eds). NOAA Technical memorandum NOS ORCA 130. Pages 98 to 101.

U.S. Environmental Protection Agency. USEPA. Contract Laboratory Program Statement of Work for Organic Analysis. OLM04.2

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 035

Simultaneously Extracted Metals (SEM) -- Sediment

Simultaneously extracted metals (SEM) are those metals released during the analysis for acid volatile sulfides
(AVS) and are defined as those metals that are released upon exposure to 1N HCl at room temperature for 1 hour. Approximately 10 grams of wet sediment are acidified with 1N HCl, and reactive sulfides that are coverted to gaseous H2S are trapped as Ag2S and measured gravimetrically. The sediment is removed by filtration and the filtrate is brought to a final volume of 250 milliliters.

Metals in the digestate are determined by 3 techniques, depending upon concentration and element. Arsenic, selenium, cadmium, and lead are determined by graphite furnace AAS, in which electrical heating is used to produce an atomic cloud. Remaining elements (and Cd or Pb when in high concentration) are determined by atomic emission using an argon plasma.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 036

Methyl Mercury in Tissue

Tissue samples for methyl-mercury analysis are extracted by the method of Uthe et al. (1972) and Wagemann et al. (1997) and analyzed by gas chromatography with electron capture detector (GC/ECD). Briefly, wet (ca. 1 g) or dry tissue (0.2-0.5 g) samples are acidified with 4 ml of HBr (2.5N) and 8 ml of CuSO4 (0.1N) solutions. The aqueous solutions are extracted with 5-7 ml of toluene three times. The combined toluene extract are washed twice with 7 ml of Na2S2O3 (0.005N) aqueous solution. About 4 ml of HBr (2.5N) and 0.5 ml of NaBr (3N) are added to the combined Na2S2O3 aquous solution. The aqueous solution is extracted with 4 ml of toluene twice. The toluene extract is dried with anhydrous sodium sulfate. An aliquot of the final toluene extract is taken and a GC external standard (tetra-chlorinated m-xylene, TCMX) is added.

Methyl mercury in the toluene extract is analyzed by a GC/ECD equipped with a 30m x 0.25 mm (i.d.) DB-17 capillary column. The GC/ECD is programed to run from 100oC to 300oC in 20 minutes. GC/ECD calibration standards are made from methyl mercury chloride and are converted to methyl mecury bromide. Methyl mercury concentrations are reported as ppm of mercury.

References:

Uthe, J.F., Solomon, J. and Grift, B. (1972) Rapid semimicro method for the determination of methyl mecury in fish tissue. J. AOAC., 55: 583-589.

Wagemann, R., Trebacz, E., Hunt, R. and Boila, G. (1997) Percent methylmercury and organic mercury in tissue of marine mammals and fish using different experimental and calculation methods. Environ. Toxicol. Chem., 16:1859-1866.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 037

Chlorophylls and other Plant Pigments

Sample Preservation and Storage Upon arrival at the laboratory samples are inspected, logged in, and unique laboratory identification numbers are assigned. Chain-of-custody protocols are followed.

Instrument Analysis After extraction with acetone and adding international standard, twenty (20) l of the sample extract is injected onto the HPLC system using an autosampler. Instrument settings are:

Column Oven Temperature ambient
HPLC Pump Run Time 35 minutes

Solvents:
A: Methanol/0.05M ammonium acetate
B: acetonitrile
C: ethyl acetate

Gradient Elution
0 min 100% A
2 min 100% B
10 min 40% B and 60%C
20 min 100% B
28 min 100% A

Sample Analysis Once optimal HPLC operating parameters have been established and the instrument is functioning according to specifications (see above), 20 l of each sample/standard are injected directly onto the HPLC system consisting of a tertiary gradient pump and a Spherisorb C18 column using an autosampler. The response of the UV/Visible absorbance detector is recorded with at 436 nm.

Calculations Peak identification was based on the comparison of retention times with that of authentic standards. Compound quantitation was based on response factors determined from standard compounds relative to international standard, amount of

internal standard added to each sample, peak areas of the internal standard and each peak, and the sample amount.

Standards Individual pigment standard was obtained from US EPA, Sigma-Aldrich, and Hoffman-LaRoche. Canthaxanthin was used as internal standard and added to each sample prior to extraction. Two hundred (200) ng of canthaxanthin was generally added to each sample. All the sample preparation, extraction, and analysis were conducted under reduced light conditions to minimize the exposure to light and the light induced decompositions.

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Qian, Y., Jochens, A.E., Kennicutt II, M.C., Biggs, D.C., 2003. Spatial and temporal variability of phytoplankton biomass and community structure over the continental margin of the northeast Gulf of Mexico based on pigment analysis. Continental Shelf Research, 23, 1-17.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 038

Biomass Determination

Total Wet Weight

Samples which are taken in the field from a known area or volume of habitat are shipped to the lab. On receipt,
the excess water is removed with fine mesh netting (60 ?m). The sample is transferred to a pre-weighed jar and weighed. The difference of the jar weight and total weight is the total wet weight is determined. If the amount of biomass is small, the sample is filtered on to preweighed moist glass fiber filter using suction. After filtering, as much water as possible is sucked of the filter

Dry weight and ash weight determination

One aliquot of the wet sample was weighed and placed in a preweighed aluminium foil boat. This aliquot was dried in an oven at 70oC for a minimum of 24 hours. The dried samples were weighed on an analytical balances after cooling to room temperature. The samples were returned to the oven for another 2 hours and the reweighed after cooling. If the weight difference between the two weighings after

drying is less than 0.02 g, the last weight was used to calculated the percent solid of the sample. If the weight difference is larger than 0.02 g, the sample was returned to the oven and dried until constant weight was achieved.

The dry aliquot is placed in a preweighed, precombusted cruciable. The samples along with the cruciables were combusted at 550oC for 24 h. Upon cooling, the samples along with the cruciables were weighed on an analytical balances. The samples were returned to the oven for additional 12 hours before the second weighing. If the weight difference between the two weighings after combustion was less than 0.01 g, the last weight was used to calculate the ashfree dry weight of the sample. If the weight difference was larger than 0.01 g, the sample was returned to the oven and combusted until constant weight was achieved. The residue is the ash weight.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 039

Hydrazine Analysis in Sediments

Sample Preservation and Storage Upon arrival at the laboratory samples are inspected, logged in, and unique laboratory identification numbers are assigned. Chain-of-custody protocols are followed. Samples were stored at -20oC.

Extraction: Samples were extracted with 3 ml of 0.1N sulfuric acid and 1 ml of acetone twice. Extraction was performed with sonication. After centrifuge, the supernatant was transferred into a vial. The volume of the combined extracted was measured and 1 ml aliquot of the extract was transferred into a 2 ml vial. 0.5 ml of pentafluorobenzaldehyde solution (1 ml in 100 ml methanol) was added to each vial and heated at 70oC for 40 min to derivatize hydrazine.

Instrument Analysis After cooling, twenty (20) æl of the derivatized sample extract is injected onto the HPLC system using an autosampler. Hydrazine and 1,1'-dimethylhydrazine were analyzed on a 25 cm x 4.6 mm Spherisorb C18 column with methanol/water (80/20, v/v) as the mobile phase. Target analytes were detected at 313 nm.

Calculations Peak identification was based on the comparison of retention times with that of authentic standards. Compound quantitation was based on response factors determined from standard compounds, peak areas of each peak, and the sample amount.

Standards Hydrazine and 1,1'-dimethylhydrazine standards were obtained from Aldrich. A working solution of 1 ug/ml was prepared by dissolving appropriate

amount of the standards into 0.1N sulfuric acid solution. Calibration standards were prepared by derivatizing with pentafluorobenzaldehyde solution.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 040

The tissue samples were extracted by the NOAA Status and Trends Method (MacLeod et al., 1985) with minor revisions (Brooks et al., 1989; Wadeet al., 1988). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and methylene chloride in a centrifuge tube. The tissue extracts were purified by silica/alumina column chromatography to isolate the aliphatic and PAH/pesticide/PCB fractions. The PAH/pesticide/PCB fraction was further purified by HPLC in order to remove interfering lipids.

The sediment samples were dried with sodium sulfate and extracted using a DIONEX accelarated solvent extraction (ASE) system. The freeze-dried sediment samples were homogenized and a 10-gram sample was weighed into the extraction thimble. Surrogate standards and methylene chloride were added and the samples extracted. The extracts were treated with copper to remove sulfur and were purified by silica/alumina column chromatography (MacLeod et al., 1985; Brooks et al., 1989) to isolate the aliphatic and aromatic/pesticide/PCB fractions.

The quantitative analyses were performed by capillary gas chromatography (CGC) with a mass spectrometer detector in the SIM mode for pesticides or PCBs. Quantification is based on the primary ion and two additional masses for each analyte are also monitored for verification of the peak identification. The masses are based on EPA protocols such as EPA 680 for PCBs and 8270c for pesticides. All 209 PCB congeeners have been identified with many co-elutions. Total homologs and Arclors can also be determined.

Brooks, J.M., T. L. Wade, E. L. Atlas, M. C. Kennicutt II, B. J. Presley, R. R. Fay, E. N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

MacLeod, W.D., D. W. Brown, A. J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R. G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Compounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS. NOAA Tech. Memo. NMFS F/NWC-92.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 041

The tissue samples were extracted by the NOAA Status and Trends Method (MacLeod et al., 1985) with minor revisions (Brooks et al., 1989; Wade et al., 1988). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and methylene chloride in a centrifuge tube. The tissue extracts were purified by silica/alumina column chromatography and finally by HPLC in order to remove interfering lipids.

The sediment samples were dried with sodium sulfate and extracted using a DIONEX accelarated solvent extraction (ASE) system. The freeze-dried sediment samples were homogenized and a 10-gram sample was weighed into the extraction thimble. Surrogate standards and methylene chloride were added and the samples extracted. The extracts were treated with copper to remove sulfur and were purified by silica/alumina column chromatography (MacLeod et al., 1985; Brooks et al., 1989).

The quantitative analyses were performed by capillary gas chromatography (CGC) with a mass spectrometer detector in the SIM mode for polybrominated diphenyl ethers (PBDEs). Quantification is based on the primary ion and two additional masses for each analyte are also monitored for verification of the peak identification. The masses are based on EPA protocols such as EPA 1614 for PBDEs.

Brooks, J.M., T. L. Wade, E. L. Atlas, M. C. Kennicutt II, B. J. Presley, R. R. Fay, E. N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

MacLeod, W.D., D. W. Brown, A. J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R. G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Compounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS. NOAA Tech. Memo. NMFS F/NWC-92.

Wade, T.L., E. L. Atlas, J. M. Brooks, M. C. Kennicutt II, R. G. Fox, J. Sericano, B. Garcia, and D. DeFreitas (1988) NOAA Gulf of Mexico Status and Trends Program: Trace Organic Contaminant Distribution in Sediments and Oysters. Estuaries, 11, 171-179.

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Lab Name: Geochemical & Environmental Research Group, Texas A&M
Method Code 042

Determination of Triazines by GC/MS

The tissue samples were extracted by the NOAA Status and Trends Method (MacLeod et al., 1985) with minor revisions (Brooks et al., 1989; Wade et al., 1988). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and methylene chloride in a centrifuge tube. The tissue extracts were purified by silica/alumina column chromatography and finally by HPLC in order to remove interfering lipids.
The sediment samples were dried with sodium sulfate and extracted using a DIONEX accelarated solvent extraction (ASE) system. The freeze-dried sediment samples were homogenized and a 10-gram sample was weighed into the extraction thimble. Surrogate standards and methylene chloride were added and the samples extracted. The extracts were treated with copper to remove sulfur and were purified by silica/alumina column chromatography (MacLeod et al., 1985; Brooks et al., 1989).
The quantitative analyses were performed by capillary gas chromatography (CGC) with a mass spectrometer detector in the SIM mode for triazines. Quantification is based on the primary ion and two additional masses for each analyte are also monitored for verification of the peak identification. The method is based on EPA protocols specified in EPA 619.

REFERENCES

Brooks, J.M., T. L. Wade, E. L. Atlas, M. C. Kennicutt II, B. J. Presley, R. R. Fay, E. N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

MacLeod, W.D., D. W. Brown, A. J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R. G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Compounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS. NOAA Tech. Memo. NMFS F/NWC-92.

Wade, T.L., E. L. Atlas, J. M. Brooks, M. C. Kennicutt II, R. G. Fox, J. Sericano, B.

Garcia, and D. DeFreitas (1988) NOAA Gulf of Mexico Status and Trends Program: Trace Organic Contaminant Distribution in Sediments and Oysters. Estuaries, 11, 171-179.

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Environmental Services, Inc. (HES) Laboratory Methods

Select one of the links below to display the method descriptions associated with HES or <HERE> to go to the Start Page.

Method Code	Method Title
001	Elemental Analysis by Inductively Coupled Plasma Spectroscopy
002	Mercury by Cold Vapor Atomic Absorption
003	Mercury in Water by Cold Vapor Atomic Absorption
004	Arsenic by Graphite Furnace
005	Arsenic in Water by Graphite Furnace
006	Selenium by Graphite Furnace
007	Selenium in Water by Graphite Furnace
008	Extraction by Separatory Funnel
009	Extraction by Sonication
010	Extraction by Soxhlet
011	Determination of Percent Lipids
012	Determination of Percent Moisture
013	Gel-Permeation Chromatography Cleanup
014	Florisil Cleanup - PCBs Only
015	Florisil Cleanup - Organochlorine Pesticides and PCBs
016	Florisil Cartridge Cleanup for PCBs and Pesticides
017	Silica Gel Cleanup and Separation
018	Sulfur Cleanup
019	Moisture Determination
020	GC Analysis for Organochlorine Pesticides and PCBs
021	Total Organic Carbon
022	Grain Size

Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 001

Elemental Analysis by Inductively Coupled Plasma Spectroscopy

SCOPE:

This method is applicable to plant and animal tissue, soil/sediment, and water.

Sample Preparation:

1) Plant and Animal Tissue

- Digest 5.00 g of tissue in Teflon vessel with 5 mL nitric acid in microwave digester. Transfer into 50 mL volumetric flask and dilute to volume with 0.005% Triton X-100 solution. Filter.

2) Soil/Sediment

- Digest 1.00 g in covered Teflon beaker on hot plate using 10 mL nitric acid. Add 30% hydrogen peroxide in 1 mL aliquots until effervescence no longer occurs. Add 1.25 mL hydrochloric acid, heat 10 minutes, and transfer to a 50 mL volumetric flask. Dilute to volume with DDI water. Filter.

3) Water

- Digest 100.0 mL sample in Teflon beaker on hot plate with 0.5 mL nitric acid and 2.5 mL hydrochloric acid. Reduce volume to 15 to 20 mL. Transfer into 50 mL volumetric flask. Dilute to volume with DDI water. Filter.

PRINCIPLE:

Each analyte concentration in the sample solution is determined by comparing its emission intensity with the emission intensities of a known series of analyte standards. The analytical wavelengths are tabulated with the raw concentration data. Analytical data is corrected for background and interfering element effects by the spectrometer program. The detection limit of each analyte is listed in the data report with each respective unknown value, it is a function of the instrument detection limit (IDL), and the sample mass and volume to which it is diluted. With each batch of 20 samples of the same matrix type, at least one duplicate, one sample spike, one analytical blank, and one appropriate reference material are assayed.

REFERENCES:

Test Methods for Evaluating Solid Waste - EPA Publication No. SW-846, 3rd edition, Methods (3030, 3040, or 3050) and 6010, US EPA, Washington DC (revised December 1987).

Dahlquist, R.L. and Knoll, J.W., "Inductively coupled Plasma - Atomic Emission Spectrometry: Analysis of Biological Materials and Soils for Major, Trace, and Ultra-Trace Elements," Applied Spectroscopy, 32 (1) 1-29 (January/February 1978).

Official Methods of Analysis - 14th Edition, method 43.292-43.296, AOAC: Arlington, Virginia (1984).

Official Methods of Analysis - 1st Supplement, 14th Edition, Method 3.A01-3.A04, AOAC, Arlington, Virginia (1985).

U.S. Environmental Protection Agency Contract Laboratory program, Statement of Work, Inorganic Analysis, Multimedia, Multi- concentration, S.O.W. 7/88.

"Inductively Coupled Plasma-Atomic Emission Spectrometric Method of Trace Element Analysis of Water and Wastes," Method 200.7, edited by Theodore D. Martin and John F. Kopp, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

"Method Procedures, Analytical Chemistry Department, Inorganic Chemistry." Method MP-ICPS-MA, Hazleton Laboratories America, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 002

Mercury by Cold Vapor Atomic Absorption

SCOPE:

This method is applicable to most materials including animal tissues, plants, soils.

PRINCIPLE:

Sample weight: 2.00 g
Sample volume: 100 mL.

Samples are digested with a mixture of sulfuric and nitric acid. Mercury is reduced with sodium borohydride for determination. The amount of mercury is determined at a wavelength of 253.7 nm by comparing the signal of the unknown sample, measured by the atomic absorption spectrophotometer with the MHS-20 hydride generation unit, with the signal of the standard solutions.

Using a 2.0-g sample, the lowest detection limit of this assay is 0.025 ppm.

REFERENCES:

Digestion: Analyst, 86:608 (1961) with modifications.

Determination: Analytical Chemistry, 40:2085 (1968).

Test Methods for Evaluating Solid Waste, EPA Publication No. SW-846, 2nd Ed., Methods 3030, 3040 or 3050 and 7470, U.S. EPA: Washington, D.C. (revised April 1984).

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 003

Mercury in Water by Cold Vapor Atomic Absorption

SCOPE:

This method is applicable to drinking, surface, saline, and waste waters, and effluents.

PRINCIPLE:

Sample volume for digest: 50 mL
Final volume: 100 mL

Samples are digested with sulfuric acid, potassium permanganate, and potassium persulfate. Mercury is reduced with sodium borohydride for determination. The amount of mercury is determined at a wavelength of 253.7 nm by comparing the signal of the unknown sample, measured by the atomic absorption spectrophotometer with the MHS-20 hydride generation unit, with the signal of the standard solutions.

Using a 50-mL sample, the lowest detection limit of this assay is 0.0004 mg/L.

REFERENCES:

Method for Chemical Analysis of Water and Wastes, EPA Publication No. 600/4-79-020, Metals 1-19 and Method 245.2, U.S. EPA: Cincinnati, Ohio.

Test Methods for Evaluating Solid Waste, EPA Publication No. SW-846, 2nd Ed., Methods 3020 and 7470, U.S. EPA: Washington, D.C. (revised April 1984).

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 004

Arsenic by Graphite Furnace

SCOPE:

This method is applicable to animal tissues, plants, sediments, sludges, and soils.

SAMPLE PREPARATION:

1) Animal or Plant Tissue

- Digest 1.00 g with nitric acid in a microwave digestor. Transfer to 100 mL.

2) Sediment or Soil

- Digest 1.00 g with nitric acid and 30% hydrogen peroxide using covered glass beakers on hot plates. Transfer to 100 mL.

PRINCIPLE:

The amount of arsenic is determined at a wave length of 193.7 nm by comparing the signal of the unknown sample, measured by the graphite furnace atomic absorption spectrophotometer, with the signal of the standard solutions. The method of standard additions is used where interferences are indicated. Nickel matrix modification is employed in the analysis.

Using a 1.00-g sample, the lowest detection limit of this assay is 0.1 ppm.

REFERENCES:

Test Methods for Evaluating Solid Waste, EPA Publication No. SW-846, 2nd Ed., Methods 3030, 3040 or 3050 and 7060, U.S. EPA: Washington, D.C. (revised April 1984).

Contract Laboratory Program Statement of Work No. 785, Method 206.2 CLP-M, U.S. EPA: Cincinnati, Ohio.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 005

Arsenic in Water by Graphite Furnace

SCOPE:

This method is applicable to waters and aqueous wastes.

SAMPLE PREPARATION:

Sample volume: 25 mL (minimum)
Final volume: 25 mL

Digest the sample with nitric acid and 30% hydrogen peroxide using covered glass beakers and hot plates. Transfer to 25 mL.

PRINCIPLE:

The amount of arsenic is determined at a wavelength of 193.7 nm by comparing the signal of the unknown sample, measured by the graphite furnace atomic absorption spectrophotometer, with the signal of the standard solutions. The method of standard additions is used where interferences are indicated. Nickel matrix modification is employed in the analysis.

Using a 25-mL sample, the lowest detection limit of this assay is 1 mg/L.

REFERENCES:

Methods for Chemical Analysis of Water and Wastes, EPA Publication No. 600/4-79-020, Metals 1-19 and Method 206.2, U.S. EPA: Cincinnati, Ohio.

Test Methods for Evaluating Solid Waste, EPA Publication No. SW-846, 2nd Ed., Methods 3020 and 7060, U.S. EPA: Washington, D.C. (revised April 1984).

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 006

Selenium by Graphite Furnace

SCOPE:

This method is applicable to animal tissues, plants, sediments, sludges, and soils.

SAMPLE PREPARATION:

1) Animal or Plant Tissue

- Digest 1.00 g with nitric acid in a microwave digestor. Transfer to 100 mL.

2) Sediment or Soil

- Digest 1.00 g with nitric acid and 30% hydrogen peroxide using covered glass beakers on hot plates. Transfer to 100 mL.

PRINCIPLE:

The amount of selenium is determined at a wavelength of 196.0 nm by comparing the signal of the unknown sample, measured by the graphite furnace atomic absorption spectrophotometer, with the signal of the standard solutions. The method of standard additions is used along with nickel matrix modification in the analysis.

Using a 1.00-g sample, the lowest detection limit of this assay is 0.1 ppm.

REFERENCES:

Test Methods for Evaluating Solid Waste, EPA Publication No. SW-846, 2nd Ed., Methods 3030, 3040, or 3050 and 7740, U.S. EPA: Washington, D.C. (revised April 1984).

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 007

Selenium in Water by Graphite Furnace

SCOPE:

This method is applicable to waters and aqueous wastes.

SAMPLE PREPARATION:

Sample volume: 25 mL (minimum)
Final volume: 25 mL

Digest the sample with nitric acid and 30% hydrogen peroxide using covered glass beakers and hot plates. Transfer to 25 mL.

PRINCIPLE:

The amount of selenium is determined at a wavelength of 196.0 nm by comparing the signal of the unknown sample, measured by the graphite furnace atomic

absorption spectrophotometer, with the signal of the standard solutions. The method of standard additions is used along with nickel matrix modification in the analysis.

Using a 25-mL sample, the lowest detection limit of this assay is 1 mg/L.

REFERENCES:

Methods for Chemical Analysis of Water and Wastes, EPA Publication No. 600/4-79-020, Metals 1-19, and Method 270.2, U.S. EPA: Cincinnati, Ohio.

Test Methods for Evaluating Solid Waste, EPA Publication No. SW-846, 2nd Ed., Methods 3020 and 7060, U.S. EPA: Washington, D.C. (revised April 1984).

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 008

Extraction by Separatory Funnel

SCOPE:

This method covers the extraction of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in ground, surface, and waste waters.

SAMPLE PREPARATION:

Measure 1000 ml of aqueous sample and transfer to a two liter separatory funnel. Adjust the pH of the sample within the range of 5 to 9 using either 10 N sodium hydroxide or 1:1 sulfuric acid. Add 1.0 ml of the pesticide spiking solution to the matrix spike and the control spike. Add 500 ul of the 2,4,5,6-tetrachloro-mxylene (TMX) surrogate spiking solution to all samples and QC samples.

PROCEDURE:

Add 60 ml of methylene chloride to the separatory funnel, cap it and shake for two minutes. Allow the organic and aqueous layers to separate for about ten minutes and then drain the organic layer into a 250 ml erlenmeyer flask.

Repeat the above extraction two additional times with fresh portions of methylene chloride, combining the organic extracts in the erlenmeyer flask.

Pour the combined extract through a drying column containing enough anhydrous sodium sulfate to absorb any water. Drain the extract into a 500 ml Kuderna-Danish (K-D) flask fitted with a 10 ml concentrator tube. Attach a three ball snyder column to

the K-D flask and concentrate the extract on a hot water bath, adjusting the temperature such that the concentration is completed within 15-20 minutes.

When the apparent volume reaches approximately 5.0 ml, remove the K-D apparatus from the water bath and allow to drain and cool for at least 10 minutes. Add 50 ml of hexane to the K-D flask and return to the hot water bath, concentrating the extract to 5.0 ml.

REFERENCES:

Environmental Protection Agency, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods - EPA Publication No. SW-846," Method 3510, Office of Solid Waste and Emergency Response, Washington, D.C. (September 1986)

"Determination of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Ground, Surface, and Waste Waters." Method MP-FWSW-MA, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 009

Extraction by Sonication

SCOPE:

This method covers the extraction of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in soils and sediments.

SAMPLE PREPARATION:

Blend 20 grams of soil or sediment with 40 grams of anhydrous sodium sulfate in a 250 ml beaker. If there is not 20 grams of sample available, then remove at least one gram for percent moistures and weigh the remainder for extracting. For wet samples, more sodium sulfate may be required. If a sufficient amount has been added, the sample will appear granular. Add 1.0 ml of the pesticide spiking solution to the matrix spike and the control spike. Add 200 ul of the 2,4,5,6-tetrachloro-m-xylene (TMX) surrogate spiking solution to all samples and QC samples. Allow the soil/sediment and sodium sulfate to dry under a hood for a couple of hours, stirring it occasionally.

PROCEDURE:

Add 100 ml of methylene chloride:acetone (1:1). Place the ultrasonic probe 1/2 inch below the surface of the solvent but above the sediment layer and sonicate for 3 minutes using the 0.75 inch horn at full power with the pulse set at 50% duty cycle.

Decant and filter the extract through Whatman #4 filter paper into a 500 ml Kuderna-Danish (K-D) flask fitted with a 10 ml concentrator tube. Repeat the above extraction two more times with two additional 100 ml portions of methylene chloride:acetone (1:1) combining the resultant extracts.

Attach a three ball snyder column to the K-D flask and concentrate the extract on a hot water bath, adjusting the temperature such that the concentration is completed within 15-20 minutes.

When the apparent volume reaches approximately 5.0 ml, remove the K-D apparatus from the water bath and allow to drain and cool for at least 10 minutes. Bring up to a volume of 10 ml with methylene chloride if the sample is to be cleaned by GPC. It is recommended that GPC cleanup be used for very contaminated extracts. If the extract is not to be cleaned by GPC then exchange the solvent to hexane.

REFERENCES:

Environmental Protection Agency, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods - EPA Publication No. SW-846," Method 3550, Office of Solid Waste and Emergency Response, Washington, D.C. (September 1986)

"Determination of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Soils and Sediments." Method MP-FWSS-MA, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 010

Extraction by Soxhlet

SCOPE:

This method covers the extraction of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in biological tissues.

SAMPLE PREPARATION:

Blend 20 grams of ground tissue with 40 grams of anhydrous sodium sulfate in a 250 ml beaker. If there is not 20 grams of sample available the remove at least one gram for percent moistures and weigh the remainder for extracting. For

wet samples, more sodium sulfate may be required. If a sufficient amount has been added the sample will appear granular. Add 1.0 ml of the pesticide spiking solution to

the matrix spike and the control spike. Add 200 ul of the 2,4,5,6-tetrachloro-m-xylene (TMX) surrogate spiking solution to all samples and QC samples. Allow the ground tissue/sodium sulfate to dry under a hood for a couple of hours, stirring it occasionally.

PROCEDURE:

Load the prepared sample into the soxhlet extractor between two plugs of pre-extracted glass wool. Place 250 ml of methylene chloride into a pre-rinsed 500 ml erlenmeyer flask containing three to five teflon boiling chips. Attach the flask to the extractor. Add 100 ml of methylene chloride to the mixing beaker, swirl, and add the solvent to the extractor prior to attaching the condenser. Adjust the temperature of the heating mantle so that the extractors cycle at a rate of 12 to 15 cycles per hour. Allow the system to cycle for 16 to 20 hours.

Allow the extract to cool after the extraction is complete. Rinse the condenser with extraction solvent and drain the soxhlet apparatus into the bottom collection flask.

Pour the extract through a Whatman #4 filter into a 500 ml K-D flask fitted with a 10 ml concentrator tube. Attach a three-ball snyder column to the K-D flask and concentrate the extract on a hot water bath, adjusting the temperature so that the concentration is completed within 15 to 20 minutes.

When the apparent volume reaches approximately 5.0 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Bring up to a volume of 10 ml with methylene chloride.

REFERENCES:

Environmental Protection Agency, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods - EPA Publication No. SW-846," Method 3540, Office of Solid Waste and Emergency Response, Washington, D.C. (September 1986)

"Determination of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Biological Tissues." Method MP-FWST-MA, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 011

Determination of Percent Lipids

SCOPE:

This method covers the gravimetric determination of percent lipids in biological tissue samples.

PROCEDURE:

One milliliter of the 10 ml extract is placed into a preweighed aluminum weighing pan. The pan is allowed to sit lightly covered in a hood overnight to allow the solvent to evaporate. The pan is then weighed again. The following equation is then used to calculate the percent lipid:

((weight(g) of pan + lipid) - weight(g) of pan) x 10 ml x 100 = %lipid

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grams extracted

REFERENCES:

"Determination of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Biological Tissues." Method MP-FWST-MA, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 012

Determination of Percent Moisture

SCOPE:

This method covers the gravimetric determination of percent moisture in soil, sediment and biological tissue samples.

PROCEDURE:

One to 10 g of the sample is placed into a preweighed aluminum weighing pan. The pan is weighed again with the sample in it. The pan and sample are then placed into an oven at 105 C for 16 hours. The sample is allowed to cool in a desiccator and then weighed again. The following equation is used to calculate the percent moisture:

((mass(g) pan + wet sample) - (mass(g) pan + dry sample))x100=% moisture

---

grams of sample

If samples are to be calculated based on dry weight, the percent moisture is converted to a correction factor (M). The calculation of the factor is:

100 / (100 - % moisture) = M

REFERENCES:

Environmental Protection Agency, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods - EPA Publication No. SW-846," Method 3550, Office of Solid Waste and Emergency Response, Washington, D.C. (September 1986)

"Determination of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Soils and Sedients." Method -FWSS-MA, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 013

Gel-Permeation Chromatography Cleanup

SCOPE:

This method covers the cleanup of soil, sediment and biological extracts by gel-permeation chromatography (GPC).

PROCEDURE:

After extraction, the sample extracts are concentrated in Kuderna- Danish (K-D) apparatus and the volume is adjusted to 10 ml with methylene chloride. Five milliliters of this extract is then injected on an ABC Laboratories Model 1002B GPC system using a column packed with 70 g of S-X3 Bio-beads and methylene chloride as the carrier solvent. A dump, collect, and rinse cycle is then run which is consistent with exhibit D, section 7.1 of reference 1 below.

The collected fraction is then quantitatively transferred to a 500 ml K-D apparatus fitted with a 10 ml concentrator tube. A three ball snyder column is attached and the extract is concentrated on a hot water bath, adjusting the temperature such that the concentration is completed within 15-20 minutes.

When the apparent volume reaches approximately 5.0 ml, the K-D apparatus is removed from the water bath. 50 ml of hexane is added to the flask and it is returned to the hot water bath and the extract is concentrated to 5.0 ml.

REFERENCES:

USEPA Contract Laboratory Program, "Statement of Work for Organic Analysis, multi-media, multi-concentration", Document number OLMOl.O (March 1990) including revisions OLMOl.l (December l990 and OLM01.2 (January 1991)

"Determination of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Biological Tissues." Method MP-FWST-MA, Hazleton Laboratories America, Inc., Madison, Wisconsin.

Instrument Operating Procedure for Gel-Permeation Chromatograph, Method OP-6004-36, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 014

Florisil Cleanup - PCBs Only

SCOPE:

This method is applicable to any sample extract in hexane that requires additional cleanup before determination of polychlorinated biphenyls (PCBs) only. It does not apply when analyzing for organochlorine pesticides.

SAMPLE PREPARATION:

The sample extract should be at a volume of 5.0 ml in hexane.

PROCEDURE:

To a Florisil column, 19 mm id x 300 mm length with a 300 ml reservoir, is added a plug of glass wool, 20 g of Florisil and topped with 2 cm of anhydrous sodium sulfate. The Florisil is prerinsed with 150 ml of petroleum ether, taking the solvent down to the top of the sodium sulfate.

The sample extract is added to the column and drawn down to the top of the sodium sulfate. The column is then eluted with 250 ml petroleum ether at the rate of approximately 5 ml/minute. A single fraction is collected in a 500 ml erlenmeyer flask as the column is allowed to run dry.

The collected fraction is then quantitatively transferred to a 500 ml K-D apparatus fitted with a 10 ml concentrator tube. A three ball snyder column is attached and the extract is concentrated on a hot water bath, adjusting the temperature such that the concentration is completed within 15-20 minutes.

When the apparent volume reaches approximately 5.0 ml, the K-D apparatus is removed from the water bath and allowed to cool for at least 10 minutes. 50 ml of hexane is added to the flask and it is returned to the hot water bath and the extract is concentrated to 5.0 ml.

This method is the same as that in reference 1 except:

a) the Florisil is rinsed with 150 ml petroleum ether instead of 60 ml hexane.

b) the sample extract is eluted with 250 ml of petroleum ether and only one fraction is collected.

c) the florisil is not activated before use.

REFERENCES:

Environmental Protection Agency, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods - EPA Publication No. SW-846," Method 3620, Office of Solid Waste and Emergency Response, Washington, D.C. (Septeber 1986)

"Florisil Cleanup of Samples for PCBs", Method OP-6004-37, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 015

Florisil Cleanup - Organochlorine Pesticides and PCBs

SCOPE:

This method is applicable to any sample extract in hexane that requires additional cleanup before determination of organochlorine pesticides and polychlorinated biphenyls (PCBs).

SAMPLE PREPARATION:

The sample extract should be at a volume of 5.0 ml in hexane.

PROCEDURE:

To a Florisil column, 19 mm id x 300 mm length with a 300 ml reservoir, is added a plug of glass wool, 20 g of Florisil and topped with 2 cm of anhydrous sodium

sulfate. The Florisil is prerinsed with 150 ml of petroleum ether, taking the solvent down to the top of the sodium sulfate.

The sample extract is added to the column and drawn down to the top of the sodium sulfate. The column is then eluted with 250 ml of a mixed solvent containing 1% ethanol, 49% diethyl ether, and 50% petroleum ether at the rate of approximately 5 ml/minute. A single fraction is collected in a 500 ml erlenmeyer flask as the column is allowed to run dry.

The collected fraction is then quantitatively transferred to a 500 ml K-D apparatus fitted with a 10 ml concentrator tube. A three ball snyder column is attached and the extract is concentrated on a hot water bath, adjusting the temperature such that the concentration is completed within 15-20 minutes.

When the apparent volume reaches approximately 5.0 ml, the K-D apparatus is removed from the water bath and allowed to cool for at least 10 minutes. 50 ml of hexane is added to the flask and it is returned to the hot water bath and the extract is concentrated to 5.0 ml.

This method is the same as that in reference 1 except:

a) the Florisil is rinsed with 150 ml petroleum ether instead of 60 ml hexane.

b) the sample extract is eluted with 250 ml of a mixed solvent containing 1% ethanol, 49% diethyl ether, and 50% petroleum ether and only one fraction is collected.

REFERENCES:

Environmental Protection Agency, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods - EPA Publication No. SW-846," Method 3620, Office of Solid Waste and Emergency Response, Washington, D.C. (September 1986)

"Florisil Cleanup of Samples for Organochlorine Pesticides and PCBs", Method OP-6004-37, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 016

Florisil Cartridge Cleanup for PCBs and Pesticides

SCOPE:

This method is applicable to extracts which require additional cleanup prior to the determination of PCBs and Pesticides.

SAMPLE PREPARATION:

The sample extract should be in hexane.

PROCEDURE:

A florisil cartridge is placed onto a vacuum manifold and at least 5.0 ml of a hexane/acetone (90:10) rinse is pulled through the cartridge. A 1.0 ml sample extract is placed on the cartridge and eluted through it with 10 ml of hexane acetone (90:10). The extract is concentrated to the desired volume under a gentle stream of nitrogen.

REFERENCES:

USEPA Contract Laboratory Program, "Statement of Work for Organic Analysis, multi-media, multi concentration", Document number OLM01.0 (March 1990) including subsequent revisions.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 017

Silica Gel Cleanup and Separation

SCOPE:

This method is applicable to any sample extract in hexane which requires additional cleanup and the separation of polychlorinated biphenyls (PCBs) from many of the organochlorine pesticides.

SAMPLE PREPARATION:

The sample extract should be at a volume of 5.0 ml in hexane.

PROCEDURE:

The silica gel (100/200 mesh) is prepared by swirling it in a slurry of 40% acetonitrile and 60% methylene chloride, vacuum filtering, and then rinsing it successively with methylene chloride and hexane. It is then dried at 140 C overnight and deactivated with 0.5% (w/v) distilled water.

Fifteen grams of this silica gel is then slurried in petroleum ether, poured into a chromatography column, and topped with anhydrous sodium sulfate. The sample extracts are then drawn into the top of the column. The first fraction is eluted with

140 ml petroleum ether. The second fraction is eluted with 250 ml of a mixture of 1% acetonitrile, 19% hexane, and 80% methylene chloride (v/v).

The first fraction should include all PCBs, p,p'-DDE, hexachlorobenzene, and mirex. It may also include some portion of p,p'-DDT, o,p'-DDE, o,p'-DDT, and trans-nonachlor. The remaining portion of these 4 pesticides, along with all other organochlorine pesticides, will be found in the second fraction.

Both fractions are then quantitatively transferred to a 500 ml K-D apparatus fitted with a 10 ml concentrator tube. A three ball snyder column is attached and the extract is concentrated on a hot water bath, adjusting the temperature such that the concentration is completed within 15-20 minutes.

When the apparent volume reaches approximately 5.0 ml, the K-D apparatus is removed from the water bath and allowed to cool for at least 10 minutes. 50 ml of hexane is added to the flask and it is returned to the hot water bath. If the extract was cleaned by gel- permeation chromatography (GPC), it is concentrated to 5.0 ml. If it did not undergo GPC cleanup then it is concentrated to 10.0 ml.

REFERENCES:

Technical Operating Procedure, "Silica Gel Cleanup and Separation of Organochlorine Pesticides and PCBs", Method OP-6004-45, Hazleton Environmental Services, Inc., Madison, Wisconsin.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 018

Sulfur Cleanup

SCOPE:

This method is applicable to any sample extract which requires removal of sulfur before determination of organochlorine pesticides and/or polychlorinated biphenyls (PCBs) by gas chromatography.

PROCEDURE:

The sample extract is placed in a Teflon-sealed vial and 1 to 3 drops of mercury are added. The vial is then vigorously agitated on a vortex shaker for approximately 30 seconds. If a black precipitate forms, it is set aside and allowed to settle. The sample extract is then drawn off with a disposable pipet and transferred to a clean vial. This procedure is repeated until there is no formation of the precipitate.

REFERENCES:

Environnental Protection Agency, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods - EPA Publication No. SW-846," Method 3660, Office of Solid Waste and Emergency Response, Washington, D.C. (September 1986)

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 019

Moisture Determination

SCOPE:

This method is applicable to plant tissue, animal tissue, and soil/sediment.

PRINCIPLE:

The prepared sample is weighed in to a tared aluminum dish and is dried in an oven to constant weight (approximately 12-18 hours) at 100 C.

SENSITIVITY:

This method is capable of detecting 0.1% moisture.

REFERENCES:

Official Methods of Analysis, 15th Ed., Methods 926.08, 925.09, Assoc. of Off. Analytical Chemists, Arlington, VA (1990) modified.

USEPA Contract Laboratory Program, Statement of Work for Inorganics Analysis, Exhibit D, S.O.W. 3/90, Document No. ILMO1.0.

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 020

GC Analysis for Organochlorine Pesticides and PCBs

PROCEDURE:

All extracts are analyzed using gas chromatography with an electron capture detector. The sample extracts are analyzed and compared with external standards for qualitative and quantitative purposes. If the extract has been separated into a pesticide and a PCB fraction using silica gel, then each fraction is analyzed only for those analytes expected in the fraction.

Pesticide Analysis

The pesticides are analyzed on a gas chromatograph with dual wide-bore capillary columns and two electron capture detectors. An initial injection of an evaluation mixture containing 4,4'-DDT and endrin is made. Breakdown of the 4,4'-DDT into 4,4'-DDD and 4,4'-DDE and/or endrin into endrin ketone and endrin aldehyde must not exceed 20%.

Calibration standards are analyzed followed by the sample extracts. Ongoing standards are injected after a maximum of ten samples to verify the system is still within specifications. Sample chromatograms are compared to the standard chromatograms to identify peaks which match on two different GC columns. Identified pesticides are quantified using the initial standards.

PCB Analysis:

The individual PCBs are identified by pattern recognition. Individual PCB quantification is done by summing the areas of at least five sample peaks and also summing the same five peaks in the initial standard closest in area to the sample (for example, the A standard of Aroclor 1242, if the amount of Aroclor 1242 in the sample is near the detection limit). Total PCBs are obtained by summing the individual Aroclors present in the sample extract.

REFERENCES:

United States Environmental Protection Agency (EPA) Method 608, Federal Register, 49, (209):43321-43336, (October 16, 1984).

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 021

Total Organic Carbon

Total Organic Carbon

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Lab Name: Environmental Services, Inc. (HES) Laboratory Method Code: 022

Grain Size

Grain Size

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Laboratory and Environmental Testing, Inc. (LET) Laboratory Methods

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Method Code	Method Title
001	Homogenization
002	L9 - Freeze drying and % Moisture
003	L1 - HNO3/HClO4 Digestion with HCl
004	L2 - HNO3 Digestion
005	L3 - Digestion for Mercury
006	L4 - HNO3/HClO4 Digestion
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008	L6 - HNO3/H2O2 Digestion
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017	Perkin-Elmer Elan 5000 ICP/MS
018	ICP on Perkin-Elmer 4300 DV
019	L15-Pb in Blood-HGA

Lab Name: Laboratory and Environmental Testing, Inc. Method Code 001

Homogenization

1. Sample homogenization will depend on the sample type and size.

2. Water samples will not need to be homogenized.

3. For sampl es weighing less than 100 grams the whole sample will be freeze-dried first, and then homogenized, unless aliquots are being sent for Organic determination, then the sample would be homogenized first and an aliquot taken for freeze-drying.

4. Larger animal samples will be homogenized with a meat grinder. Then an aliquot of approximately 100 grams will be freeze-dried and then further homogenized using a blender, or if necessary, a Spex mixer mill with a Tungsten Carbide vial and ball.

5. Soil and Sediment samples will be mixed and aliquots of 100-200 grams taken for freeze-drying. After freeze-drying, soils will be sieved with a 20 mesh sieve and sediments will be sieved with a 10 mesh sieve followed by grinding with a Spex mixer mill, using a Tungsten Carbide vial and ball.

6. Plant samples will be freeze-dried and then homogenized with a blender, followed if necessary by grinding in a Spex mixer mill with a Tungsten Carbide vial and ball. If aliquots are being sent for Organic determinations, then the samples will be homogenized first, followed by freeze-drying, and further homogenization.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 002

L9 - Freeze drying and % Moisture

1. Choose an appropriately sized container for the sample. Usually a Whirl-Pak works best for tissue samples. If the sample weighs less than 50 grams and is not being split for organics then use the whole sample.

2. Weigh and record the weight of the bag. If the sample weighs more than 2 grams then a three-place balance should be used. Small samples may require the use of a four or five-place balance.

3. Weight the bag, record the weight and transfer the sample to the bag. Weigh the bag and sample and record the weight. Seal the container or bag and place in a freezer at least overnight or until frozen solid.

4. After the samples are frozen, they are ready to place in the freeze-drier. Turn on the freeze-drier and start the refrigeration. When the temperature reaches -50 C open the container or Whirl-Pak and place in the chamber of the freeze-drier. Close the chamber and start the vacuum pump.

5. Depending on the number of samples and the amount of water present freeze-drying may take 1 - 5 days. When the pressure stops going lower, the samples may be done. If, upon removal, the samples are still cold, place back in the freeze-drier for a longer period of time.

6. After the samples are dry, remove them from the chamber. Then seal the container and weigh on the same balance. Record the weight of the bag and dry sample.

7. Calculate the weight of the dry sample and the weight of the wet sample. To calculate % Moisture divide the weight of the dry sample by the weight of the wet sample, subtract 1 and multiply by 100. Ignore the - sign.

Notes:

1. If the samples do not require % Moisture, then all of the weighing steps can be eliminated.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 003

L1 - HNO3/HClO4 Digestion with HCl

1. Using a clean beaker or flask, write the sample number on the container in permanent black ink.

2. Weigh or measure the sample an d transfer to the container. Record the sample weight or volume on the paperwork.

3. Add 20 ml. of concentrated trace metal grade HNO3 and 2.5 ml. of concentrated trace metal grade HClO4. Then cover with a watch glass.

4. Place on a hot plate and adjust the temperature to allow reflux without significant loss of the acid.

5. Allow to reflux for 4 hours or overnight. Samples for Se or samples containing high lipid will require the longer reflux time.

6. After reflux, turn up the temperature to allow the HNO3 to be driven off without severe boiling, bumping or charring of the sample. When definite HClO4 fumes are observed, remove the sample from the hot plate and allow to cool.

7. Add 1.0 ml. of concentrated trace metal grade HCl to the samples and replace on the hot plate with watch glass, at the same temperature that was used to drive off the HNO3. Swirl the containers to help drive off the HNO3 residue and the HCl. After dense HClO4 fumes are observed allow the samples to reflux for 2 - 5 minutes. Remove from the hot plate and cool.

8. Add 2.0 ml. of concentrated trace metal grade HCl to the samples and place on the hot plate at a temperature that will boil water. When the solution is hot (2-5 minutes) add approximately 20 ml. of D.I. water and heat until clear or until the solution boils.

9. Remove and allow to cool. Dilute to 50.0 ml. with D.I. water and transfer to a clean 2 oz. bottle.

Notes:

1. Steps 3 and 8: for samples diluted to other than 50.0 ml. the volumes should be adjusted in relation to the other volume.

2. Step 9: the final volume may be other than 50.0 ml. If so an appropriate size container should be used.

3. This digestion can be used for water, biological fluids, plant and animal tissues, waste, soils and sediments. For soils and sediments the digestion will not be complete, being a hot acid extraction.

4. The digestion can be used for Flame AA, Flame Emission, ICP, Hydride for Se, and some elements by HGA.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 004

L2 - HNO3 Digestion

1. Weigh up to 0.5 g. of sample on a three place balance and transfer to a clean 100 ml. beaker or flask. For liquids use a calibrated pipet to transfer up to 25 ml. to the beaker or flask.

2. Add 20.0 ml. of concentrated trace metal grade HNO3 .

3. Place container on the hot plate or kjeldahl rack and turn on heat high enough to allow the HNO3 to reflux.

4. Allow samples to reflux for a minimum of two hours. Overnight may be needed for difficult samples.

5. After the reflux, turn up the heat to allow the HNO3 to evaporate to a volume of approximately 2 ml.

6. Cool, add a pproximately 20 ml. of D.I. water and heat to near boiling to allow any solids a chance to dissolve.

7. Dilute the sample to 50.0 ml. with D.I. water and transfer to a 2 oz. labeled bottle.

Notes:

1. The digestion can be used for water, biological fluids, plant and animal tissue, waste, soils and sediments. For soils and sediments the digestion is not complete, being a hot acid extraction.

2. The digests can be analyzed by Flame AA, HG A and ICP.

3. For some elements Teflon or quartz containers will give lower blanks.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 005

L3 - Digestion for Mercury

1. Weigh up to 0.5 g. of sample on a three place balance and transfer to a clean 50 ml. round bottom flask. For liquids use a calibrated pipet to transfer up to 10 ml. to a clean 50 ml. round bottom flask.

2. Add 5.0 ml. of concentrated trace metal grade HNO3 .

3. Place flask under condenser, making sure water is flowing through the condenser for reflux, and turn on heat high enough to allow the HNO3 to reflux.

4. Allow sample to reflux for two hours, then turn off the heat and allow to cool.

5. Rinse condenser into the flask with 1% HCl, remove the flask and rinse condenser tip into flask.

6. Dilute the sample to 50.0 ml. with 1% HCl and transfer to a 2 oz. labeled glass bottle.

Notes:

1. The digestion can be used for water, biological fluids, plant and animal tissue, waste, soils and sediments. For soils and sediments the digestion is not complete, being a hot acid extraction.

2. Soils and sediments should be diluted at least 1/3 before analysis.

3. Sample weights can be as high as 1 g.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 006

L4 - HNO3/HClO4 Digestion

1. Using a clean beaker or flask, write the sample number on the container in permanent black ink.

2. Weigh or measure the sample and transfer to the container. Record the sample weight or volume on the paperwork.

3. Add 20 ml. of concentrated trace metal grade HNO3 and 2.5 ml. of concentrated trace metal grade HClO4. Then cover with a watch glass.

4. Place on a hot plate and adjust the temperature to allow reflux without significant loss of the acid.

5. Allow to reflux for 4 hours or overnight. Samples containing high lipid will require the longer reflux time.

6. After reflux, turn up the temperature to allow the HNO3 to be driven off without severe boiling, bumping or charring of the sample. When definite HClO4 fumes are observed, allow the sample to reflux for 5 - 10 minutes. Then remove from the hot plate and allow to cool.

7. Place on the hot plate at a temperature that will boil water. Add approximately 20 ml. of D.I. water and heat until clear or until the solution boils.

8. Remove and allow to cool. Dilute to 50.0 ml. with D.I. water and transfer to a clean 2 oz. bottle.

Notes:

1. Step 3: for samples diluted to other than 50.0 ml. the volumes can be adjusted appropriately for the other volumes.

2. Step 8: the final volume may be other than 50.0 ml. If so an appropriately sized container should be used.

3. This digestion can be used for water, biological fluids, plant and animal tissues, waste, soils and sediments. For soils
and sediments the digestion will not be complete, being a hot acid extraction.

4. The digestion can be used for Flame AA, Flame Emission, ICP, Hydride for As and Se, and some elements by HGA.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 007

L5 - Magnesium Dry Ash

1. Weigh 0.5 g. of sample on a three-place balance and transfer to a cleaned 100 ml. glass beaker with etched numbers. Record the beaker number as well as the sample weight.

2. Wet with 3 ml. of methanol. Then add 5 drops of anti-foam agent, 10 ml. of 40% (W/V) Magnesium Nitrate Hexahydrate, 10 ml. of concentrated trace metal grade HNO3 and 2 ml. of concentrated trace metal grade HCl.

3. Cover with a watch glass and reflux on a hot plate overnight (8-12 hours) at low heat (70-80 C).

4. After reflux increase temperature to 200 C. Slide the watch glass to the side to allow for faster evaporation and cook to complete dryness. This may take 8-12 hours.

5. When no moisture is visible, cover fully with the watch glass and allow to cool.

6. Transfer samples to the cold muffle furnace and use the following program: Start at 250 C and ramp to 500 C at a rate of 1 degree per minute. When 500 C is reached hold for 3 hours then turn off and allow samples to cool to room temperature.

7. Place the cooled samples on a hot plate and add 20 ml. of 50% trace metal grade HCl. Allow the samples to gently boil for 1 hour. After 1 hour readjust volume to 20 ml. with 50 % HCl. Do not allow the samples to go dry. If necessary add more 50 % HCl during the heating.

8. Allow the samples to cool. Then dilute to 50.0 ml. with D.I. water and transfer to a clean 2 oz. labeled bottle.

Notes:

1. This digestion can be used for As or Se by Hydride Generation AA.

2. This digestion must be used on fish for As by Hydride Generation AA.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 008

L6 - HNO3/H2O2 Digestion

1. Weigh up to 0.5 g. of sample on a three-place balance and transfer to a clean 100 ml. beaker or flask. For liquids use a calibrated pipet to transfer up to 25 ml. to the beaker or flask.

2. Add 20.0 ml. of concentrated trace metal grade HNO3.

3. Place container on the hot plate or Kjeldahl rack and turn on heat high enough to allow the HNO3 to reflux.

4. Allow samples to reflux for a minimum of two hours. Overnight may be needed for difficult samples.

5. After the reflux turn up the heat to allow the HNO3 to evaporate to a volume of approximately 2 ml.

6. After cooling add 1 ml. aliquots of high purity H2O2 and heat. Do this until the liquid is colorless or pale yellow. Use a maximum of 20 ml. of H2O2.

7. Cool, add approximately 20 ml. of D.I. water and heat to near boiling to allow any solids a chance to dissolve.

8. Dilute the sample to 50.0 ml. with D.I. water and transfer to a 2 oz. labeled bottle.

Notes:

1. The digestion can be used for water, biological fluids, plant and animal tissue, waste, soils and sediments. For soils and sediments the digestion is not complete, being a hot acid extraction.

2. The digests can be analyzed by Flame AA, HGA and ICP.

3. For some elements Teflon or quartz containers will give lower blanks.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 009

L10 - Microwave Digestion

1. Weigh 0.5 g of dry sample into a clean Teflon digestion vessel. Record the weight to three decimal places.

2. Add 5.0 ml. of concent rated trace metal grade HNO3.

3. Loosely seal to allow release of pressure from the initial acid reaction with the sample.

4. After a few minutes open the vessel and add 1.0 ml of high purity H2O2.

5. Loosely seal the vessel to allow release of pressure.

6. Cap the vessel at the recommended pressure and place in the microwave. Run the program set up for this type of sample.

7. After the microwave heating is complete and the samples have cooled to room temperature, open the vessels and dilute the sample to 50.0 ml. with D.I. water and transfer to a clean 2 oz. plastic bottle. Any vessels that vented during the digestion will need to have the sample redigested and either use less sample or a longer ramp at the lower temperatures.

Notes:

1. Different sample types will require different heating programs to prevent losses due to exceeding the maximum vessel pressure.

2. To keep the same sample dilution, as little as 0.25 g of sample can be weighed and diluted to a final volume of 25.0 ml. using 1/2 of the HNO3 and H2O2.

3. This digestion can be used for Flame AA, HGA, CV, and ICP.

4. If Mercury is to be run, remove a 10 ml aliquot immediately after dilution and place in a plastic tube and add 100 microliters of concentrated Trace Metal grade Hydrochloric Acid.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 010

PARTICLE SIZE ANALYSIS HYDROMETER METHOD

Principle of the Method

1.1 This method quantitatively determines the proportions of sand, silt and clay soil particles based on their settling rates in aqueous solution using a hydrometer. Settling rates are based on the principle of sedimentation as described by Stokes' Law.

1.2 The use of an ASTM 152H-type hydrometer is based on a temperature of 20oC and a particle size density of 2.65 g cm-3.

1.3 Dispersion is achieved with a 5% solution of sodium hexametaphosphate.

Range and Sensitivity

2.1 The method has a detection limit of 2% sand, silt and clay on a dry basis.

Interferences

3.1 Soluble salts, organic matter, carbonates and iron oxides may need to be removed by pretreatment.

Precision and Accuracy

4.1 The method is reproducible to + 8%.

Equipment

5.1 Balance.

5.2 Mixer.

5.3 Sodium hexametaphosphate (Calgonä).

5.4 Settling cylinder with a one liter mark that is 36 + 2 cm from the bottom.

5.5 Hydro meter (Bouyoucus).

5.6 Plunger.

5.7 Timer.

5.8 Thermometer.

5.9 Watch glass.

Preparation

6.1 Prepare the sodium hexametaphosphate solution by dissolving 50 g in 1000 mL of deionized water.

Procedure

6.1 Weigh 40.0 g of air-dried soil.

6.2 Transfer soil into mixer. Add 100 mL of sodium hexametaphosphate solution and 300 mL of deionized water.

6.3 Mix 1 minute in the mixer on the low speed setting.

6.4 Transfer the suspension quantitatively into settling cylinder.

6.5 Add deionized water to bring volume to 1000 mL

6.6 Fill a cylinder with 100 mL of 5% hexametaphosphate and 900 mL of deionized water. This will be the blank sample.

6.7 Allow suspensions to come to room temperature (22 to 27oC)—approximately two hours.

6.8 Insert plunger into the cylinder and carefully move up and down to thoroughly mix the contents of the cylinder. Be sure to displace sediment on the bottom of the cylinder. Finish mixing with two to three smooth strokes.

6.9 Remove the plunger and lower the hydrometer into the suspension.

6.10 After 30 seconds from the plunger removal, record the hydrometer reading as hydrometer #1 reading. Record a reading on the blank.

6.11 Remove the hydrometer carefully, rinse the surface and wipe it dry.

6.12 Cover cylinders with watch glasses to prevent foreign material from entering solutions during the settling period.

6.13 After 6 hours record temperature and refer to the temperature correction table (taken from the Western States Laboratory Proficiency Testing Program (9.3). Do not move the cylinder or reshake the suspension during the standing period.

6.14 Reread hydrometer at the prescribed time. Record as hydrometer #2 reading. Repeat a reading on the blank.

Calculations

Table 1. Suspension temperature effect on time of hydrometer reading for clay determination

Temperature Settling time for clay

oC	**hours and minutes**
18	8:09
19	7:57
20	7:45
21	7:35
22	7:24
23	7:13
24	7:03
25	6:53
26	6:44
27	6:35
28	6:27

Classification of soil texture

Soil texture can be classified by the guide for textural classification from the USDA Natural Resource Conservation Service. In the USDA textural triangle below, the corners represent 100 percent sand, silt, or clay (gravel and organic soils are not included). The triangle is divided into 10 percent portions of clay, silt, and sand. Heavy lines show the divisions between the12 basic soil textural classes. If the percentage for any two of the soil separates is known, the correct textural class can be determined. However, the summation of the three percentages must total 100 percent. Sometimes the point representing the texture of a soil sample falls exactly on the line between two texture names. It is customary to use the finer texture class when this happens. For example, a sample containing 40 percent clay, 30 percent silt, and 30 percent sand is called clay rather than clay loam.

References

8.1 McElreath, D. L. and Johnson, G.V. 1990. Soil texture–hydrometer method. In Laboratory Procedures Manual. Oklahoma State University Soil, Water, and Forage Analytical Laboratory.

8.2 Michigan State University. Manual of Laboratory Procedures. Soil and Plant Nutrient Laboratory. Michigan State University. Dept. of Crop and Soil Sci. East Lansing, MI 48824.

8.3 Miller, R. O., J. Kotuby-Amacher, and J. B. Rodriguez. 1998. Western States Laboratory Proficiency Testing Program-Soil Plant and Analytical Methods. Ver. 4.10.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 011

SOIL ORGANIC MATTER Loss-On-Ignition

Principle of Method

1.1 This method estimates organic matter by measuring weight loss that results from the ignition of organic matter (Loss On Ignition, LOI) in a high temperature oven. It requires that soil is adequately dried before ignition, and then organic matter is quantitatively destroyed without altering other soil constituents such that soil weight is changed.

1.2 Various methods using different heating times and temperatures have been investigated. These are noted in the references 8.1, 8.2 and 8.4. A minimum heating temperature of 105o C for 24 hours is necessary to eliminate hygroscopic water and water of hydration from minerals such as gypsum. Excessive heating may result in weight loss associated with carbonates, structural water of silicate clays, oxidation of Fe+2 and dehydration of salts. The method noted here is adapted from Storer (8.7).

Range and Sensitivity

2.1 This method has been used with soils ranging in organic matter content from <1 to 45%. It has a sensitivity of 0.2 to 0.5% organic matter.

Sources of Error

2.1 Loss of water from incomplete preheating dehydration can result in over-estimation of organic matter. The problem is particularly likely in high clay soils with low organic matter, such as with subsoils. The method is not considered suitable for calcareous soils.

Precision and Accuracy

3.1 This method directly estimates organic matter, and it correlates well with organic carbon determinations. Yet it results in greater estimates of organic matter than with methods previously used. So organic matter is estimated from this method by regression of data with other established methods.

3.2 Mineral composition and soil horizons may affect LOI results.

3.3 Consistent analytical results are possible with a range of sample sizes, ashing vessels, ashing temperatures and length of ashing times.

3.4 Repeated analyses should provide results with a maximum coefficient of variability of 1 to 4%.

Equipment

5.1 NCR-13 2-g scoop.

5.2 10 mL glass beakers

5.3 Oven capable of heating to approximately 360o C.

5.4 Stainless steel racks for holding beakers.

5.5 Balance sensitive to ± 1 mg in draft-free environment.

Procedure

6.1 Scoop or weigh 2 g of air-dried soil into tared 10-mL glass beakers.

6.2 Dry for at least 2 hours at 150o C.

6.3 Record pre-weight to + 1 mg.

6.4 Heat at 360oC for 2 hours after oven temperature reaches 360o C.

6.5 Move the beakers from the oven to a lab bench; allow cooling approximately 15 minutes to cool. NOTE: if samples can not be weighted immediately then they should be re- dried in an oven at 150o C for 2 hours prior to recording post-weight.

6.6 Record post-weight to + 1 mg.

Calculations

7.1 Calculate loss of weight on ignition (LOI)

7.2 Estimate soil organic matter

Estimation of organic matter from LOI is done by regression analysis. Sixty soils were selected at random from those submitted to the lab. LECO-C was determined on these samples as well as % LOI. Percent organic matter was determined from LECO-C by multiplying % C by 1.79. LOI was regressed on LECO-OM forcing the intercept through the origin. The resulting equation is used to convert % LOI values into % organic matter. The equation is:

References

7.1 Ball, D. F. 1964. Loss-on-ignition as an estimate of organic matter and organic carbon in non-calcareous soils. J. Soil Sci. 15:84-92.

7.2 Combs, M. and M. V. Nathan. 1998. Soil Organic Matter. Ch. 12. In J. R. Brown (ed.). Recommended Chemical Soil Test Procedures for the North Central Region, N.C. Reg. Res. Pub. 221 (Revised). (Mo. Agric. Exp. Stn. SB 1001).

7.3 Goldin, A. 1987. Reassessing the use of loss-on-ignition for estimating organic matter content in non-calcareous soils. Commun. Soil Sci. Plant Anal. 18:1111-1116.

7.4 Handbook on reference methods for soil analysis. 1992. Soil and Plant Analysis Council, Inc. Georgia University Station, Athens, GA 30612-0007.

7.5 Michigan State University. Manual of Laboratory Procedures. Soil and Plant Nutrient Laboratory. Michigan State University. Dept. of Crop and Soil Sci. East Lansing, MI 48824.

7.6 Nelson, D. W. and L. E. Sommers, 1996. Total carbon, organic carbon, and organic matter. In D. L. Sparks (ed.). Methods of Soil Analysis, Chemical Methods, Part 3. Soil Science Soc. Am. Madison, WI.

7.7 Storer, D. A. 1984. A simple high sample volume ashing procedure for determining soil organic matter. Commun. Soil Sci. Plant Anal. 15:759-772.

7.8 Western States Laboratory Proficiency Testing Program, Soil and Plant Analytical Methods. 1998. Version 4.10.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 012

Hydride Generation AA

Turn on the computer, printer, 3100, FIAS 200.and Argon. Place the appropriate lamp in the instrument and if an EDL turn to its required power. Place the furnace in the burner compartment if it is not already present.

When the computer is ready double click on the WinLab Analyst icon. If the technique is not already FI-Hydride then click on technique and change to FI-Hydride. After the computer has confirmed the IEEE connections are OK, click on Workspace and double click fias.fms. When the screens come up double click on the method and double click on either the Se-Fias or As-Fias method. Click on FIAS and turn on the cell.

When the lamp has had time to warm up click on lamps and enter the element and click on EDL. Check lamp alignment and wavelength to give the maximum signal. Close lamps.

Prepare the 10% HCl, 0.2% NaBH4-0.05% NaOH, Calibration standards, and check standards. Change the FIAS tubing and mixing cell if it is not already the set for this element. Change the position of the tubing or new tubes, if both positions have been used.

Check the alignment of the furnace in the light path by clicking on Tools and Continuous graphics. Autozero, then check all three positional knobs to get the lowest reading. Autozero whenever necessary.

Start the pumps and place the tubes in the HCl and Borohydride. Run a 5 or 10 PPB standard until the
sensitivity has stabilized and consecutive readings vary by less than 2%.

Enter the samples to be run into the Sample Information File. Enter a name for the Data file, and make sure that print log and store data are checked. When the instrument is ready click on Analyze All.

Calibration is done with 0, 1.0, 5.0, 15.0 PPB. QC checks are 10.0 and a known Reference sample (Usually ERA). The 5.00 PPB standard is checked every 10 tubes and if is more than 5% from 5.00 the instrument is recalibrated. If the value is more than 10% from 5.00, then the last 10 samples must be rerun.

After the analysis is finished, rinse system with D.I. water, turn off the pumps (release the pressure), turn off the EDL lamp, the Argon, FIAS and 3100. Click on File then Exit to close the WinLabs Analyst.

Click on WinLab Reformat Icon. Click on Open Design. Pick the design for As or Se FIAS. Then Browse and find the file name given the data. Place a 3.5" disk in the computer and click on Save Results.

Transfer disk to computer and using Excel calculate the results.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 013

Cold Vapor AA

Turn on the computer, printer, 3100, FIAS 200,and Argon. Place the appropriate lamp in the instrument and if an EDL turn to its required power. Place the furnace in the burner compartment if it is not already present.

When the computer is ready double click on the WinLab Analyst icon. If the technique is not already FI-Hydride then click on technique and change to FI-Hydride. After the computer has confirmed the IEEE connections are OK, click on Workspace and double click fias.fms. When the screens come up double click on the method and double click on the Hg-CV method. Click on FIAS and turn on the cell.

When the lamp has had time to warm up click on lamps and enter the Hg and click on EDL. Check lamp alignment and wavelength to give the maximum signal. Close lamps.

Prepare the 10% HCl, 5% Stanous Chloride-10% HCl, Calibration standards, and check standards. Change the FIAS tubing and mixing cell if it is not already the set for Mercury. Change the position of the tubing or new tubes, if both positions have been used or determining a different element.

Check the alignment of the furnace in the light path by clicking on Tools and Continuous graphics. Autozero, then check all three positional knobs to get the lowest reading. Autozero whenever necessary.

Start the pumps and place the tubes in the HCl and Stanous Chloride. Run a 10 or 20 PPB standard until the sensitivity has stabilized and consecutive readings vary by less the 2%.

Enter the samples to be run into the Sample Information File. Enter a name for the Data file, and make sure that print log and store data are checked. When the instrument is ready click on Analyze All.

Calibration is done with 0, 1.0, 5.0, 30.0 PPB. QC checks are 10.0, 20.0 and a known Reference Sample(Usually ERA). The 5.00 PPB standard is checked every 10 tubes and if is more than 5% from 5.00 the instrument is recalibrated. If the value is more than 10% from 5.00, then the last 10 samples must be rerun.

After the analysis is finished, rinse system with D.I. water, turn off the pumps (release the pressure), turn off the EDL lamp, the
Argon, FIAS and 3100. Click on File then Exit to close the WinLabs Analyst.

Click on WinLab Reformat Icon. Click on Open Design. Pick the design for Hg-CV. Then Browse and find the file name given the data. Place a 3.5" disk in the computer and click on Save Results.

Transfer disk to computer and using Excel calculate the results.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 014

Flame AA

Turn on the computer, printer, 3100, FIAS 200, Acetylene and Compressed Air. Place the appropriate lamp in the 3100 and if an EDL turn to its required power. Place the burner head in the burner compartment if it is not already present.

When the computer is ready double click on the WinLab Analyst icon. If the technique is not already Flame then click on technique and change to Flame. After the computer has confirmed the IEEE connections are OK, click on Workspace and double click Manual.fms. When the screens come up double click on the method and double click on the Method for the element you are going to run.

Click on lamps and enter the element and click on EDL or HCL depending on which is being used. Check lamp alignment and wavelength to give the maximum signal. Close lamps.

Prepare the Calibration standards and check standards. The acid matrix should match for samples, standards and check standards.

Light the flame and check the alignment of the burner in the light path by clicking on Tools and Continuous graphics. Autozero, then check all three positional knobs to get the highest reading while aspirating a standard.

Start the pumps and place the tubes in the HCl and Borohydride. Run a 5 or 10 PPB standard until the sensitivity has stabilized and consecutive readings vary by less the 2%.

Enter a name for the Data file, and make sure that print log and store data are checked. Calibration is done with 0,and 1 – 3 standards depending on the element and the range of concentration of the samples. After calibration, read the 0 and three to five standards covering the range of the calibration. Analyze the check standard. If this is acceptable then read the zero standard 10 times to determine the detection limit. Run a check standard every 10-12 samples and if it is more than 5% from its true value, then recalibrate. If it is more than 10% from its true value then recalibrate and rerun the samples since the last standard.

After the analysis is finished, rinse the system with D.I. water and turn off the flame. If using an EDL lamp turn it off. Turn off the the FIAS, 3100, Compressed Air and Acetylene.

Click on File then Exit to close the WinLabs Analyst.

Click on WinLab Reformat Icon. Click on Open Design. Pick the design for the element run. Then Browse and find the file name given the data. Place a 3.5" disk in the computer and click on Save Results.

Transfer disk to computer and using Excel calculate the results.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 015

ICP - Jarrell-Ash Model 61

Turn on the Instrument power, PM Tubes switch and hit reset. Turn on the Line and Control on the RF Generator. Turn on the Water and Argon. Turn on the computer and double click on the JAICP Icon.

Place fresh D.I. water in the rinse beaker and start the peristaltic pump. After a few minutes, light the Plasma. Allow the instrument to warm up for 1 hour after the Plasma is lit.

Prepare standards and check standards in the same acid type and concentration as the samples.

After warm up is complete, check the profile with the Mercury light and adjust the alignment to +/- 0.1 spectrum shifter units of zero. Load the method to be run and calibrate. After calibration run the detection limit by changing to 10 integrations and running the zero standard. Run the check standard(s). If everything is acceptable start the analysis.

Run the Standards every 10-12 samples and check the profile after each set. If the profile is off from zero by more than 0.1 readjust the profile. If the standards are off more than 10% recalibrate and rerun the last set of samples.

If any samples have high concentrations of Fe, Al or unusual levels for other elements then run a high purity sample for those elements to allow Spectral Interferences to be checked.

After the analysis is completed turn off the Plasma, Argon, Water, and Pump. Convert the data to Excel using Enable and place on a 3.5" disk. Calculate the data using Excel, being sure to correct for Blank and Spectral Interferences.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 016

Graphite Furnace using the 5100 Zeeman

Turn on the Furnace Coolant, Argon, Computer, Furnace, Zeeman Power Supply and 5100 in that order. Make sure the lamp is in the right position in the turret, and if using an EDL turn on the lamp to the proper power.

Double click on the AA WinLab Analyst icon. After the ZHGA-600 and 5100 icons have a green check by them, click on workspace. Double click on LET.fm, then double click on the method. Choose the method to be run and double click on the name. Click on Browse by the Sample Information File, and then pick one for standards. (Example: Pbstds.sif) Click on sample information icon to edit the file with sample names, dilutions, etc. Click Browse for the Results Data Set and enter the name of the file to store data. (Example: Pb010101) Click on Analyze. When samples have been prepared and ready for analysis, click on Analyze All if doing calibration or Analyze Samples if just running samples. The instrument is usually calibrated with a zero and one standard. Then a zero and 3-5 standards are run to check the calibration, followed by an instrument check standard and detection limit. If this is acceptable then the samples are run. Be sure to check that the correct modifier is being used for the element being run. Some samples may require the method of Standard Additions.

After the analysis is completed, close AA WinLab Analyst, turn off furnace coolant, Argon, 5100, Zeeman Power supply, Furnace and EDL power supply. Double click on the WinLab Reformat icon. Click on Open Design and choose the design for your element and double click on the name. Click on Browse and find the data file you want to reformat. Double click on the name. Make sure there is a floppy disk in the disk drive and click on Save Results. Transfer to another computer and calculate using Excel.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 017

Perkin-Elmer Elan 5000 ICP/MS

Turn on the instrument control computer. Enter elan at login.

Turn on the instrument and set up with RF power 1.0 kw, Ar floe 15 l/min plasma, 0.75 l/min nebulizer, 0.90 l/min auxilary flow, Ion lens P, 50, B 58, S2 17, E1 28, Read Delay 45 second and Method USDI 2A.

Set up peristaltic pump for 2-channel IS operation using Rh. Turn on the plasma and turn on peristaltic pump as soon
as plasma is on. Allow plasma to warm up at least 30 minutes before starting analysis. Fill out ICP-MS Data Log.

Under the Applications menu select Quantitative Analysis, load method USDI 2A.

Run 1% HNO3 as a blank. The Rh should read more than 20,000 c.s. If not adjust the x-y position and nebulizer flow or perform CEM optimization. Record the Rh count rate on the ICP-MS data log sheet.

Prepare the sample table for the samples to be run. Standards should be the same acid and concentration as the samples. Calibrate the instrument. Then run Detection Limit and Check samples. Run Standards after every 10-12 samples.

After analysis is complete, select "Report" on the screen. Choose "Computer Format to File". Click on "To File". Give a file name using the convention dmmddyy.txt, mmddyy being month day and year.

Reprocess the data. When finished, select "Report". De-select by clicking on "Computer Format to File". Then click on "Report" again.

Select a new screen by pressing Alt-F1. Enter "vipx". Then change directory by typing "cd elan", "cd data", or "cd reports".

Insert a 3.5 " disk into the drive and then enter "Copy" DMMDDYY.TXT, A:.

Return to Elan window by pressing Alt-F1. Select :File" the Close Application". Select "Shutdown" under ‘Application Menu. Wait for "Safe to power off" message to appear. Then turn off the power to the computer. Turn off RF power supply to the Elan 500.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 018

ICP on Perkin-Elmer 4300 DV

Make sure the instrument, Chiller, Air compressor, and gases are on, and at the proper temperatures and pressures. Turn on the computer and double click on the WinLAb32 icon.

Prepare standards and check samples to match the acid matrix of the samples to be analyzed. Change the pump tubing.

Click on "file", then "Open", and then "Method". Click on the method to be used and then click "OK", TO start the ICP program and call up the Method with the elements to be determined.

Click on the Plasma icon, and click on pump to start the pump and make sure the tubes are in the pump properly. Start the plasma by clicking the "On" icon. Click on the X in the upper right corner to close the Plasma Control. Allow the instrument to warm-up while the samples and standards are loaded into the auto-sampler racks. If the Sample Info table was not filled out previously, then fill in the sample information and save the table using the Batch ID.

Before starting the run, check the Hg wavelength by clicking on "Tools", and then "Spectrometer Control". Click on Hg Realign. When that is complete, aspirate a 10.0 Mn Standard and click on "Align View". After Align View is completed, close the box.

When ready to start analysis, click on the "Auto" icon, make sure that the data is being stored in a file with the correct name for the Batch, and that the right method is being used. Click the "Analyze" icon and click on "Analyze All".

When the run is completed, click on "File", then "Utilities", then "Data Manager". Highlight the file, and then click on "Export" icon. Click "Use Existing Design". Click "Browse" and choose the appropriate template (usually LET-ICP). Click "Open", place a disk in the "A" drive, and click "Finish". Click on "Export Data" to transfer data to disk in Drive "A".

Transfer data to the main computer and calculate the final Concentrations.

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Lab Name: Laboratory and Environmental Testing, Inc. Method Code 019

L15-Pb in Blood-HGA

METHOD/PROCEDURE

1. Prepare an adequate volume of matrix modifier to dilute the samples and perform the analysis. The matrix modifier is 0.2% HNO_3, 0.5% Triton-X 100 and 0.2% $NH_4H_2PO_4$. Samples are diluted 1 part Blood and 9 parts Modifier.

2. Set up Perkin-Elmer 5100 Zeeman for Lead. Wavelength 283.3 with an EDL lamp. Furnace program is dry 110 C with 25 second ramp and 25 second hold. Char step is 800 C with a 15 second ramp and 25 second hold. A cool down to 20 C is next with a 1 second ramp and 9 second hold. Atomization is 1900 C with a 0 second ramp and 5 second hold, followed by a clean out at 2600 C with a 1 second ramp and a 5 second hold.

3. The Instrument is calibrated with a 25 or 50 ppb standard, followed by running a standard curve of 5 to 6 values between 2.5 and 100 ppb.

4. Lead is determined in the samples with a 14 ul injection. The detection limit is determined by injecting the 2.5 ppb standard 7 times and taking 3 times the standard deviation of the mean.

5. Blanks are the Triton-X Modifier and Duplicates are separate dilutions of the blood. Spikes are diluted separately and an aliquot of a Lead standard added to give 25 ppb spike. Reference material is a BLLRS Blood supplied by CDC, also diluted 1:10 like the bloods.

6. If sample concentration is above 50 ppb, further dilution of the blood is made with the riton-X Modifier.

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Midwest Research Institute (MRI) Laboratory Methods

Select one of the links below to display the method descriptions associated with MRI or <HERE> to go to the Start Page.

Method Code	Method Title
001	Analytical Methodology for Mercury in Sediment
002	Analytical Methodology for Mercury in Tissue
003	Analytical Methodology for Mercury in Water
004	Analytical Methodology for ICP Scan for Water
005	Analytical Methodology for ICP Scan for Sediment
006	Analytical Methodology for ICP Scan for Tissue
007	Analytical Methodology for Arsenic and Selenium in Tissue
008	Analytical Methodology for Arsenic and Selenium in Water
009	Analytical Methodology for Arsenic, Selenium and Cadmium in Soil
010	Homogenization and Moisture Determination of Tissue
011	Homogenization and Moisture Determination of Sediments
012	Analytical Methodology for ICP Scan for Water using Microwave Digestion
013	Analytical Methodology for Selenium in Water by Hydride Atomic Absorption
014	Analytical Methodology for Selenium in Soil by Hydride Atomic Absorption
015	Analytical Methodology for GFAA Analysis of Arsenic, Selenium and Cadmium in Water using Microwave Digestion
016	Analytical Methodology for Total Cyanide in Sediment
017	Analytical Methodology for Total Organic Carbon in Sediment/Soil
018	Analytical Methodology for Grain Size in Sediment/Soil
019	Tissue Percent Moisture Determination (Oven-Drying Method)
020	Analytical Method for Mercury in Water by FIMS CVAAS
021	Analytical Method for Mercury in Sediment by FIMS CVAAS
022	Analytical Method for Mercury in Tissue by FIMS CVAAS

023 Analytical Method for Trace Elements in Water by ICPMS

024 Analytical Method for Trace Elements in Tissue by ICPMS

025 Analytical Method for Trace Elements in Sediment by ICPMS

026 Microwave Digestion and the Analysis of Trace Elements in Sediment

027 Procedure for Moisture Determination and Homogenization of Solid Samples

Lab Name: *Midwest Research Institute* Method Code: 001

Analytical Methodology for Mercury in Sediment

Summary: A 0.2 gram fresh weight aliquot is digested in sulfuric and nitric acids at 95oC for two minutes then cooled. Potassium permanganate and hydroxylamine sulfate are added to the digestate and the sample is returned to the water bath for an additional 30 minute digestion at 95oC. Excess permanganate is reduced with potassium persulfate and the sample is diluted to 200 mL. The determination is performed by cold vapor atomic absorption using a PSA Merlin Plus mercury analyzer. The nominal detection limit is 0.2 ug/g.

Reference:

U.S. Environmental Protection Agency, Office of Water Regulations and Standards, Method 1620, Draft, September 1989.

U.S. Environmental Protection Agency, Contract Laboratory Program Statement of Work for Inorganics Analysis of Multi-Media Multi- Concentration Samples, Document No. ILM03.0.

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Lab Name: *Midwest Research Institute* Method Code: 002

Analytical Methodology for Mercury in Tissue

Summary: A 0.5 gram aliquot of wet homogenized tissue is digested in sulfuric and nitric acids at 60oC for one hour. Potassium permanganate and hydroxylamine sulfate are added to the digestate and the sample is returned to the water bath for an additional 30 minute digestion at 95oC. Excess permanganate is reduced with potassium persulfate and the sample is diluted to 200 mL. The determination is performed by cold vapor atomic absorption using a PSA Merlin Plus mercury analyzer. The nominal detection limit is 0.08 ug/g on a wet weight basis.

Reference:

Methods For The Determination of Metals in Environmental Samples, Environmental Monitoring Systems Laboratory, Office Of Research and Development, U.S. Environmental Protection Agency, Report No. EPA-600/4-91-010 (1991) Method 245.6.

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Lab Name: *Midwest Research Institute* Method Code: 003

Analytical Methodology for Mercury in Water

Summary: A 100 mL aliquot of water is digested using sulfuric acid, nitric acid, potassium permanganate, and potassium persulfate for one hour in a 95oC water bath. Excess permanganate is reduced with sodium chloride-hydroxylamine sulfate persulfate and the sample is diluted to 200 mL. The determination is performed by cold vapor atomic absorption using a PSA Merlin Plus mercury analyzer. The nominal detection limit is 0.2 ug/L.

Reference:

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Governmental Printing Office, Washington, DC, SW-846, Method 7470 (1986).

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Lab Name: *Midwest Research Institute* Method Code: 004

Analytical Methodology for ICP Scan for Water

Summary: 100 mL of water is combined with 10 mL of concentrated nitric acid and evaporated gently to approximately 10 mL(1). The contents are transferred to a 100 mL volumetric flask and diluted to volume using Milli-Q reagent grade water. The analyses are performed using a Thermo Jarrell Ash Model 61E simultaneous inductively coupled plasma emission spectrometer (2).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Governmental Printing Office, Washington, DC, SW-846,

Method 3020 (1986).

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 6010A (1986).

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Lab Name: *Midwest Research Institute* Method Code: 005

Analytical Methodology for ICP Scan for Sediment

Summary: A one gram aliquot for dried coarsely ground soil is transferred to a beaker and metals are extracted using nitric acid and hydrogen peroxide (1). The contents are transferred to a 100 mL volumetric flask and diluted to volume using reagent grade water. The analyses are performed using a Thermo Jarrell Ash Model 61E simultaneous inductively coupled plasma emission spectrometer (2).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office,

Washington , DC , SW-846, Method 3050A (1986).

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 6010A (1986).

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Lab Name: *Midwest Research Institute* Method Code: 006

Analytical Methodology for ICP Scan for Tissue

Summary: A one gram aliquot of freeze dried coarsely ground sample is weighed and digested using nitric acid and hydrogen peroxide (1). The contents are transferred to a 100 mL volumetric flask and diluted to volume using reagent grade water. The analyses are performed using a Thermo Jarrell Ash Model 61E simultaneous inductively coupled plasma emission spectrometer (2).

Reference:

(1) Methods For The Determination Of Metals in Environmental Samples, Environmental Monitoring Systems Laboratory, Office Of Research and Development, U.S. Environmental Protection Agency, Report No. EPA-600/4-91-010 (1991) Method 245.6.

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and

Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 6010A (1986).

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Lab Name: *Midwest Research Institute* Method Code: 007

Analytical Methodology for Arsenic and Selenium in Tissue

Summary: A one gram aliquot of freeze dried coarsely ground sample is weighed and digested using nitric acid and hydrogen peroxide (1). The contents are transferred to a 100 mL volumetric flask and diluted to volume using reagent grade water. The analyses are performed using Varian SpectrAA Graphite Furnace Zeeman Corrected single element atomic absorption spectrometer (2).

Reference:

(1) Methods For The Determination Of Metals In Environmental Samples, Environmental Monitoring Systems Laboratory, Office Of Research And Development, U.S. Environmental Protection Agency, Report No. EPA- 600/4-91-010 (1991) Method 200.3.

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 7000 (1986).

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Lab Name: *Midwest Research Institute* Method Code: 008

Analytical Methodology for Arsenic and Selenium in Water

Summary: 100 mL of water is combined with 10 mL of concentrated nitric acid and evaporated gently to approximately 10 mL (1). The contents are transferred to a 100 mL volumetric flask and diluted to volume using reagent grade water. The analyses are performed using a Varian SpectrAA Graphite Furnace Zeeman Corrected single element atomic absorption spectrometer (2).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and

Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 3020.

(2) Test Methods for Evaluating Solid Waste, Physical/chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 7000 (1986).

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Lab Name: *Midwest Research Institute* Method Code: 009

Analytical Methodology for Arsenic, Selenium and Cadmium in Soil

Summary: A one gram aliquot of the dried soil is transferred to a beaker and metals are extracted using nitric acid and hydrogen peroxide (1). The contents are transferred to a 100 mL volumetric flask and diluted to volume using reagent grade water. The analyses are performed using a Varian SpectrAA Graphite Furnance Zeeman Corrected single element absorption spectrometer (2).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 3050A (1986).

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 7000 (1986).

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Lab Name: *Midwest Research Institute* Method Code: 010

Homogenization and Moisture Determination of Tissue

Summary: Samples of tissue are homogenized by grinding the sample twice in a Hobart auger-fed grinder. A vessel is preweighed (vessel weight) and an aliquot of the homogenized tissue is weighed in the tared vessel (aliquot weight). The sample is then lyophilized and a vessel + lyophilized tissue weight is recorded. The tissue is ground to a coarse powder with a glass rod.

Percent Moisture = [1 - (vessel + lyophilized tissue - vessel weight/aliquot weight)] X 100

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Lab Name: *Midwest Research Institute* Method Code: 011

Homogenization and Moisture Determination of Sediments

Summary: A suitable vessel is preweighed (vessel weight) and an aliquot of sediment sample is added to the tared vessel (aliquot weight). The sample is allowed to dry for 24 hours in an oven at 105oC. After drying the sample is placed in a dessicator to cool. The vessel + dry sample weight is recorded.

The dried sample is transferred to a mortar and pestle for grinding. The ground sample is passed through a #10 sieve (2.0 mm mesh size).

Percent Moisture = [1 - (vessel + dry weight - vessel weight/aliquot weight)] X 100

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Lab Name: *Midwest Research Institute* Method Code: 012

Analytical Methodology for ICP Scan for Water using Microwave Digestion

Summary: A 45-mL of aliquot of water is digested in 5 mL of concentrated nitric acid in a heavy duty microwave vessel for 20 minutes using a CEM MDS 2100 microwave system (1). The contents are transferred to a 50-mL centrifuge tube for analysis. Analyses are performed using a Thermo Jarrell Ash Model 61E simultaneous inductively coupled plasma emission spectrometer (2).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Integrated Manual, Final Update III, June 1997, Method 3015 Revision 1.

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, SW-846 Method 6010A (1986).

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Lab Name: *Midwest Research Institute* Method Code: 013

Analytical Methodology for Selenium in Water by Hydride Atomic Absorption

Summary: A 50 mL aliquot of a well-mixed, acid-preserved sample is transferred to abeaker and digested with nitric and sulfuric acids. The selenium in a 10-mL aliquot of the digestate is then converted to a volatile hydride with hydrogen produced from a sodium borohydride reaction (1). Selenium absorption is measured using a GBC 902 atomic emission/absorption spectrometer with deuterium background correction. Selenium concentration is proportional to the absorbance of the signal (2).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Integrated Manual, Final Update III, June 1997, Method 7741-A, Revision 1.

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, SW-846 Method 7000 (1986).

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Lab Name: *Midwest Research Institute* Method Code: 014

Analytical Methodology for Selenium in Soil by Hydride Atomic Absorption

Summary: A one gram aliquot of the dried soil is transferred to a beaker and metalsare extracted using nitric acid and hydrogen peroxide (1). A 50 mL aliquot the extract is transferred to a beaker and digested with nitric and sulfuric acid. The selenium in a 10-mL aliquot of the digestate is then converted to a volatile hydride with hydrogen produced from a sodium borohydride reaction (2). Selenium absorption is measured using a GBC 902 atomic emission/absorption spectrometer with deuterium background correction. Selenium concentration is proportional to the absorbance of the signal (3).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 3050A (1986).

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Integrated Manual, Final Update III, June 1997, Method 7741A, Revision 1.

(3) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 7000 (1986).

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Lab Name: *Midwest Research Institute* Method Code: 015

Analytical Methodology for GFAA Analysis of Arsenic, Selenium and Cadmium in Water using Microwave Digestion

Summary: A 45-mL of aliquot of water is digested with 5 mL of concentrated nitric acid in a heavy duty microwave vessel for 20 minutes using a CEM MDS 2100 microwave system (1). The contents are transferred to a 50-mL centrifuge tube for analysis. Analyses are performed using a Varian SpectrAA Graphite Furnace Zeeman Corrected single element atomic absorption spectrometer (2).

Reference:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Integrated Manual, Final Update III, June 1997, Method 3015 Revision 1

(2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd Edition, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, SW-846 Method 7000 (1986)

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Lab Name: *Midwest Research Institute* Method Code: 016

Analytical Methodology for Total Cyanide in Sediment

Summary: Approximately 1-gram of wet sample is added to 50 mL of reagent water. Any deviation from the target weight of 1 gram is accounted for in the calculation. The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide concentration in the absorbing solution is then determined colorimetrically. The estimated detection limit for the method is 1 mg/Kg.

Reference:

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Final Update III, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 9010B (Revision 2, 1996).

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Lab Name: *Midwest Research Institute* Method Code: 017

Analytical Methodology for Total Organic Carbon in Sediment/Soil

Summary: Organic carbon is measured in sediment/soil using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO_2) by either catalytic combustion or wet chemical oxidation. Carbon is converted to CO_2 by means of a high temperature combustion furnace in the sediment/soil matrix. The amount of sample will vary dependent on the total organic carbon concentration.

References:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Final Update III, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington, DC, SW-846, Method 9060 (Revision 0, 1986).

(2) Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, EPA-600/4-79-020 (Revised March 1983), Method 415.1.

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Lab Name: *Midwest Research Institute* Method Code: 018

Analytical Methodology for Grain Size in Sediment/Soil

Summary: This method covers the quantitative determination of the distribution of particle sizes in sediment/soil. The distribution of particle sizes larger than 75 ?m is determined by sieving, while the distribution of particle sizes smaller than 75 ?m is determined by a sedimentation process, using a hydrometer.

Reference: American Society for Testing and Materials, ASTM D422-63 (Reapproved 1998).

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Lab Name: *Midwest Research Institute* Method Code: 019

Tissue Percent Moisture Determination (Oven-Drying Method)

Summary: A suitable vessel is preweighed (vessel weight) and an aliquot of tissue sample is added to the tared vessel (aliquot weight). The sample is allowed to dry for 24 hours in an oven at 105oC. After drying the sample is placed in a desiccator to cool. The vessel plus dry sample weight is recorded.

% Moisture = [1 - ((vessel plus dry weight - vessel weight)/aliquot weight)] X 100

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Lab Name: *Midwest Research Institute* Method Code: 020

Analytical Method for Mercury in Water by FIMS CVAAS

Summary: A 25-mL aliquot is digested using sulfuric acid, nitric acid, potassium permanganate and potassium persulfate for 2 hours at 95C in a heating block. Excess permanganate is reduced with sodium chloride/hydroxylamine hydrochloride and the sample is diluted to 50 mL. Mercury is reduced with stannous chloride to elemental mercury and measured by cold vapor atomic absorption spectroscopy (CVAAS) using a Perkin Elmer FIMS mercury analyzer, or equivalent. The nominal detection limit is 0.2 ug/L

References:

Method 245.1 Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry. Rev 3.0, EMMC Version. Methods for the Determination of Metals in Environmental Samples, Supplement 1. United States Environmental Protection Agency, Office of Research and Development, Washington DC 20460 . EPA/600/R-94/111.

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Lab Name: *Midwest Research Institute* Method Code: 021

Analytical Method for Mercury in Sediment by FIMS CVAAS

Summary: A 0.2-g aliquot of dry sediment is digested with aqua regia at 95*C for 2 minutes. Potassium permanganate is added and the sample is digested for an

additional 30 minutes. Excess permanganate is reduced with sodium chloride/hydroxylamine hydrochloride and the sample is diluted to 50 mL. Mercury is reduced with stannous chloride to elemental mercury and measured by cold vapor atomic absorption spectroscopy (CVAAS) using a Perkin Elmer FIMS mercury analyzer, or equivalent. The nominal detection limit is 0.05 ug/g on a dry weight basis.

References:

Method 245.5 Determination of Mercury in Sediments by Cold Vapor Atomic Absorption Spectrometry. Rev 2.3, April 1991. Methods for the Determination of Metals in Environmental Samples. United States Environmental Protection Agency, Office of Research and Development, Washington DC 20460 . EPA/600/4-91-010.

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Lab Name: *Midwest Research Institute* Method Code: 022

Analytical Method for Mercury in Tissue by FIMS CVAAS

Summary: A 0.2-g aliquot of dry tissue is solubilized with tetramethylammonium hydroxide (TMAH) at 60*C for 1 hour, followed by digestion with nitric acid in a heating block at 95*C for 1 hour and the sample is diluted to 50 mL. The digest is subjected to on-line oxidation with potassium permanganate followed by reduction with sodium borohydride to elemental mercury and measured by cold vapor atomic absorption spectroscopy (CVAAS) using a Perkin Elmer FIMS mercury analyzer, or equivalent. The nominal detection limit is 0.05 ug/g on a dry weight basis.

References:

Method 200.11 Determination of Metals in Fish Tissue by Inductively Coupled Plasma Atomic Emission Spectrometry. Rev 2.1, April 1991. Methods for the Determination of Metals in Environmental Samples. United States Environmental Protection Agency, Office of Research and Development, Washington DC 20460 . EPA/600/4-91-010.

Determination of Total Mercury in Biological Tissues by Flow Injection Cold Vapor Generation Atomic Absorption Spectrometry Following Tetramethylammonium hydroxide digestion. Tao,G., Willie,S.N., and Sturgeon,R.*, Analyst, June 1998, Vol.123 (1215-1218) *Institute for National Measurement Standards, National Research Council of Canada , Ottawa , Ontario , Canada , K1A 0R9

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Lab Name: *Midwest Research Institute* Method Code: 023

Analytical Method for Trace Elements in Water by ICPMS

Summary: A 50-mL aliquot of water is digested with nitric acid and hydrochloric acid in a heating block at 95 C for 3 hours and the sample is diluted to 50 mL. Determination is performed by inductively coupled plasma-mass spectrometry. Typical detection limits are in the 0.05-5 ug/L range.

References:

Method 200.8 Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry. Rev 5.4, EMMC Version. Methods for the Determination of Metals in Environmental Samples, Supplement 1. United States Environmental Protection Agency, Office of Research and Development, Washington DC 20460 . EPA/600/R-94/111.

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Lab Name: *Midwest Research Institute* Method Code: 024

Analytical Method for Trace Elements in Tissue by ICPMS

Summary: A 1-g aliquot of dry tissue is solubilized with tetramethylammonium hydroxide (TMAH) at 60*C for 1 hour, followed by digestion with nitric acid (and optionally hydrochloric acid) in a heating block at 95*C for 1 hour and the sample is diluted to 50 mL. Determination is performed by inductively coupled plasma-mass spectrometry on ten-fold dilutions of the digested solution. Typical detection limits are in the 0.01-5 ug/g range on a dry weight basis.

References:

Method 200.11 Determination of Metals in Fish Tissue by Inductively Coupled Plasma Atomic Emission Spectrometry. Rev 2.1, April 1991. Methods for the Determination of Metals in Environmental Samples. United States Environmental Protection Agency, Office of Research and Development, Washington DC 20460 . EPA/600/4-91-010.

Method 200.8 Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry. Rev 5.4, EMMC Version. Methods for the Determination of Metals in Environmental Samples, Supplement 1. United States Environmental Protection Agency, Office of Research and Development, Washington DC 20460 . EPA/600/R-94/111.

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Lab Name: *Midwest Research Institute* Method Code: 025

Analytical Method for Trace Elements in Sediment by ICPMS

Summary: A 1-g aliquot of dry sediment is digested with nitric acid and hydrochloric acid in a heating block at 95*C for 30 minutes and the sample is diluted to 50 mL. Determination is performed by inductively coupled plasma-mass spectrometry on ten-fold dilutions of the digested solution. Typical detection limits are in the 0.01-5 ug/g range on a dry weight basis.

References:

Method 200.8 Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry. Rev 5.4, EMMC Version. Methods for the Determination of Metals in Environmental Samples, Supplement 1. United States Environmental Protection Agency, Office of Research and Development, Washington DC 20460 . EPA/600/R-94/111.

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Lab Name: *Midwest Research Institute* Method Code: 026

Microwave Digestion and the Analysis of Trace Elements in Sediment

Summary: A 0.5-g aliquot of dry sediment is digested with nitric acid (and optionally hydrochloric acid) in a microwave oven at a temperature of 170-180*C for 20 minutes and the sample is diluted to 50 mL. Determination is performed by inductively coupled plasma-mass spectrometry on ten-fold dilutions of the digested solution or by cold vapor atomic absorption spectrometry for mercury. Typical detection limits are in the 0.01-5 ug/g range on a dry weight basis.

References:

Method 3051 Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils. Rev. 0, 1992. Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846, 3rd Edition. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency response, U.S. Government Printing Office, Washington , D.C. 20460

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Lab Name: *Midwest Research Institute* Method Code: 027

Procedure for Moisture Determination and Homogenization of Solid Samples

Summary: A suitable vessel is preweighed, an aliquot of wet sample is added and the vessel is reweighed. The sample is freeze dried and the vessel is weighed again. After subtraction of the vessel weight, wet and dry sample mass is used to determine moisture content. For homogenization, 2 Teflon balls (0.25" diameter) are added and the vessel is shaken in a paint shaker for 30 minutes. Aliquots of the dried, homogenized samples are used for digestion and analysis.

References:

Analytical Methods for Trace Elements in Fish Liver by Atomic Absorption Spectrophotometry. NOAA Technical Memorandum NOS ORCA 71. Sampling and Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984-1992 Volume III. Comprehensive Descriptions of Elemental Analytical Methods. National Oceanic and Atmospheric Administration, Silver Springs , Maryland , July 1993.

GERG Trace Element Quantitation Techniques. NOAA Technical Memorandum NOS ORCA 71. Sampling and Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984-1992 Volume III. Comprehensive Descriptions of Elemental Analytical Methods. National Oceanic and Atmospheric Administration, Silver Springs , Maryland , July 1993.

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Mississippi State Chemical Laboratory (MSCL) Laboratory Methods

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002	Analysis For Organochlorine Pesticides and PCBs In Soil and Sediment
003	Analysis For Aliphatic and Polynuclear Aromatic Hydrocarbons In Animal and Plant Tissue
004	Analysis For Organochlorine Pesticides and PCBs, Aliphatic and Polynuclear Aromatic Hydrocarbons In Soil and Sediment
005	Analysis For Organochlorine Pesticides and PCBs, Polynuclear Nuclear Aliphatic and Hydrocarbons In Water
006	Analysis For Chlorinated Hydrocarbon Pesticides And Related Compounds - Micro Method
007	Analysis for Organochlorine Pesticides, N/P Pesticides, Aliphatic and Polynuclear Aromatic Hydrocarbons, and Chlorophenoxy Acid Herbicides in Water
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010	Analysis For Oil and Grease In Soil and Sediment
011	Analysis for Organochlorine Pesticides and PCBs in Blood Serum, Plasma or Whole Blood
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016	Analysis for AHH Active PCB Congeners in Animal Tissue

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022 Analysis For Selected Organophosphate and Synthetic Pyrethroid Pesticides In Animal and Plant Tissue.

023 Analysis For Selected Organophosphate and Synthetic Pyrethroid Pesticides and Triazine Herbicides in Soil and Sediment.

025 Analysis For Selected Organophosphate and Synthetic Pyrethroid Pesticides and Triazine Herbicides in Water

026 Analysis For PCP, Carbaryl, 4-nonylphenol, and Permethrin by GC/MS

027 Method for Substituted Urea Herbicides in Water

028 Analysis For AHH active PCB Congeners In Soil and Sediment

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030 DETERMINATION OF STARLICIDE IN WATER

031 Accelerated Solvent Extraction (ASE) and Analysis of Synthetic Pyrethroid Pesticides in Filter Disks.

032 Accelerated Solvent Extraction (ASE) and Analysis of Synthetic Pyrethroid Pesticides in Plant Tissue.

033 Accelerated Solvent Extraction (ASE) and Analysis of Disyston, Disyston Metabolites, and Oxygen Analoges in Filter Disks.

034 Analysis for Chlorophenoxy Acid Herbicides in Liver

035 Analysis for Organochlorine Pesticides and PCBs in Soil and Sediment by ASE.

036 Analysis of Soil and Vegetation for Glyphosate

038 Dioxins

039 Analysis For Selected Organophosphates in Soil and Sediment by ASE and GPC.

040 Analysis for PAHs in Soil/Sediment and plant tissue by ASE and GPC.

041 Analysis of Water -- Special for E. Snyder-Conn

042 Analysis for Organochlorine Pesticides, N/P Pesticides, Aliphatic and

	Polynuclear Aromatic Hydrocarbons, and Chlorophenoxy Acid Herbicides in Water
043	Analysis For Triazine Herbicides in Soil, Sediment and Tissue.
044	Analysis of Synthetic Pyrethroid Pesticides in Bat Guano.
045	Analysis for PCB Congeners In Animal Tissue or Soil/Sediment
046	Dioxin and Furan in Tissue
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050	Analysis For Organochlorines Pesticides and PCBs in Animal Tissue using soxhlet/GPC.
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057	Analysis for TPH (total petroleum hydrocarbon) in Soil and Sediment
059	Analysis for Paraquat and Diquat in Water
060	Analysis for Aliphatic and Polynuclear Aromatic Hydrocarbons in Animal Tissue
061	Analysis for Atrazine metabolites in Water
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064	Analysis for Organophosphate Pesticides and Triazine Herbicides in Animal Tissue using ASE/GPC
065	Analysis for Pirate and Metabolites Tissue using ASE/GPC.
066	Analysis for Organochlorine Pesticides and PCBs in Animal Tissue using ASE/GPC.
067	Analysis for Organochlorine Pesticides and PCBs, Aliphatic and Polynuclear Aromatic Hydrocarbons in Soil and Sediment

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069 Imadacloprid in Tissue

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071 Anticoagulant Rodenticides in Blood and Liver

072 Analysis of Water for Imazapic

073 Analysis for TPH (total petroleum hydrocarbon) in Water

099 % Moisture

Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 001

Analysis For Organochlorine Pesticides and PCBs In Animal and Plant Tissue

Ten gram tissue samples are thoroughly mixed with anhydrous sodium sulfate and soxhlet extracted with hexane for seven hours. The extract is concentrated by rotary evaporation; transferred to a tared test tube, and further concentrated to dryness for lipid determination. The weighed lipid sample is dissolved in petroleum ether and extracted four times with acetonitrile saturated with petroleum ether. Residues are partitioned into petroleum ether which is washed, concentrated, and transferred to a glass chromatographic column containing 20 grams of Florisil. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction II). Fraction II is concentrated to appropriate volume for quantification of residues by packed or capillary column electron capture gas chromatography. Fraction I is concentrated and transferred to a Silicic acid chromatographic column for additional cleanup required for separation of PCBs from other organochlorines. Three fractions are eluted from the silicic acid column. Each is concentrated to appropriate volume for quantification of residues by packed or megabore column, electron capture gas chromatography. PCBs are found in Fraction II.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 002

Analysis For Organochlorine Pesticides and PCBs In Soil and Sediment

Twenty-five gram soil or sediment samples are extracted with acetone followed by hexane, by allowing to soak one hour in each with intermittent shaking. The combined extracts are centrifuged and decanted into a separatory funnel containing sufficient water to facilitate partitioning of residues into hexane portion. The hexane is washed twice with water and concentrated to appropriate volume for transfer to a 1.6 gram Florisil mini-column topped with 1.6 grams sodium sulfate. Residues are eluted from the column in two elution fractions. Fraction I consists of 12 milliliters hexane followed by 12 milliliters of 1% methanol in hexane, and Fraction II consists of an additional 24 milliliters of 1% methanol in hexane. If additional cleanup is required to separate PCBs from other organochlorines in Fraction I, further chromatography on a Silicic acid column is performed. Quantification of residues in the two Florisil fractions and three Silicic acid fractions is by packed or megabore column, electron capture gas chromatography.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 003

Analysis For Aliphatic and Polynuclear Aromatic Hydrocarbons In Animal and Plant Tissue

A sample of appropriate size (i.e. 15 grams animal or plant tissue, 2 grams adipose, 5 grams eggs) is digested in 6N aqueous potassium hydroxide for 24 hours at 35 degrees C. Cool digestate thoroughly in an ice bath and carefully neutralize with glacial acetic acid. Extract the neutralized reaction mixture three times with methylene chloride; concentrate the combined extracts to near dryness and reconstitute in petroleum ether for transfer to a 20 gram 1% deactivated silica gel column, topped with 5 grams neutral alumina. Aliphatic and polynuclear aromatic hydrocarbon residues are separated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) followed by elution of aromatics using first, 100ml 40% methylene chloride/60% petroleum ether, then 50 ml methylene chloride (Combined eluates, Fraction II). If needed, Fraction I containing aliphatics is subjected to additional cleanup by concentration and transfer to a deactivated (2% water) Florisil column. Aliphatic residues are eluted from the Florisil column using 200 ml 6% diethyl ether/94% petroleum ether. The eluate is concentrated to appropriate volume for quantification by capillary column, flame ionization gas chromatography. The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride, and subjected to gel permeation chromatography (GPC) cleanup prior to quantification by capillary, flame ionization gas chromatography and fluorescence HPLC.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 004

Analysis For Organochlorine Pesticides and PCBs, Aliphatic and Polynuclear Aromatic Hydrocarbons In Soil and Sediment

Twenty gram soil or sediment samples are extracted with acetone, followed by petroleum ether, by allowing to soak one hour in each with intermittent shaking. A final acetone/petroleum ether extraction is done, and the extracts are combined, centrifuged, and transferred to a separatory funnel containing sufficient water to facilitate partitioning of residues into petroleum ether portion. The petroleum ether is washed twice with water and concentrated by Kuderna-Danish to appropriate volume. An aliquot of the concentrated extract for pesticide determination is transfered to a 1.6 gram Florisil mini-column topped with 1.6 grams sodium sulfate. Residues are eluted from the column in two elution fractions. Fraction I consists of 12 milliliters hexane followed by 12 milliliters of 1% methanol in hexane, and Fraction II consists of an additional 24 milliliters of 1% methanol in hexane. If additional cleanup is required to separate PCBs from other organochlorines in Fraction I,

further chromatography on a Silicic acid column is performed. Quantification of residues in the two Florisil fractions and three Silicic acid fractions is by packed or megabore column, electron capture gas chromatography.

A second aliquot of the concentrated extract for hydrocarbon determination is transfered to a 20 gram 1% deactivated silica gel column, topped with five grams neutral alumina. Aliphatic and polynuclear aromatic hydrocarbon residues are fractionated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) followed by elution of aromatics using first, 100 ml 40% methylene chloride/60%petroleum ether, then 50 ml methylene chloride (Combined eluates, Fraction II). If needed, Fraction I containing aliphatics is subjected to additional cleanup by concentration and transfer to a deactivated (2% water) Florisil column. Aliphatic residues are eluted from the Florisil column using 200 ml 6% diethyl ether/94% petroleum ether. The eluate is concentrated to appropriate volume for quantification by capillary column, flame ionization gas chromatography. The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride, and subjected to gel permeation chromatographic (GPC) cleanup prior to quantification by capillary, flame ionization gas chromatography and fluorescence HPLC.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 005

Analysis For Organochlorine Pesticides and PCBs, Polynuclear Nuclear Aliphatic and Hydrocarbons In Water

A 500 milliliter water sample is extracted four times by shaking with 50 milliliter portions of methylene chloride. The four extracts are combined and concentrated by Kuderna- Danish to near dryness, then reconstituted in 5 milliliters petroleum ether. An appropriate aliquot is removed for organochlorine and PCB analysis and transfered to a 20 gram Florisil column. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction II). Fraction II is concentrated to appropriate volume for quantification of residues by packed or capillary column electron capture gas chromotography. Fraction I is concentrated and transferred to a silicic acid chromatographic column for additional cleanup required for separation of PCBs from other organochlorines. Three fractions are eluted from the silicic acid column. Each is concentrated to appropriate volume for quantification of residues by packed or megabore column, electron capture gas chromotography. PCBs are found in Fraction II. The remainder of the petroleum ether from the above methylene chloride extraction is transferred to a 20 gram 1% deactivated silica gel column, topped with 5 grams neutral alumina. Aliphatic and polynuclear aromatic hydrocarbon residues are separated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) folllowed by elution of aromatics using first, 100 ml 40% methylene

chloride/60% petroleum ether then 50 ml methylene chloride (combined eluates, Fraction II). If needed, Fraction I containing aliphatics is subjected to additional cleanup by concentration and transfer to a deactivated (2% water) Florisil column. Aliphatic residues are eluted from the Florisil column using 200 ml 6% diethyl ether/94% petroleum ether. The eluate is concentrated to appropriate volume for quantification by capillary column, flame ionization gas chromotography. The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride, and subjected to gel permeation chromatographic (GPC) cleanup prior to quantification by capillary, flame ionization gas chromatography and fluorescence HPLC.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 006

Analysis For Chlorinated Hydrocarbon Pesticides And Related Compounds - Micro Method

This method is necessary when sample size is limited (below 4 g, approximately) and in case of organ tissue as substrate and is a modified version of the method described in "Manual of Analytical Methods for the Analysis of Pesticides in Humans and Enviromental Samples", EPA-600/8-80-038, June 1980, Section 5, A (2). It is suitable for adipose, kidney, liver, muscle, brain, and other tissues:

1. Weigh 0.5 g or less of well-mixed tissue into a size 22 Duall tissue grinder.

2. Extract tissue by grinding three times with acetonitrile; the first time being with 4 ml followed by two 2.5 ml portions.

3. Remove the pestle after each grinding and centrifuge, decanting the extract into a 50 ml glass stoppered graduated mixing cylinder.

4. Combine all extracts and record the total volume of the three extracts.

5. Add a volume of PRQ water equivalent to 3.3 times the extract volume. Then add 2 ml saturated NaCl solution.

6. Extract the aqueous acetonitrile mixture with 5 ml hexane by vigorous shaking for 1 minute.

7. Allow layers to separate, and remove the hexane layer with a Pasteur pipet into a 15 ml screw-capped culture tube.

8. Re-extract twice with 2 ml hexane each time, combining the extracts into the culture tube.

9. Concentrate the combined hexane extracts under nitrogen to approximately 0.5 ml volume.

10. Clean-up on a florisil mini-column as described in Method 2, Steps 8, 9, 10. and 11.

Note: For brain tissue additional treatment is necessary before column clean-up:

11. Proceed through Steps 1-9 above, add 0.3 ml acetic anhydride and 0.3 ml pyridine, cap tightly and incubate for 30 minutes in a water bath at 60-65 degrees C.

12. Add 8 ml PRQ water and 1 ml saturated NaCl and extract three times with 2 ml hexane, combining the extracts into a clean tube.

13. Concentrate the combined extracts under nitrogen to about 0.3 ml and proceed with florisil mini-column clean-up. (Step 10)

Note: The following changes in sample handling, particularly column clean-up, should be observed for Kepone analysis:

14. Maintain the integrity of the analyte in sample extracts by insuring that the samples are not allowed to reach dryness during concentration steps. Kepone easily adheres to glass, but the use of polar solvents such as methanol and acetonitrile within the analysis will provide better recoveries of this analyte.

15. Modifications to florisil mini-column clean-up are as follows:

* Following addition of sample to the column, apply a 1ml rinse of 1% methanol in hexane to the sample tube. This rinse should be added after the first phase of of the first fraction (12mls hexane) and will insure removal of trace quantities of kepone adhered to glass. Decrease the total volume of the second phase of the first fraction (12mls 1% methanol/hexane) to 11mls.

* Modify the total volume of the second fraction from 24mls to 36mls 1% methanol/hexane. This fraction contains Kepone.

* Concentrate column fractions on N-EVAP and transfer with 1% methanol/hexane to calibrated test tubes. Adjust sample volume to calibrated level and proceed to determination by gas chromatograph.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 007

Analysis for Organochlorine Pesticides, N/P Pesticides, Aliphatic and Polynuclear Aromatic Hydrocarbons, and Chlorophenoxy Acid Herbicides in Water

This procedure was developed to allow for sample extraction to be performed in the same container in which the sample is collected, thus eliminating the need to transfer the sample to other glassware and eliminating the possibility of incomplete extraction of any analyte that may adhere to glass surfaces. Five pint acid bottles, calibrated at two liters, are convenient for sample collection/extraction.

This procedure may be used with any volume of sample with the appropriate volume reduction of solvents, reagents, and glassware. However, if transfer from the collection container is necessary, this container should be rinsed with the extraction solvent to insure complete recovery of all analytes.

1. Reduce sample volume to 2 l or record volume if < 2 l.

2. Add PRQ 6N KOH to sample, seal and shake vigorously 30s. Test pH with glass rod (pH = 8). Adjust to proper pH by dropwise addition of 6N KOH. Allow to stand one hour at pH 8.

3. Add 100 ml CH_2Cl_2 and shake 2 min. with periodic venting. Remove CH_2Cl_2 into 500 ml French Square bottle using glass suction device attached to aspirator system. Reverse suction device to return water in CH_2Cl_2 layer to the bottle. Repeat 2 X 100 ml CH_2Cl_2. After final step, assure return of all water to bottle using Pasteur pipet. This combined extract contains organochlorine, nitrogen- /phophorous-containing pesticides, aliphatic hydrocarbons and polynuclear aromatic hydrocarbons.

4. Acidify water with PRQ 12N H_2SO_4. Shake 30s and test pH with glass rod. Adjust pH to ó 2 with dropwise addition of acid if necessary.

5. Extract water with 200ml Ethyl ether (EtoEt) by shaking two minutes. Remove either layer with suction device to 1000ml French Square. Return excess water to bottle. Repeat 2 x 100ml EtoEt. Extract with a final aliquot of 100ml petroleum ether. Remove all water from French Square with Pasteur pipet. This combined extract contains chlorophenoxy acid herbicides.

6. Concentrate acid and neutral extracts with Kuderna-Danish evaporators and reduce volume to adequate size for column clean-up.

7. Column Clean-Up:

* NEUTRAL FRACTION (N/P and organochlorine pesticides, Aliphatic and Polynuclear aromatic hydrocarbons) - adjust sample extract to exact volumn and remove an appropriate aliquot for column clean-up techniques specific to analyte; for pesticides use Mini-florisil (described in Method 2), for hydrocarbons use 1 % deactivated silica gel (described in Method 4).

* ACID FRACTION (Chlorophenoxy acid herbicides) - Derivitization: Reduce sample volume to approximately 0.5ml and ethylate using diazoethane (15 min.). Exchange to hexane (N-EVAP) and reduce volume to 0.2ml.

Column clean-up: Place 2.0g of 1% deactivated silica gel in a 7mm i.d. chromatography column (#22 Kontes). Top with 1cm Na2SO4 and prewetcolumn with 10ml hexane. Collect sample eluents in three fractions as follows:

Fraction A: add sample and rinse container with two 0.5ml washes of 20% benzene in hexane. Elute with 9ml of the same solution.(Conta ins PCP.)

Fraction B: add 10ml 40% benzene in hexane. Add 10ml 60% benzene in hexane.(Contains Dalapon, PNP, Silvex, Dinoseb, portion of Dicamba.) Fraction C: add 10ml 80% benzene in hexane. Add 10ml 100% benzene.(Contains remaining Dicamba, Dichlorprop, 2,4-D, 2,4,5-T, 2,4-DB, Bentazon, Blazer.)

[Reference for column clean-up for acid herbicides:Shafik, T. A.,H. C. Sullivan, H. R. Enos, 1973." Multiresidue Procedure for Halo- and Nitrophenols. Measurement of Exposure to Biodegradable Pesticides Yielding These Compounds as Metabolites." J. Agr. Food Chem. 21:295-298.]

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 008

Analysis for Organochlorine Pesticides, Aliphatic and Polynuclear Aromatic Hydrocarbons, and Chlorophenoxy Acid Herbicides in Soil and Sediment

1. Weigh 20 g soil into a PRQ centrifuge bottle. (Add 10 ml PRQ H2O to dry samples) Adjust pH to ó 2 with PRQ 12N sulfuric acid (about 1 ml). Add 50 ml acetone and shake 6 times over a one and one-half hour period (about every 15 mins.). Add 50 ml of a 1:1 petroleum ether/ ethyl ether mixture and repeat shaking. Centrifuge and decant liquid into a 500 ml separatory funnel containing 200 ml PRQ water. Re- extract soil by shaking one minute with 50 ml 1:1 PE:EtoEt (may need to add 10 ml H2O & adjust to pH < 2), then centrifuge and decant liquid into sep. funnel.

2. Using PRQ 6N KOH (5 ml), adjust contents of sep. funnel to pH ò 12. Shake vigorously 2 min, then allow to stand 30 min. with intermittent shaking. Drain water layer and reserve ether layer. Re-extract H2O layer with 100 ml 1:1 PE:EtoEt. Cap and reserve combined ether extracts.(This contains organochlorine pesticides, aliphatic and polynuclear aromatic hydrocarbons.)

3. Adjust aqueous layer to pH ó 2 using 3 ml of PRQ 12 N sulfuric acid and extract with 100 ml 1:1 PE:EtoEt. Reserve this extract and re-extract H2O with 100 ml 1:1 PE:EtoEt. Combine extracts (these extracts contain chlorophenoxy acid herbicides).

4. Concentrate acid and basic extracts with Kuderna-Danish evaporators and reduce volume to adequate size for column clean-up.

5. Column Clean-up:

* BASIC FRACTION (N/P and Organochlorine pesticides, Aliphatic and Polynuclear aromatic hydrocarbons) - adjust sample extract to exact volume and remove an appropriate aliquot for column clean-up techniques specific to analyte; for pesticides use Mini-florisil (described in Method 2), for hydrocarbons use 1% deactivated silica gel (described in Method 4).

* ACID FRACTION (Chlorophenoxy acid herbicides) - Derivitization: Reduce sample volume to approximately 0.5ml and ethylate using diazoethane (15 min.). Exchange to hexane (N-EVAP) and reduce volume to 0.2ml. Column clean-up: Place 2.0g of 1% deactivated silica gel in a 7mm i.d. chromatography column (#22 Kontes). Top with 1cm Na2SO4 and prewet column with 10ml hexane. Collect sample eluents in three fractions as follows:

Fraction A: add sample and rinse container with two 0.5ml washes of 20% benzene in hexane. Elute with 9ml of the same solution.(Contains PCP.)

Fraction B: add 10ml 40% benzene in hexane. Add 10ml 60% benzene in hexane.(Contains Dalapon, PNP, Silvex, Dinoseb, portion of Dicamba.)

Fraction C: add 10ml 80% benzene in hexane. Add 10ml 100% benzene.(Contains remaining Dicamba, Dichlorprop, 2,4-D, 2,4,5-T, 2,4-DB, Bentazon, Blazer.)

[Reference for column clean-up for acid herbicides: Shafik, T. A.,H. C. Sullivan, H. R. Enos, 1973." Multiresidue Procedure for Halo- and Nitrophenols. Measurement of Exposure to Biodegradable Pesticides Yielding These Compounds as Metabolites." J. Agr. Food Chem. 21:295-298.]

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 009

Determination of Organochlorine Pesticides, Arochlors, Aliphatic Hydrocarbons and Polynuclear Aromatic Hydrocarbons in Hexane Extracts From Passive In-Situ Samplers

1. Measure and record the total extract volume; transfer to a 1 l Kjeldahl- shaped flask.

2. Concentrate the hexane extract to about 1 ml (45øC bath temp.).

3. Transfer the extract with petroleum ether through a 9" Pasteur pipet containing about 1 inch of anhydrous Na2SO4 into a 16 mm X 150 mm screw- capped culture tube.

4. Carefully concentrate to about 0.5 ml on the N-Evap.

5. Transfer to an appropriate size calibrated screw-capped tube with hexane and adjust volume to 2.0 ml (or other appropriate volume).

6. Determine residues using appropriate glc techniques.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 010

Analysis For Oil and Grease In Soil and Sediment

Fifty gram soil or sediment samples are extracted with acetone, followed by petroleum ether, by allowing to soak one hour in each with intermittent shaking. The samples are centrifuged, and the supernatant is decanted into a separatory funnel containing sufficient water to facilitate partitioning of residues into petroleum ether portion. Two further acetone/petroleum ether extractions are done, and the extracts are sequentially centrifuged, and transferred to the separatory funnel. The aqueous portion is extracted with petroleum ether and the combined ether extracts are washed twice with water and concentrated by Kuderna-Danish to appropriate volume for transfer. The sample is transferred with petroleum ether rinsing through a bed of sodium sulfate into a tared glass tube. Solvent is removed under nitrogen (N-EVAP), and tube weights are allowed to equilibrate prior to the determination of oil and grease values.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 011

Analysis for Organochlorine Pesticides and PCBs in Blood Serum, Plasma or Whole Blood

Reference: Manual of Analytical Methods for the Analysis of Pesticides in Humans and Enviromental Samples, Section 5, A, (3), (a)

Modified as follows:

A 2-ml aliquot of serum, Plasma or whole blood, is extracted with 6 ml of hexane in a 16 X 125 mm screw-capped(teflon liner) culture tube. The extraction is conducted for 2 hours at 50 RPM on a rotating mixer. Alternatively, the extraction may be accomplished by vigorous mixing on a vortex mixer for 2 minutes. The formation of emulsion is unlikely, but if it should occur, centrifugation may be used to effect separation of the layers. In most instances, quantification by ec/gc can be performed on the hexane extract without further treatment; otherwise a suitable aliquot of the hexane layer may be quantitatively transferred to an evaporative concentrator tube to which is affixed a modified micro-Snyder column. The extract is concentrated in a water or steam bath, and the final volume is adjusted to correspond to the expected concentration of the pesticide residue. A suitable aliquot is analyzed by electron capture gas chromatography.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 012

Analysis for Amitrol-T in Soil and Sediment

1. Weigh 20 g well-mixed, homogeneous soil/sediment into a 200 ml screw-capped centrifuge bottle. Add 50 ml 80 % ethanol/H2O and allow to soak 1 1/2 hour with frequent intermittent shaking.

2. Centrifuge appropriately and transfer the aqueous extract to a 250ml separatory funnel.

3. Repeat the extraction with 80 % ethanol/H2O twice more as before, reducing the extraction time to 30 minutes. Combine the aqueous extracts in the separatory funnel.

4. Wash the aqueous extract with 20 ml hexane, discarding the hexane.

5. Repeat hexane wash of aqueous extract twice more, discarding hexane each time.

6. Transfer the extract to a 300 ml, 24/40 pear-shaped flask using PRQ H2O, and concentrate to ca 2 ml on a rotary evaporator with the waterbath set at 60 degrees C.

7. Transfer the sample with PRQ H2O to a screw-capped, calibrated culture tube and adjust volume to the calibration mark (10 ml normally used) with H2O. Quantitate by capillary gas chromatography (TSD optimized for N2 detection).

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 013

Analysis For Oil and Grease In Water

Sample volume and pH are determined, and an 800 ml aliquot is extracted with 75 ml methylene chloride three times. The combined ether extracts are washed twice with water and concentrated by Kuderna-Danish to appropriate volume for transfer. The sample is transferred with petroleum ether rinsing through a bed of sodium sulfate into a tared glass tube. Solvent is removed under nitrogen (N-EVAP), and tube weights are allowed to equilibrate prior to the determination of oil and grease values.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 014

Elution Profiles for Florisil, Silica Gel and Silicic Acid Column Separations

A. Florisil Column:

1. Fraction I - (6% ethyl ether containing 2% ethanol, 94% petroleum ether) HCB, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordane, heptachlor epoxide, gamma-chlordane, trans-nonachlor, toxaphene, PCB's, o,p'-DDE, alpha-Chlordane, p,p'-DDE, p,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, mirex, dicofol, endosulfan I (Split with FII).

2. Fraction II - (15% ethyl ether containing 2% ethanol, 85% petroleum ether) dieldrin, endrin, dacthal, endosulfan I (split with FI), endosulfan II (split with FIII), endosulfan sulfate (split with FIII).

3. Fraction III - (50% ethyl ether containing 2% ethanol, 50% petroleum ether) endosulfan II (split with FII), endosulfan sulfate (split with FII), malathion.

B. Florisil Mini-Column:

1. Fraction I - (12 ml hexane followed by 12 ml 1% methanol in hexane) HCB, gamma-BHC (25%), alpha-BHC (splits with FII), trans-nonachlor, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD (splits with FII), o,p'-DDT, p,p'-DDT, mirex, cis-nonachlor, cis-chlordane, trans-chlordane, PCB's, Photomirex and derivatives.

2. Fraction II - (24 ml 1% methanol in hexane) gamma BHC (75%), beta-BHC, alpha-BHC (splits with FI), delta-BHC, oxychlordane, heptachlor epoxide, toxaphene, dicofol, dacthal, endosulfan I, endosulfan II, endosulfan sulfate, octachlorostyrene, Kepone (with additional 12mls 1% methanol in hexane).

C. Silica Gel:

1. SG Fraction I - (100 ml petroleum ether) n-dodecane, n-tridecane, n-tetradecane, ocylcyclohexane, n-pentadecane, nonycyclohexane, n-hexadecane, n-heptadecane, pristane, n-octadecane, phytane, n-nonadecane, n-eicosane.

2. SG Fraction II - (100 ml 40% methylene chloride in petroleum ether followed by 50 ml methylene chloride) napthalene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, 1,2-benzanthracene, chrysene, benzo [b] fluoranthene, benzo [k] fluoranthene, benzo [e] pyrene, benzo [a] pyrene, 1,2:5,6-dibenzanthracene, benzo [g,h,i] perylene.

D. Silicic Acid:

1. SA Fraction I - (20 ml petroleum ether) HCB, mirex

2. SA Fraction II - (100ml petroleum ether) PCB's, p,p'-DDE (splits with SA III)

3. SA Fraction III - (20 ml mixed solvent: 1% acetonitrile, 80% methylene chloride, 19% hexane) alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordane, heptachlor epoxide, gamma-chlordane, trans-chlordane, toxaphene, o,p'-DDE, alpha-chlordane, p,p'-DDE (splits with SAII), o,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, dicofol.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 015

Analysis for Purgeable Organic Compounds by Capillary Column GC/MS.

This method is applicable to all types of samples (Water, Soil/Sediment, Animal Tissues, and Plant Tissues). GC/MS CONDITIONS

1) The MS must pass spectrum criteria for 4-bromofluorobenzene as stated in EPA methods 524.2 and 8260.

2) Generate a five point standard curve that passes the EPA criteria.

WATER

Measure ten (10) ml of water in a glass syringe. Add four-hundred (400) ng of internal standards (dibromotetrafluoroethane, d-6 benzene, and d-5 chlorobenzene) and four- hundred (400) ng of surrogates (bromofluorethane, bromofluoromethane, bromofluorobenzene). Load the purge and trap unit (Tekmar LSC2000 & ALS2016) with the sample and purge with He using the needle sparger. Trap the compounds on a Vocarb 3000 Trap (Supelco) and determine the compounds using a Varian Saturn ion trap with a Sixty (60) m 0.32 RTX-Volatile capillary column.

SOIL/SEDIMENT

Weigh up to five (5) grams soil into test tube. Add internal standards and surrogates to ten (10) ml reagent water. Add this to the soil and purge with needle sparger. Follow the water procedure from here.

TISSUES

Weigh up to two (2) grams of frozen tissue into the test tube. Follow the above procedure for Soil/Sediment.

REFERENCES:

Method 524.2 Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry Rev.3.0, 1989, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

Method 8260 Volatile organic compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary column technique, 1990. Test Methods for Evaluating Solid Wastes Volume 1B: Laboratory Manual Physical/Chemical Methods, U.S. Environmental Protection Agency.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 016

Analysis for AHH Active PCB Congeners in Animal Tissue

Five gram tissue samples are thoroughly mixed with anhydrous sodium sulfate and soxhlet extracted with hexane for seven hours. The extract is concentrated by rotary evaporation; transferred to a tared test tube, and further concentrated to dryness for lipid determination. The weighed lipid sample is dissolved in petroleum ether and

extracted four times with acetonitrile saturated with petroleum ether. Residues are partitioned into petroleum ether which is washed, concentrated, and transferred to a glass hromatographic column containing 20 grams of Florisil. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I). Fraction I is concentrated and transferred to a Silicic acid chromatographic column for additional cleanup required for separation of PCBs from other organochlorines. Three fractions are eluted from the silicic acid column. Fraction I is 20 ml of Petroleum Ether. Fraction II is 150 ml of Petroleum ether and contains the PCB's. Fraction III contains most of the other organochlorine pesticides.

Fraction II is concentrated to one ml and fractionated on a carbon column. This column consists of 2 grams of 1:20 AX-21 Carbon on activated silica gel. (AX-21 Activated Carbon, Anderson Development Co., Adrian, MI, vacuum washed with 200 ml each water, methanol, toluene, acetone, methylene chloride, 2X pet ether) (Silica gel EM Science Silica gel 60-230 mesh activated @ 130 C more than 10 days) Fraction A consists of 100 ml Pet Ether followed by 50 ml 10% methylene chloride in Pet Ether. Fraction B is 100 ml of 50% methylene chloride in Pet Ether. Fraction C is 50 ml toluene. Fraction B contains PCB# 105, 114, 118, 156. Fraction C contains PCB# 077, 126, 169. The PCB fractions were chromatographed on a Varian 3400 GC using a 60m 0.25 DB-5 capillary column. Confirmation was done on a 30m 0.53 DB-608 megabore column.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 017

Analysis For Carbamates In Sediment

Analysis of samples for carbamates was performed on the waters Carbamates analysis system equipped with a WISP model 712 Autosampler. Sediment samples (20g) were extracted with 20ml methanol by sonication for 30 minutes, then allowed to sit one hour. The samples were filtered through 0.45u nylon filters. A dilution in water (1:10) was made, and 500ul injected. The separation of the analytes was at room temperature, and the column used was a 3.9mm x 150mm waters nova-par C10 (4u). The solvent gradient used was as follows:

TIME	FLOW	%WATER	%METHANOL	%ACETONITRILE	CURVE
INIT	1	88	12	0	*
4	1	88	12	0	1
4.1	1	68	16	16	5
16.1	1	30	35	35	10
25	1	88	12	0	9

The post-column derivatization was done at 80.0 C. The samples were hydrolyzed with 0.05m NaOH, then reacted with o-phthaldialdehyde and 2- mercaptoethenol in 0.05m sodium borate decahydrate to yield a highly fluorescent isoindole product. The flow from the post-column pumps was 0.5ml/min. The detector was a waters 470 scanning fluorescence detector at 338nm and at 400nm.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 018

Analysis For Carbamates In Water

Analysis of samples for carbamates was performed on the waters Carbamates analysis system equipped with a WISP model 712 Autosampler. All samples were filtered through 0.45u nylon filters. Water samples (500ul) were directly injected.

The separation of the analytes was at room temperature, and the column used was a 3.9mm x 150mm waters nova-par C (4u). The solvent gradient used was as follows:

TIME	FLOW	%WATER	%METHANOL	%ACETONITRILE	CURVE
INIT	1	88	12	0	*
4	1	88	12	0	1
4.1	1	68	16	16	5
16.1	1	30	35	35	10
25	1	88	12	0	9

The post-column derivatization was done at 80.0 C. The samples were hydrolyzed with 0.05 m NaOH, then reacted with o-phthaldialdehyde and 2-mercaptoethenol in 0.05m sodium borate decahydrate to yield a highly fluorescent isoindole product. The flow from the post-column pumps was 0.5ml/min. The detector was a waters 470 scanning fluorescence detector at 338nm and at 400 nm.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 019

Grain Size

Disperse sample of soil or sediment to pass 2 mm sieve and place a weighed 40 gram in a 600 ml beaker. Take additional 30 gram sample for moisture determination so that air-dried weight may be corrected to oven-dried weight. Add 50 ml 10% "Calgon" solution (sodium meta- phosphate with sufficient sodium carbonate to give a pH of approximately 8.3 in a 10% solution) to 40 gram sample and allow to soak for at least 10 minutes. After soaking, quantitatively transfer sample with distilled water to Waring blender cup so that cup is approximately half full. Blend for four minutes and transfer suspension to sedimentation cylinder adjusting liquid level to 1000 ml mark with distilled water. Place cylinder in constant temperature water bath (approximately 38oC). Prepare sample "blank" by adding 50 ml 10% "Calgon" solution to second sedimentation cylinder and add distilled water to the 1000 ml mark. When the suspension reaches water bath temperature, the mixture is thoroughly stirred prior initiation of sedimentation. The time that stirring ceases is noted as the zero settling time. At the end of eight hours, lower the hydrometer (ASTM 152 H) gently into the suspension and read the scale at the end of the meniscus. Record the time of hydrometer reading, the hydrometer reading, and water bath temperature. After thorough mixing, record the hydrometer reading in the sample "blank" solution of water and "Calgon". After hydrometer readings are recorded, pour the suspension onto a 270 mesh, 53 micron sieve and wash all silt and clay out with the water. Transfer sample material remaining on the sieve into an evaporating dish; place in 110oC oven and allow to dry for 24 hours. After cooling, weigh the sample to determine the weight of oven-dry sand left on the sieve. Using moisture data determined, correct sample air-dry weight to oven-dry weight. Calculate the concentration of suspension in grams per liter from the following equation:

C = R - R< where:

- C = concentration (gm/liter)
- R = hydrometer reading in suspension
- R< = hydrometer reading in "Calgon" solution.

Calculate the Clay percentage, PC from the following:

- Pc = 100 * C/Co; where Co represents the oven-dry weight Co of soil per liter of suspension.

Calculate the Sand percentage, Ps from the following:

- Ps = 100 * S/Co; where S is the weight of the oven-dry sand Co left on screen and Co is as in the Clay formula.

Silt percentage (S) is:

- S = 100 - Pc – Ps

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 020

Total Organic Carbon

Weigh approximately 0.35 g sample into a numbered glazed ceramic boat. Record the weights and boat numbers.

In an acid safe hood, add 1:1 HCl dropwise to each sample until completely moistened, usually 5 to 10 drops. Observe the samples for any bubbling, and note this. Heat the samples on a hot plate until dry. The addition of HCl and hot plate drying must be repeated until no further bubbling occurs. Dry samples in a drying oven at 100oC for 1 hours.

Samples were analyzed using a Leco CR-412 Carbon Analyzer. The instrument was calibrated using CaCO3.

The right anhydrone tube of the furnace was replaced with a chlorine trap before TOC samples were analyzed.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 021

Semivolatile Organic Compounds

Semivolatiles were analyzed using EPA SW-846, Method 8270; Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry/Capillary Technique.

Ten grams of soil/sediment was dried with 50 grams of Na2SO4 and soxhlet extracted for 16 hours with methylene chloride. The extract was concentrated to 1 ml and shot on a Finnigan Incos 50 mass spec.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 022

Analysis For Selected Organophosphate and Synthetic Pyrethroid Pesticides In Animal and Plant Tissue.

This method is suitable for the extraction and quantitation of the following analytes:

1) Organophosphate pesticides- chlorpyrifos, diazinon, EPN, ethyl parathion, malathion, methyl chlorpyrifos, and methyl parathion,

2) synthetic pyrethroids- cypermethrin and fenvalerate, Ten gram tissue samples are thoroughly mixed with anhydrous sodium sulfate and soxhlet extracted with hexane for seven hours. The extract is concentrated by rotary evaporation; transferred to a tared test tube, and further concentrated to dryness for lipid determination. The weighed lipid sample is dissolved in petroleum ether and extracted four times with acetonitrile saturated with petroleum ether. Residues are partitioned into petroleum ether which is washed, concentrated, and transferred to a glass chromatographic column containing 20 grams of Florisil. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction II). For organophosphates, an additional Fraction III (200 ml 50% diethyl ether/50% petroleum ether) is required for complete recovery of requested analytes. Fraction II (and Fraction III, if required) is concentrated to appropriate volume for quantification of residues by capillary column electron capture gas chromatography. Fraction I is concentrated and transferred to a Silicic acid chromatographic column for additional cleanup required for separation of PCBs from other analytes. Three fractions are eluted from the silicic acid column. Each is concentrated to appropriate volume for quantification of residues by megabore column, electron capture gas chromatography. PCBs are found in Silicic acid Fraction II.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 023

Analysis For Selected Organophosphate and Synthetic Pyrethroid Pesticides and Triazine Herbicides in Soil and Sediment.

This method is suitable for the extraction and quantitation of the following analytes: 1) Organophosphate pesticides- chlorpyrifos, diazinon, EPN, ethyl parathion, malathion, methyl chlorpyrifos, and methyl parathion, 2) synthetic pyrethroids- cypermethrin and fenvalerate, 3) Triazine herbicides- atrazine, metribuzin, propazine, simazine, 4) Other herbicides- alachlor and metolachlor.

Twenty gram soil or sediment samples are extracted with acetone, followed by petroleum ether, by allowing to soak one hour in each with intermittent shaking. A final acetone/petroleum ether extraction is done, and the extracts are combined, centrifuged, and transferred to a separatory funnel containing sufficient water to

facilitate partitioning of residues into petroleum ether portion. The petroleum ether is washed twice with water and concentrated by Kuderna-Danish to appropriate volume. An aliquot of the concentrated extract for pesticide determination is transfered to a 1.6 gram Florisil mini-column topped with 1.6 grams sodium sulfate. Residues are eluted from the column in two elution fractions. Fraction I consists of 12 milliliters hexane followed by 12 milliliters of 1% methanol in hexane, and Fraction II consists of an additional 24 milliliters of 1% methanol in hexane. Quantification of residues in the two Florisil fractions is by 30M DB-608 and DB-5 megabore columns, electron capture gas chromatography and by TSD (thermionic specific detector), to detect N and P containing compounds.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 025

Analysis For Selected Organophosphate and Synthetic Pyrethroid Pesticides and Triazine Herbicides in Water

This method is suitable for the extraction and quantitation of the following analytes: 1) Organophosphate pesticides- acephate, azinphos methyl, chlorpyrifos, coumaphos, dementon, diazinon, dichlorvos, dicrotophos, dimethoate, disulfoton, ethoprop, EPN, famphur, fensulfothion, fenthion, malathion, methylparathion, mevinphos, monocrotophos, parathion, phorate, terbufos and trichlorfon 2) synthetic pyrethroids- cypermethrin and fenvalerate, and permethrin 3) Triazine herbicides- atrazine, metribuzin, propazine, simazine.

A 800 milliliter water sample is extracted four times by shaking with 75 milliliter portions of methylene chloride. The four extracts are combined and concentrated by Kuderna-Danish to near dryness, then exchanged into iso-octane, and reconstituted to appropriate volume in hexane. Quantification of residues is by megabore column, electron capture gas chromatography.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 026

Analysis For PCP, Carbaryl, 4-nonylphenol, and Permethrin by GC/MS

A 800 milliliter water sample or all the sample if less than 800 ml is extracted four times by shaking with 50 milliliter portions of methylene chloride. The four extracts are combined and concentrated by Kuderna-Danish. Sample volume is adjusted to one milliliter by N-evap and quantified by Gas Chromatography - Mass Spec.

Standard solutions are diluted with an appropriate solvent and determined by direct injection on GC/MS.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 027

Method for Substituted Urea Herbicides in Water

A measured volume (1000ml) of water is placed into a 2-L separatory funnel and surrogate standard is added. The pH of the sample is adjusted to 7 by the addition of phosphate buffer, then 100g NaC1 is added. The sample is shaken to dissolve the salt. Methylene chloride (60ml) is added and the sample is extracted by shaking for 2 minutes with periodic venting. After the layers have been allowed to separate, the organic layer is drained into a flask, and the extraction repeated for a total of three times. The methylene chloride extracts are combined, then drained through a small amount of sodium sulfate to remove water, into a Kuderna-Danish concentrator. The solvent is concentrated to a small volume, then taken just to dryness with a stream of dry nitrogen. One ml of acetonitrile containing the internal standard is added, and the sample mixed 30 seconds on a Vortex mixer. After filtration, the samples are analyzed on a high performance liquid chromatograph using UV detection at 254 nm.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 028

Analysis For AHH active PCB Congeners In Soil and Sediment

Twenty gram soil or sediment samples are weighed into a pesticide residue quality (PRQ) centrifuge bottle. If the samples are less than 10% moisture, then 10 ml water is added. Fifty ml B & J high purity acetone is added and the sample shaken well six times over a ninety minute period (about every 15 minutes). Fifty mls redistilled petroleum ether is added to the sample and the shaking repeated. The sample is centrifuged and the liquid decanted into a 500 ml separatory funnel containing 200 ml PRQ water and 15 ml saturated sodium chloride solution. Fifty ml of a 1:1 acetone/pet ether mixture is added to the sample and it is shaken, centrifuged, and the liquid added to the separatory funnel. The separatory funnel is shaken vigorously for two minutes and the layers allowed to separate. The pet ether is removed, and the water fraction extracted again with 50 ml petether. The combined pet ether is washed twice with water and concentrated by Kuderna-Danish to appropriate volume. The sample is transferred to a glass chromatographic column containing 20

grams of Florisil. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I). Fraction I is concentrated and transferred to a Silicic acid (Mallinckrodt SillicAR CC-4, activated at least 7 days in a 1300C oven) chromatographic column for additional cleanup required for separation of PCBs from other organochlorines. Three fractions are eluted from the silicic acid column. Fraction I is 20 ml of Petroleum Ether. Fraction II is 150 ml of Petroleum ether and contains the PCBs. Fraction III is 20 mls of 1:19:80 acetonitrile:hexane:methylene chloride and contains most of the other organochlorine pesticides.

Fraction II is concentrated to one ml and fractionated on a carbon column. This column consists of 2 grams of 1:20 AX-21 Carbon on activated silica gel. (AX-21 Activated Carbon, Anderson Development Co., Adrian, MI, vacuum washed with 200 ml each water, methanol, toluene, acetone, methylene chloride, 2X pet ether) (Silica gel: EM Science, Silica gel 60-230 mesh activated @130oC more than 10 days). Fraction A consists of 50 ml Pet Ether. Fraction B is 50 ml 10% methylene chloride in Pet Ether. Fraction C is 120 ml of 50% methylene chloride in Pet ether. Fraction D is 50 ml toluene. Fraction B contains PCB# 128, 138, 158, 166, and 170. Fraction C contains PCB# 105, 114, 118, 123, 156, 157, 167,and 189. Fraction D contains PCB# 077, 126, and 169. The PCB fractions were chromatographed on a Varian 3400 GC using a 60m 0.25mm DB-5 capillary column. Conformation was conducted on a 30m 0.53mm DB-608 megabore column.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 029

Analysis For AHH active PCB Congeners - Micro Method

This method is necessary when sample size is limited (below 4 g, approximately) and in case of organ tissue as substrate and is a modified version of the method described in Manual of Analytical Methods for the Analysis of Pesticides in Humans and Enviromental Samples, EPA-600/8-80-038, June 1980, Section 5, A (2). It is suitable for adipose, kidney, liver, muscle, and other tissues:

Weigh 0.5 g or less of well-mixed tissue into a size 22 Duall tissue grinder. Extract tissue by grinding three times with acetonitrile; the first time being with 4 ml followed by two 2.5 ml portions. Remove the pestle after each grinding and centrifuge,decanting the extract into a 50 ml glass stoppered graduated mixing cylinder. Combine all extracts and record the total volume of the three extracts. Add a volume of PRQ water equivalent to 3.3 times the extract volume. Then add 2 ml saturated NaCl solution. Extract the aqueous acetonitrile mixture with 5 ml hexane by vigorous shaking for 2 minutes. Allow layers to separate, and remove the hexane layer with a Pasteur pipet into a tared 20 ml screw-capped culture tube. Re-extract twice with 4 ml hexane each time, combining the extracts into the culture tube.

Concentrate the combined hexane extracts under nitrogen just to dryness. Weigh the tube to determine the lipid.

The weighed lipid sample is dissolved in petroleum ether and transferred to a glass chromatographic column containing 20 grams of Florisil. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I). Fraction I is concentrated and transferred to a Silicic acid (Mallinckrodt SillicAR CC-4, activated at least 7 days in a 1300C oven) chromatographic column for additional cleanup required for separation of PCBs from other organochlorines. Three fractions are eluted from the silicic acid column. Fraction I is 20 ml of Petroleum Ether. Fraction II is 150 ml of Petroleum ether and contains the PCBs. Fraction III is 20 mls of 1:19:80 acetonitrile:hexane:methylene chloride and contains most of the other organochlorine pesticides.

Fraction II is concentrated to one ml and fractionated on a carbon column. This column consists of 2 grams of 1:20 AX-21 Carbon on activated silica gel. (AX-21 Activated Carbon, Anderson Development Co., Adrian, MI, vacuum washed with 200 ml each water, methanol, toluene, acetone, methylene chloride, 2X pet ether) (Silica gel: EM Science, Silica gel 60-230 mesh activated @130oC more than 10 days). Fraction A consists of 50 ml Pet Ether. Fraction B is 50 ml 10% methylene chloride in Pet Ether. Fraction C is 120 ml of 50% methylene chloride in Pet ether. Fraction D is 50 ml toluene. Fraction B contains PCB# 128, 138, 158, 166, and 170. Fraction C contains PCB# 105, 114, 118, 123, 156, 157, 167,and 189. Fraction D contains PCB# 077, 126, and 169. The PCB fractions were chromatographed on a Varian 3400 GC using a 60m 0.25mm DB-5 capillary column. Conformation was done on a 30m 0.53mm DB-608 megabore column.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 030

DETERMINATION OF STARLICIDE IN WATER

Standard Preparation The concentrated standard solution was made by dissolving 25 mg of 3-chloro-p-toluidine hydrochloride in 25 ml distilled water. Serial dilutions were made from this stock to the appropriate levels for analysis. After trials on the HPLC, 1.0, 0.5, 0.1, and 0.05 ppm were determined to be the best working concentrations. All samples and standards were stored and extracted under as low light conditions as practical to prevent photodegradation of the Starlicide.

HPLC Conditions Separate reservoirs of filtered (0.45 um) degassed water and acetonitrile were delivered at 20% water / 80% acetonitrile. An Alltech Econosil C-18, 5-um, 4.6x250 mm column was used for the separation. The flow rate was 1 ml/min at room temperature. The detector was 241 nm UV.

Method Validation Direct injections of the water samples determined the concentration of Starlicide to be too low to accurately quantitate. The limit of detection of Starlicide in a 100 ul injection was determined to be 0.05 ppm. Three 200 ml 0.01 ppm water spikes were concentrated by passing through 360 mg C-18 Sep-Paks then eluding with 4 mls 100% acetonitrile. This resulted in an increase in detection limit to 0.001 ppm.

Sample	Fortification Level (ug/ml)	Observed Level (ug/ml)
#1	0.01	0.0073
#2	0.01	0.0072
#3	0.01	0.0082
#4	0.001	0.00074

References PM Resources Laboratory Method #840-1 U.S.D.A. Denver Wildlife Research Center Method #28A U.S.D.A. Denver Wildlife Research Center Method #36A

Sample Perparation

Two hundred mls of each sample was passed through a 360 mg C-18 Sep-Pak and eluded with 4 mls 100% acetonitrile. Each sample was injected on the HPLC using the conditions described above.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 031

Accelerated Solvent Extraction (ASE) and Analysis of Synthetic Pyrethroid Pesticides in Filter Disks.

This method is suitable for the extraction and quantitation of synthetic pyrethroids, cypermethrin, and fenvalerate in filter disks. The filter disks were folded and each placed in a clean 11 cm extraction cell. Each cell was extracted with 100% hexane using the following ASE conditions: 5 min. heating cycle, 2x2 min. static cycles, 60% flushing volume, and 2000 psi @ 100oC. The resulting ~18 ml of extract was evaporated to one ml under nitrogen and quantitated on a megabore column with an electron capture gas chromatograph.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 032

Accelerated Solvent Extraction (ASE) and Analysis of Synthetic Pyrethroid Pesticides in Plant Tissue.

This method is suitable for the extraction and quantitation of synthetic pyrethroids, cypermethrin, and fenvalerate in plant tissue. Two grams of chopped plant tissue was mixed with 4.5 grams of Hydromatrix and placed in a clean 33 cm extraction cell. An additional 1 to 2 grams of Hydromatrix was add to each cell to fill the remaining void. The cells were extracted with 50:50 hexane:acetone using the following ASE conditions: 5 min. heating cycle, 2x2 min. static cycles, 60% flushing volume, and 2000 psi @ 100oC. The resulting ~ 55 ml of extract was rotovaporated @ 45oC to near dryness and transfered through sodium sulfate with hexane. The extract was then evapotated under nitrogen to ~5 ml and transfered to a cleanup column containing 25 grams of Florisil topped with 1 cm of sodium sulfate. The column was prewashed with 50 ml hexane and the hexane extract transfered with 3 small hexane rinses. Fraction I was eluted with 200 ml methylene chloride:hexane (20:80) @ ~5 ml/min. This fraction contained cis-permethrin. Fraction II was eluted with 200 ml of acetonitrile:methylene chloride:hexane (0.35:50:50) @ ~5 ml/min. This fraction contained the remaining pyrethroids. Both fractions were combined in a 1000 ml round bottom flask and rotovaped to near dryness @ 45oC. The extract was concentrated to the appropriate volume for quantitation on a megabore column with an electron capture gas chromatograph.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 033

Accelerated Solvent Extraction (ASE) and Analysis of Disyston, Disyston Metabolites, and Oxygen Analoges in Filter Disks.

This method is suitable for the extraction of Disyston, Disyston sulfoxide, Disyston sulfone, and the oxygen analoge of each in filter disks. The filter disks were folded and each placed in a clean 11 cm extraction cell. Each cell was extracted with 50:50 (hexane:acetone) using the following ASE conditions: 5 min. heating cycle, 2x2 min. static cycles, 60% flushing volume, and 2000 psi @ 20oC. The resulting ~18 ml of extract was evaporated to one ml under nitrogen and the water removed by passing sample in petroleum ether through sodium sulfate. The samples were injected on a megabore column with an electron capture gas chromatograph. Only Disyston and Disyston sulfone could be quantitated on the ECD because of thermal break down and conversion to Disyston sulfone of all the other compounds. Disyston sulfoxide and the oxygen analoges can be quantitated if only that single compound is present, but when combined with the Disyston or Disyston sulfone they appear to convert to the Disyston sulfone making quantitation impossible.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 034

Analysis for Chlorophenoxy Acid Herbicides in Liver

Weigh 0.5 gm of liver into a size 22 Duall tissue grinder. Add 2 ml PRQ water and 6 drops 6N HCl. Grind the liver. Extract the liver with 4 ml diethyl ether by grinding. Centrifuge and remove ether to a screw top tube. Extract the liver 2X with 2 ml diethyl ether and once with 2 ml pet. ether. This combined extract contains chlorophenoxy acid herbicides.

Derivitization: Reduce sample volume to approximately 0.5ml and ethylate using diazoethane (15 min.). Exchange to hexane (N-EVAP) and reduce volume to 0.2ml.

Column Clean-Up:

Column clean-up: Place 2.0g of 1% deactivated silica gel in a 7mm id. chromatography column (#22 Kontes). Top with 1cm Na2SO4 and prewet column with 10ml hexane. Collect sample eluents in three fractions as follows:

Fraction A: add sample and rinse container with two 0.5ml washes of 20% benzene in hexane. Elute with 9ml of the same solution. (Contains PCP.)

Fraction B: add 10ml 40% benzene in hexane. Add 10ml 60% benzene in hexane. (Contains Dalapon, Silvex, Dinoseb, portion of Dicamba.)

Fraction C: add 10ml 80% benzene in hexane. Add 10ml 100% benzene. (Contains remaining Dicamba, Dichlorprop, 2,4-D, 2,4,5-T, 2,4-DB, Bentazon, Blazer.)

[Reference for column clean-up for acid herbicides: Shafik, T. A.,H. C. Sullivan, H. R. Enos, 1973." Multiresidue Procedure for Halo- and Nitrophenols. Measurement of Exposure to Biodegradable Pesticides Yielding These Compounds as Metabolites." J. Agr. Food Chem. 21:295-298.]

[Reference for extraction: A modification of Determination of Pentachlorphenol in Blood, Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, Section 5,A,(3),(b).EPA June 1980.

Recoveries: N=3	
Dicamba	93%
Dichloroprop	101%
24D	115%

PCP	96%
Silvex	105%
245T	114%
24DB	98%
Blazer	90%

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 035

Analysis for Organochlorine Pesticides and PCBs in Soil and Sediment by ASE.

Weigh 10-gram soil or sediment sample and 5-grams of Hydromatrix into a PRQ (SOP 1.105) beaker. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. The sample can be stored in a desiccator over night. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cell with a 1.91-cm cellulose filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 6-ml total of pet ether (SOP 1.81) and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 60% solvent flush, 120 sec purge cycle, 100 degrees C @ 2000psi, 1:1 pet ether:acetone (SOP 1.255). Prepare a 500-ml separatory funnel with 200-ml PRQ water (SOP 1.255) and 15-ml PRQ saturated sodium chloride (SOP 1.255). Rinse the sample extract into the separatory funnel with 50ml of 1:1 acetone: pet ether. The separatory funnel is shaken vigorously for one minute and the layers allowed to separate. The pet ether is removed, and the water fraction extracted again with 50-ml pet ether. The combined pet ether is washed twice with 50-ml water and concentrated by Kuderna-Danish (SOP 1.261) to appropriate volume. An aliquot of the concentrated extract representing 2 grams of sample is transferred to a column containing 20 grams of Florisil. The column is eluted with 200 ml 6% diethyl ether/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction II). Fraction II is concentrated to appropriate volume for quantification of residues by a Varian 3600 gas chromatograph equipped with dual 30M DB-5/DB-608 megabore columns and electron capture detectors. Fraction I is concentrated to 5 ml and transferred to a silicic acid (SOP 1.255) chromatographic column (custom columns 1 cm OD x 40 cm with a 100 ml reservoir on top, Ace Glass) for additional cleanup required for separation of PCBs from other organochlorines. Five grams of hot silicic acid is put into the column, which already has a glass wool plug and about 3-mm sodium

sulfate in the bottom. The silicic acid is topped with 10-mm sodium sulfate and prewashed with 10-ml hexane. Three fractions are eluted from the silicic acid column. The sample in 5-ml solvent is added to the column and rinsed into the column with 3,1,1-ml hexane. Then the sample is eluted with 20-ml pet ether (fraction SAI). Fraction SAII is 150-ml pet ether. Fraction SAIII is 20 ml of a mixed solvent consisting of 1 part acetonitrile: 19 parts hexane: 80 parts methylene chloride (SOP 1.255). Each is concentrated to appropriate volume for quantification of residues by megabore column, electron capture gas chromatography. HCB and Mirex are in SAI. PCBs are found in SAII. The rest of the compounds are in SAIII.

GC determinations for SAI and SAIII were run on a Varian 3600 GC with a Varian Star Data System and a Varian 8200 Autosampler. All GCs were equipped with dual DB-608 (0.83u film thickness, J & W Scientific # 125-1730) and DB-5 (1.5u film thickness, J & W Scientific # 125-5032) 30M megabore columns. All compounds were calculated using a three point standard curve forced through the origin using external standards (SOP 1.267).

PCB's (SAII) are shot on a Varian 3350 with a 60M DB-5 capillary column and an EC detector and a Varian Star Data System, version 4.5. All the mixture standards are at 0.5 ng/ul with one ul shot.

Starting with Arochlor 1260, 4 peaks that are unique to this mixture are located. The areas of the standards are summed and the same peaks located in the sample and also summed. Arochlor 1260 is calculated by the following formula to obtain PPM 1260.

$$\frac{\text{(Area sample) (weight of std shot in ng)}}{\text{(Area 1260 std) (basis shot in mg)}}$$

Arochlor 1254 is calculated by locating the major peaks in the mixture that are normally found in samples. The areas of these peaks are summed. Because some of this area comes from Arochlor 1260 and not all from Arochlor 1254, the contribution from the 1260 has to be subtracted from the total area. Arochlor 1254 is calculated by using the formula:

$$\frac{\text{\{(Area sample)-[((PPM 1260) (basis) (area from 1260))/ng 1260 std]\} (wt 1254 std in ng)}}{\text{(Area 1254 std) (Basis shot in mg)}}$$

Results are in PPM.

Arochlor 1248 and Arochlor 1242 are calculated in a similar fashion, subtracting the contribution from 1254 in the 1248 and the 1248 in the 1242.

Total PCBs are calculated by adding the sum of Arochlor 1242, 1248, 1254, and 1260.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 036

Analysis of Soil and Vegetation for Glyphosate

The samples were analyzed using the method of Moye, A. et al., Validation of An Analytical Residue Method for Glyphosate and Metabolite: An Interlaboratory Study. J. Agri. & Food Chem., 1986. 34:955-960. A Partisil-10 SAX column was used as the primary column and a Hamilton PRP X-400 column used for conformation.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 038

Dioxins

Dioxins were analyzed using EPA Method 8290; Analysis of Samples for the Presence of Polychlorinated Dibenzo-p-Dioxins and Dibenzofurans by High-Resolution Chromatography/High-Resolution Mass Spectrometry.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 039

Analysis For Selected Organophosphates in Soil and Sediment by ASE and GPC.

This method is suitable for the extraction and quantitation of the following analytes:

1) Organophosphate pesticides- chlorpyrifos, diazinon, EPN, malathion, methyl parathion, ethyl parathion, phorate, azinphos-methyl, coumaphos, demeton, ethoprop, dimethoate, fensulfothion, fenthion, mevinphos, terbufos, and famphur.

2) Other herbicides- alachlor.

Weigh 10-gram soil or sediment sample and 5-grams of Hydromatrix into a PRQ (SOP 1.105) beaker. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. The sample can be stored in a desiccator over night. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 6-ml total of pet ether (SOP 1.81) and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA Method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 60% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 1:1 pet ether: acetone (SOP 1.255). Prepare a 500-ml separatory funnel with 200-ml PRQ water (SOP 1.255) and 15-ml PRQ saturated sodium chloride (SOP 1.255). Rinse the sample extract into the separatory funnel with 50ml of 1:1 acetone: pet ether. The separatory funnel is shaken vigorously for one minute and the layers allowed to separate. The pet ether is removed, and the water fraction extracted again with 50-ml pet ether. The combined pet ether is washed twice with 50-ml water and concentrated by Kuderna-Danish (SOP 1.261) and exchanged into methylene chloride and adjusted to 4 ml. Two ml of the sample injected on a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into iso-octane. Quantification of residues is by electron capture gas chromatography with both 30M DB-608 and DB-5 megabore columns, and by TSD (thermionic specific detector) with a 30M DB-608 megabore column to detect N and P containing compounds.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 040

Analysis for PAHs in Soil/Sediment and plant tissue by ASE and GPC.

Weigh 10-gram soil/sediment or plant tissue sample and 5-grams of Hydromatrix into a PRQ (SOP 1.105) beaker. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. The sample can be stored in a desiccator over night. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cEll with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 6-ml total of pet ether (SOP 1.81) and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the

collection vial. Operate the ASE according to SOP 1.260 (EPA Method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 60% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 1:1 pet ether: acetone (SOP 1.255).

Prepare a 500-ml separatory funnel with 200-ml PRQ water (SOP 1.255) and 15-ml PRQ saturated sodium chloride (SOP 1.255). Rinse the sample extract into the separatory funnel with 50ml of 1:1 acetone: pet ether. The separatory funnel is shaken vigorously for one minute and the layers allowed to separate. The pet ether is removed, and the water fraction extracted again with 50-ml petether. The combined pet ether is washed twice with 50-ml water and concentrated by Kuderna-Danish (SOP 1.261) to appropriate volume. The sample is dissolved in 4 ml of methylene chloride and 2 ml injected into a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into hexane. The sample is transferred to a column containing 20 grams of 1% deactivated silica gel (SOP 1.255) column (silica gel is added to the column in a pet ether slurry) topped with five grams neutral alumina (SOP 1.255). Aliphatic and polynuclear aromatic hydrocarbon residues are fractionated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) followed by elution of aromatics using first, 100 ml 40% methylene chloride (SOP 1.255)/60% petroleum ether, then 50 ml methylene chloride (Combined elutes, Fraction II). The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride to a known volume, and quantified by gas chromatography - mass spec (SOP 1.276).

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 041

Analysis of Water -- Special for E. Snyder-Conn

1. Record sample volume. Add 800 ml of sample to a 1000-ml separatory funnel. Add 100 grams of sodium chloride and 50 mls phosphate buffer to the funnel and shake to mix.

2. Adjust to pH 8 by adding PRQ 6N KOH (SOP 1.255) or 6N sulfuric acid to sample, and shake vigorously 30 sec. Allow sample to stand one hour at pH 8. Rinse the sample container with 25-ml CH_2Cl_2 (SOP 1.255) and add this to the funnel. Pour the remainder of the sample back into the sample container and rinse the graduated cylinder 2 X 25 ml CH_2Cl_2.

3. Shake 2 min. with periodic venting. Drain CH_2Cl_2 through a funnel of CH_2Cl_2 washed sodium sulfate into a 500 ml French Square bottle if there is no emulsion. Otherwise, drain lower layer into centrifuge bottle and centrifuge to separate layers.

Repeat 2 X 60 ml CH_2Cl_2. This combined extract contains organochlorine and nitrogen/phosphorous containing pesticides.

4. Adjust pH to less than 2 with drop wise addition of PRQ 6N H_2SO_4 (SOP 1.255).

5. Extract water with 100ml Ethyl ether (SOP 1.255) by shaking two minutes. Drain the water from the separatory funnel and pass the ether layer through acidified sodium sulfate into a 500-ml French square bottle. Repeat 2 x 60ml EtoEt. Extract with a final aliquot of 100ml petroleum ether (SOP 1.81). This combined extract contains chlorophenoxy acid herbicides.

6. Concentrate acid and base/neutral extracts separately with Kuderna-Danish evaporators (SOP 1.261).

7. Acid extract (chlorophenoxy acid herbicides) derivitization: Reduce the sample volume to approximately 0.5ml and ethylate using diazoethane (15 min.)(SOP 1.10). Add acid and base/neutral extracts together and concentrate to 1 ml for GC-MS analysis.

8. GC-MS analysis was conducted using a Saturn Ion Trap with a 3400 GC. A SPI 1078 injector was used. Instrument conditions as follows: Injector temperature 220oC, 30M DB-5ms column, 80 o C start temp, 1 min, ramp to 290 o C @8o/min, then hold for 2.75 min. Multiplier 1750 volts, A/M amplitude 3.0 volts, trap temp 190oC, Manifold 50oC, Transfer 285oC. 2ul injection using an 8200 autosampler.

Phosphate Buffer:

- 29.6 ml 0.1N hydrochloric acid & 50 ml 0.1M dipotassium phosphate
- 0.1N hydrochloric acid = 2.5 ml HCl in 296 ml H_2O
- 0.1M dipotassium phosphate = 8.7g dipotassium phosphate to 500 ml H_2O

(This makes approximately 800 ml buffer.)

References:

- E.P.A. Method 507
- E.P.A. Method 508
- E.P.A. National Survey of Pesticides in Drinking Water Wells (11/90) Method 4

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 042

Analysis for Organochlorine Pesticides, N/P Pesticides, Aliphatic and Polynuclear Aromatic Hydrocarbons, and Chlorophenoxy Acid Herbicides in Water

1. Record sample volume and pH. Add 800 ml of sample to a 1000-ml separatory funnel. Add 100 grams of sodium chloride and 50 mls phosphate buffer to the funnel and shake to mix.

2. Adjust to pH 8 by adding PRQ 6N KOH (SOP 1.255) or 6N sulfuric acid to sample, and shake vigorously 30 sec. Allow sample to stand one hour at pH 8. Rinse the sample container with 25-ml CH_2Cl_2 (SOP 1.255) and add this to the funnel. Pour the remainder of the sample back into the sample container and rinse the graduated cylinder 2 X 25 ml CH_2Cl_2.

3. Shake 2 min. with periodic venting. Drain CH_2Cl_2 through a funnel of CH_2Cl_2 washed sodium sulfate into a 500 ml French Square bottle if there is no emulsion. Otherwise, drain lower layer into centrifuge bottle and centrifuge to separate layers. Repeat 2 X 60 ml CH_2Cl_2. This combined extract contains organochlorine, aliphatic and aromatic hydrocarbons, and nitrogen/phosphorous containing pesticides.

4. Adjust pH to less than 2 with drop wise addition of PRQ 6N H_2SO_4 (SOP 1.255).

5. Extract water with 100ml Ethyl ether (SOP 1.255) by shaking two minutes. Drain the water from the separatory funnel and pass the ether layer through acidified sodium sulfate into a 500-ml French square bottle. Repeat 2 x 60ml EtoEt. Extract with a final aliquot of 100ml petroleum ether (SOP 1.81). This combined extract contains chlorophenoxy acid herbicides.

6. Concentrate acid and neutral extracts separately with Kuderna-Danish evaporators (SOP 1.261).

7. Column Clean-Up:

* NEUTRAL FRACTION (N/P and organochlorine pesticides, Aliphatic and polynuclear aromatic hydrocarbons). Adjust sample extract to exact volume and remove an appropriate aliquot for column clean-up techniques specific to the analyte, for pesticides use florisil (SOP 1.194), for hydrocarbons use 1% deactivated silica gel (SOP 1.203), for organophosphates, shoot directly on a NP-detector.

*ACID FRACTION (Chlorophenoxy acid herbicides) - Derivitization: Reduce the sample volume to approximately 0.5ml and ethylate using diazoethane (15 min.)(SOP 1.10). Exchange to hexane (SOP 1.68) and reduce volume to 0.2ml.

* Column clean-up (CPA): Place 2.0g of 1% deactivated silica gel (SOP 1.255) in a 7mm id. chromatography column (#22 Konte s). Top with 1cm Na_2SO_4 (SOP 1.255) and prewet column with 10ml hexane (SOP 1.255). Collect sample eluents in three fractions as follows:

Fraction A: add sample and rinse container with two 0.5ml washes of 20% benzene (SOP 1.255) in hexane. Elute with 9ml of the same solution.(Contains PCP.)

Fraction B: add 10ml 40% benzene in hexane. Add 10ml 60% benzene in hexane. (Contains Dalapon, Silvex, Dinoseb, portion of Dicamba.)

Fraction C: add 10ml 80% benzene in hexane. Add10ml 100% benzene. (Contains remaining Dicamba, Dichlorprop, 2,4-D, 2,4,5-T, 2,4-DB, Bentazon, Blazer.)

Reference for column clean up for acid herbicides: Shafik, T. A., H. C. Sullivan, H. R. Enos, 1973." Multiresidue Procedure for Halo- and Nitrophenols. Measurement of Exposure to Biodegradable Pesticides Yielding These Compounds as Metabolites." J. Agr. Food Chem. 21:295-298.

Phosphate Buffer: 29.6 ml 0.1N hydrochloric acid & 50 ml 0.1M dipotassium phosphate

- 0.1N hydrochloric acid = 2.5 ml HCl in 296 ml H2O
- 0.1M dipotassium phosphate = 8.7g dipotassium phosphate to 500 ml H2O (This makes approximately 800 ml buffer.)

References:

- E.P.A. Method 507
- E.P.A. Method 508
- E.P.A. National Survey of Pesticides in Drinking Water Wells (11/90) Method 4

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 043

Analysis For Triazine Herbicides in Soil, Sediment and Tissue.

This method has been found suitable for the extraction and quantitation of the following triazine herbicides- atrazine, metribuzin, and cyanazine.

Weigh 10-gram soil / sediment or 5-gram tissue sample and 5-grams of Hydromatrix into a PRQ (SOP 1.105) beaker. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. The sample can be stored in a desiccator over night. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 5-ml total of methylene chloride and add the rinses to the cell. Place the

top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA Method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 60% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 10% methanol in methylene chloride. The sample is concentrated by Turbovap to appropriate volume (SOP 1.298). The sample is dissolved in 4 ml of methylene chloride and 2 ml injected into a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into hexane.

Quantification is by Varian 3400 GC with a 30M RTX-200 meg abore column and a TSD (thermionic specific detector), to detect N containing compounds.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 044

Analysis of Synthetic Pyrethroid Pesticides in Bat Guano.

This method is suitable for the extraction and quantitation of the following synthetic pyrethroids-, cis and trans permethrin, cypermethrin and fenvalerate.

A five-gram guano sample is weighed into centrifuge bottle. If the samples are less than 40% moisture, then 5 ml of pet ether extracted water is added to each sample.

The samples are extracted with 50-ml acetone, followed by 50-ml petroleum ether, by soaking samples for 1 1/2 hour in each with intermittent shaking. The samples are centrifuged, and transferred to a 500-ml separatory funnel containing 200-ml pet ether extracted water and 15-ml saturated sodium chloride solution. A final 50 ml 1:1 acetone/petroleum ether extraction is done. The samples are centrifuged and the extracts combined in the separatory funnel. The petroleum ether is washed twice with water and concentrated by Kuderna-Danish to 5-ml. The sample is transferred to a 300 ml glass chromatographic column (Kontes # 420280-0242) containing 20 grams of Florisil topped with 1 cm of sodium sulfate and the sample tube rinsed three times with about 2-ml pet ether. The column is eluted with 200-ml 6% diethyl ether /94% petroleum ether followed by 200-ml 15% diethyl ether/85% petroleum ether. The diethyl ether used in this analysis contains 2% ethanol. The samples are concentrated to an appropriate volume for quantification of residues by megabore column electron capture gas chromatography (DB-608 and DB-5 dual columns). GC determinations were run on a Varian 3600 GC with a Varian Star Data System and a Varian 8200 Autosampler. All GCs were equipped with dual DB-608 (0.83u film thickness, J & W Scientific # 125-1730) and DB-5 (1.5u film thickness, J & W Scientific # 125-5032) 30M megabore columns. All compounds were calculated

using a three point standard curve forced through the origin using external standards.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 045

Analysis for PCB Congeners In Animal Tissue or Soil/Sediment

Five-gram tissue samples or ten-gram sediment samples are weighed into a 150-ml beaker then thoroughly mixed with 5-grams of Hydromatrix. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample. Rinse the funnel, spatula, and beaker with no more than 5-ml total of methylene chloride and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 60% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, methylene chloride (SOP 1.255).

If organochlorine pesticides and PCB congeners are requested on the same sample and the sample size is too small to run both analysis then the following modification is used. The samples are extracted as in method 1. The remainder of the sample not shot on GPC is used for PCB congeners starting with clean-up on the H4IIE columns. The lipid tube is rinsed with 20 ml of methylene chloride and the GPC vial is rinsed with 30 ml for a total of 50 ml of methylene chloride on column. This is added at step 6 below.

Column Chromatography Clean-Up of Tissue for H4IIE Bioassay

1. PRQ 2-cm i.d. column. Place glass wool in bottom of each column and cover with ca1-cm anhydrous sodium sulfate.

2. Add 10 ml KS (potassium silicate) to each column and gently tap to settle and level column.

3. Next, add 10-ml 40/60 SA/SG (sulfuric acid/silica gel), again gently settling and leveling.

4. Place a 1-cm layer of sodium sulfate over the SA/SG. Then add 15 ml of 30/70 SA/SG and settle.

5. Presaturate each column with 25-30 ml dichloromethane, allowing the solvent to descend just to the top of the absorbent. Close the stopcocks and place 60 ml I-Chem vial under each column. Have a second vial ready for each sample.

6. Pour the sample extract from the ASE extraction on to the top of the column and open the stopcock. Allow the sample to flow into the SA/SG until the extract is about 3 to 4 cm above the SA/SG. Close the stopcock and stir the top layer of SA/SG with a metal rod to remove the gas bubbles. Disturb the sodium sulfate as little as possible. Rinse the rod with dichloromethane back into the sample.

7. Open the stopcock and allow the sample solution to descend into the absorbent. Rinse the sample vial with three 5-ml portions of dichloromethane, sequentially adding these rinses to the column. Wash the column walls with three 3-ml portions of dichloromethane. When all the washes have descended into the absorbent, close the stopcock and add approx. 1 cm of sodium sulfate to the top of the column. Change the receiver vial.

8. Gently pour 50 ml of dichloromethane into the column reservoir and open the stopcock. Flow at 3 ml/min until all effluent is collected.

9. Turbovap extracts to about 10-ml, combine extracts into one vial per sample,add 6-ml of isooctane to each flask and turbovap to 3-ml. Hold extracts for addition to next column.

10. Pack a 1-cm i.d. column as follows: a plug of glass wool, 1-cm sodium sulfate, 10-ml SG-60 silica gel (washed with dichloromethane), 3-ml KS, 5-ml 40/60 SA/SG, and 1-cm sodium sulfate. Presaturate column with 3% dichloromethane: 97% hexane. Place 60 ml I-Chem vial under column. Have a second vial ready for each sample. Transfer sample to top of column and rinse sample flask with three 3-ml portions of eluant, sequentially applying the rinses to the column. Pour 70-ml of the eluant into the column and adjust flow rate to 3-ml/min. Change the vial when the column is about half finished.

11. Add 1-ml of hexane to each vial as a "keeper" and turbovap to about 10-ml. Combine extracts and concentrate to about 1 ml. The sample is now ready for the carbon column.

The sample is fractionated on a carbon column. This column consists of 2 grams of 1:20 AX-21 Carbon on activated silica gel (SOP 1.255). Fraction A consists of 200-ml methylene chloride and contains most of the PCB's. Fraction B is 75-ml redistilled toluene (SOP 1.255) and contains PCB# 77, 81, 126, and 169.

Use a 200ml chromatography column fitted with a stopcock (such as Kontes 420280-0213). Pack the column as follows: a plug of glass wool, 1 cm sodium sulfate, 2 grams of carbon/silica gel, and 1 cm sodium sulfate. The carbon/silica gel needs to be added to the 200 ml column through a long glass funnel that will reach the bottom of the column. If PCB#52 is requested, then pre-wash the column with 200 ml CH_2Cl_2. If this analyte is not requested, then pre-wash the column with 25 ml

CH2Cl2.. Do not let the column go dry. Add the sample and rinse 3X 1ml methylene chloride. Elute the first fraction (C-1) with 200 ml methylene chloride. Elute the 2nd fraction (C-2) with 75 ml redistilled toluene. Concentrate and exchange C-1 to hexane, with the volume dedending on the concentration of PCB's in the sample as determined in the OC analysis. C-2 is concentrated to 1 ml.

The PCB fractions were chromatographed on a Varian 3400 GC using a 60m 0.25mm DB-XLB capillary column (SOP 1.265) and calculated by the software using a three point standard curve forced through the origin. Confirmation was done on a 60m 0.25mm DB-5 capillary column (SOP 1.265). Mass spec confirmations were run on a Saturn 2000 ion trap with a 60m DB-XLB capillary column and a 1078 injector.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 046

Dioxin and Furan in Tissue

Add 5-g hydromatrix to a 10g portion of ground tissue. Load into ASE cell. Add 20ul of the surrogate mixture to the sample. A 10-g sample requires the addition of 1000 pg. 13C12-2,3,7,8-TCDD to give the required 100-ppt fortification level.

ASE extract with methylene chloride, 60% flush Column Chromatography Clean-Up of Fish for H411E Bioassay

1. PRQ 2-cm id. column. Place glass wool in bottom of each column and cover with ca 1-cm anhydrous sodium sulfate.

2. Add 10 ml KS (potassium silicate) to each column and gently tap to settle and level column.

3. Next, add 10-ml 40/60 SA/SG (sulfuric acid/silica gel), again gently settling and leveling.

4. Place a 1-cm layer of sodium sulfate over the SA/SG. Then add 15 ml of 30/70 SA/SG and settle.

5. Presaturate each column with 25-30 ml dichloromethane, allowing the solvent to descend just to the top of the absorbent. Close the stopcocks and place 60-ml vial under column.

6. Pour the sample extract on to the top of the column and open the stopcock. Allow the sample to flow into the SA/SG until the extract just reaches the sodium sulfate between the SA/SG layers. Close the stopcock and stir the top layer of SA/SG with a

metal rod to remove the gas bubbles. Disturb the sodium sulfate as little as possible. Rinse the rod with dichloromethane back into the sample.

7. Open the stopcock and allow the sample solution to descend into the absorbent. Rinse the sample vial with three 5-ml portions of dichloromethane, sequentially adding these rinses to the column. Wash the column walls with three 3-ml portions of dichloromethane. When all the washes have descended into the absorbent, close the stopcock and add approx. 1 cm of sodium sulfate to the top of the column.

8. Gently pour 50 ml of dichloromethane into the column reservoir, change collection vial, and open the stopcock. Flow at 3 ml/min until all effluent is collected.

9. Turbovap both vials of extract to about 10-ml. Combined both fractions into one vial using hexane. Turbovap to about 5 ml of hexane. Hold extracts for addition to next column.

10. Pack a 1-cm id. column as follows: a plug of glass wool, 1-cm sodium sulfate, 10-ml SG-60 silica gel (washed with dichloromethane), 3-ml KS, 5-ml 40/60 SA/SG, and 1-cm sodium sulfate. Presaturate column with 3% dichloromethane: 97% hexane. Place 60-ml vial under column. Transfer sample to top of column and rinse sample flask with three 3-ml portions of eluant, sequentially applying the rinses to the column. Pour 70-ml of the eluant into the column and adjust flow rate to 3-ml/min. Change vial so that each vial contains about half of the column eluant (approx. 50-ml).

11. Add 1-ml of hexane to each flask as a “keeper” and turbovap evaporate to about 1-ml.

Alumina Column Cleanup

Pack a column (glass, 30 cm X 10.5 mm) fitted with a Teflon stopcock, with alumina as follows:

Glass wool plug, 4 g sodium sulfate, 4 g activated neutral alumina, 4 g sodium sulfate. Elute with 10-ml hexane.

Apply sample to top of alumina column. Rinse flask 2X 2ml hexane. Elute with 20 ml hexane, save. Elute with 15 ml of 60% methylene chloride/hexane. Concentrate to 2 ml.

Prepare an AX-21/Celite 545 column as follows: Mix 5.4-g active carbon AX-21 and 62.0 g Celite 545 to produce an 8% (w/w) mixture. Activate the mixture at 130oC for 6 hours and store in a desiccator.

Cut off both ends of a 10 ml disposable serological pipet to give a 10 cm long column. Fire polish both ends and flare. Insert a glass wool plug at one end, the pack the column with 1 cm Celite 545, then 1-g carbon mixture, then 1-cm Celite 545 and another glass wool plug.

Calibration of carbon: add 50ul of the continuing calibration solution to 950-ul hexane. Run this through the column, concentrate to 50ul and analyze. If the recovery of any analytes is less than 80%, discard the carbon.

Rinse the carbon column with 5 ml toluene, then 2 ml 75:20:5(v/v) methylene chloride/methanol/toluene, then 1 ml 1:1 (v/v) cyclohexane/methylene chloride, and 5 ml hexane. Flow rate less than 0.5 ml/min. Add sample to the column, rinse tube with 2X 1ml hexane. Elute with 2X2ml hexane, 2-ml cyclohexane/methylene chloride (50:50) and 2-ml methylene chloride/methanol/toluene (75:20:5). Turn the column upside down and elute the PCDD/PCDF fraction with 20-ml toluene. Concentrate to 0.1ml; transfer to calibrated 0.2- ml autosampler insert and concentrate to 50 ul. Rinse container 3X 300ul of 1%toluene in methylene chloride and add to insert, evaporating sample as necessary. Add 20ul nonane internal standard solution. (13C12-1,2,3,4-TCDD at 200pg/ul per compound) Store samples in the dark, in a freezer until ready for MS. Just before shooting, adjust the sample to 100ul final volume. The samples are shot on a Varian Saturn 2000 ion trap in MS/MS mode. A 50ul sample is shot using an 8200 autosampler with a 100-ul syringe. The ion trap has a programable 1078 split / splitless injector operated in splitless mode. A 60M DB-XLB capillary column with a 0.25 film thickness was used.

For fish tissue:	**(2,3,7,8-TCDD)**
Lower MCL	1.0ppt
Upper MCL	200ppt
Weight (g)	10
IS Spiking level (pg/ul)	40
Final Extract Vol.(ul)	100

Reagent Preparations Preparation of Washed Silica Gel

Place 70-230 mesh silica gel in a large glass fritted funnel and wash with dichloromethane (pesticide residue grade). Let dichloromethane flow by gravity. Use 500-ml dichloromethane per 300 ml (150 grams) of silica gel. When funnel is no longer dripping, apply a vacuum until all liquid solvent is removed and silica gel begins to dry. Pour silica gel into shallow PRQ glass pan and air dry in hood overnight. Cover the pan with PRQ aluminum foil and activate in 130 C oven.

Preparation of Sulfuric Acid / Silica Gel (SA/SG)

Determine the amount of each type of SA/SG needed for sample clean up since the shelf life of these products is only six months. (If these products appear lumpy before the six months have elapsed, discard and make a fresh batch.) Weight washed silica gel into 4-liter glass bottle with Teflon lined cap. Calculate 2/3 of the weight of the silica gel and add this amount of concentrated sulfuric acid for 40/60 SA/SG or 3/7 of the weight for 30/70 SA/SG. Shake until there are no lumps and the silica gel appears dry and powdery. Label each bottle with contents and expiration date. Store at room temperature.

Preparation of Potassium Silicate (KS)

Pour 750 ml of PRG methanol into a 2 liter round bottom flask (be sure to use Teflon sleeve in flask joint). Weigh out 168 grams of KOH pellets and add to flask. Place flask on Rotovap with full water bath (water will dissipate the heat). Rotate flask until KOH dissolves. Remove flask from Rotovap heat water bath to 55 C. Slowly add 300 grams of washed silica gel, swirling the flask to prevent the formation of lumps. Return flask to the Rotovap and rotate for 90 mins.

Set up a large column plugged with glass wool and two 600-ml beakers to catch the column effluent. Swirl the flask and quickly pour as much of the slurry as possible into the column. Exchange beakers and resuspend the solid material in the flask with the methanolic KOH effluent until all of the silica gel is in the column. Allow the liquid level to descend to the top of the KS, then wash 3X with 100 ml of methanol. When the methanol level has reached the top of the KS after the last wash, add 175-ml dichloromethane. Wash with dichloromethane twice more and allow column to drain after the final wash. Apply approx. 2 psi of nitrogen to the top of the column and continue blowing until column is no longer cold (approx. 4 hours). Pour KS into shallow glass pans and air dry in hood overnight. Cover pan with PRQ aluminum foil and activate at 130 C overnight. Pour all KS into 4 liter bottle and roll to mix.

Weigh 1.00 gram of KS into 125 ml Erlenmeyer flask and add 25 ml distilled water.

Stopper flask and shake for 30 min. Prepare 1.21 mM/ml HCl solution (5.0 ml conc. HCl + 45.0 ml distilled water). Calibrate pH meter at pH 10 and pH 7. Add a stirring bar to the KS flask and slowly titrate until pH 6.9 to 7.1 has been attained. The solution is slow to equilibrate after each addition of acid, because of the KOH leaching out of the pores of the silica gel. Determine the moles of KOH / gram of KS from the amount of HCl needed to neutralize the KS. The KS should contain between 4.8 and 5.3 mMoles / gram to be suitable for use. Pour KS into smaller bottles, label, and store at 130 C until needed.

Reference:

Sample Preparation for fish in the BEST Mississippi Basin Study-H411E Bioassay, Standard Operating Procedures of Midwest Science Center, 4200 New Haven Road, Columbia, MO, 65201.

EPA Method 8290: Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography / High-Resolution Mass Spectrometry (HRGC/HRMS)

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 047

Synthetic Pyrethroid Pesticides / Micromethod

This method is suitable for the extraction and quantitation of the following analytes: cis and trans permethrin, cypermethrin and fenvalerate. One gram of tissue (or the entire sample if smaller) is weighed into a size 22 Duall tissue grinder. The sample is extracted three times with methylene chloride. The extracts are filtered through sodium sulfate into a screw top tube. The Methylene chloride is evaporated and the residue brought up to 1 ml with toluene. The samples are shot directly into a GC containing dual megabore columns with no clean-up.

Note: This procedure is hard on the GC, but necessary for very small samples.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 048

Analysis For Carbamates In Tissue

Ten-gram samples are weighed into a 150-ml beaker then thoroughly mixed with 5-grams of Hydromatrix. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 5-ml total of methylene chloride and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 60% solvent flush, 60 sec purge cycle, 40 degrees C @ 1500psi, 100% methylene chloride. The extract is concentrated by Turbovap; transferred to a 4-ml calibrated test tube through a Pasteur pipette containing sodium sulfate using methylene chloride, and concentrated to the 4-ml mark. The fat is removed by injecting 2 ml (5 gram basis) on a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap to 1 ml in methylene chloride. Transfer sample solution onto aminopropyl Bond-Elute extraction cartridge(100 mg/1 ml volume) prewashed with 1 ml methylene chloride. Rinse sample tube with 1 ml 1.5% methanol in methylene chloride and add to column. Elute carbamate residues with 5 ml 1.5% methanol in methylene chloride. (Note: If recovery of aldicarb sulfoxide drops below 80% increase wash volume by 5 ml.) N-evap to dryness at no more than 30 degrees Centrigrade. Do not over dry. Dissolve residue in 1 ml of MeOH. Add 4-ml water for a 1gram/ml basis. Filter samples through 0.45 um teflon filters before injection on HPLC.

Analysis of samples for carbamates was performed on the Waters Carbamates analysis system equipped with a WISP model 712 autosampler (SOP 1.102). 500ul of sample was injected. The separation of the analytes was at room temperature, and the column used was a 3.9mm x 150mm waters Nova-pak C18(4u). The solvent gradient used was as follows:

TIME	FLOW	%WATER	%METHANOL	%ACETONITRILE	CURVE
INIT	1	88	12	0	*
4	1	88	12	0	1
4.1	1	68	16	16	5
16.1	1	30	35	35	10
25	1	88	12	0	9

The post-column derivatization was done at 80.0oC. The samples were hydrolyzed with 0.05m NaOH, then reacted with o-phthaldialdehyde and 2-mercaptoethenol in 0.05m sodium borate decahydrate to yield a highly fluorescent isoindole product. The flow from the post-column pumps was 0.5ml/min. The detector was a Waters 470 scanning fluorescence detector with ex at 338nm and em at 400nm.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 049

Analysis for Pyrethroids in Animal Tissue

This method has been found suitable for the extraction and quantitation of the following Pyrethroids: Cis and trans permethrin, cybermethrin and fenvalerate.

Weigh 5-gram tissue sample and 5-grams of Hydromatrix into a PRQ (SOP 1.105) beaker. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. The sample can be stored in a desiccator over night. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 5-ml total of methylene chloride and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA Method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 60% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 1:1 hexane acetone. The sample is concentrated by Turbovap to appropriate volume (SOP 1.298). The sample is

dissolved in 4 ml of methylene chloride and 2 ml injected into a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into hexane. Quantification is by Varian 3400 GC with a 60M DB-XLB capillary column and a ECD. A secondary detection system is a Varian 3600 with dual 30M megabore columns, DB-608 and DB-5.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 050

Analysis For Organochlorines Pesticides and PCBs in Animal Tissue using soxhlet/GPC.

Five-gram fish samples or two-gram egg samples are weighed into a 250 ml beaker then thoroughly mixed with 150 grams (5g samples) or 75 grams (2g samples) of anhydrous sodium sulfate (SOP 1.255). The samples are stored in a desiccator overnight. The samples are then soxhlet extracted (SOP 1.259) with 600 ml hexane (SOP 1.255) for seven hours. The extract is concentrated by rotary evaporation (SOP 1.129); transferred to a tarred test tube through a Pasteur pipette containing sodium sulfate, and further concentrated to dryness for lipid determination (SOP 1.264). The weighed lipid sample is dissolved in 4 ml of methylene chloride and the fat removed by injecting 2 ml on a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into iso-octane. The sample is transferred to a 300 ml glass chromatographic column (Kontes # 420280-0242) containing 20 grams of Florisil (SOP 1.255) topped with 1 cm of sodium sulfate and the sample tube rinsed three times with about 2 ml pet ether. The column is eluted with 200 ml 6% diethyl ether (SOP 1.255)/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction II). If Endosulfan II and/or Endosulfan Sulfate analysis is required, then 200 ml of 50% diethyl ether/petroleum ether (Fraction III) is required. The diethyl ether used in this analysis contains 2% ethanol (SOP 1.255). Fractions II and III are concentrated to an appropriate volume for quantification of residues by megabore column electron capture gas chromatography (SOP 1.265)(DB-608 and DB-5 dual columns) Dieldrin and Endrin are in Fraction II, and some delta BHC. Fraction I is concentrated to 5 ml and transferred to a Silicic acid (SOP 1.255) chromatographic column (custom columns 1 cm OD x 40 cm with a 100 ml reservoir on top, Ace Glass) for additional cleanup required for separation of PCBs from other organochlorines. Five grams of hot Silicic Acid is put into the column, which already has a glass wool plug and about 3-mm sodium sulfate in the bottom. The Silicic acid is topped with 10-mm sodium sulfate and prewashed with 10-ml hexane. Three fractions are eluted from the silicic acid column. The sample in 5-ml solvent is added to the column and rinsed into the column with 3,1,1-ml hexane. Then the sample is eluted with 20-ml pet ether (fraction SAI). Fraction SAII is 150-ml pet ether. Fraction SAIII is 20 ml of a mixed solvent consisting of 1 part acetonitrile,

19 parts hexane and 80 parts methylene chloride (SOP 1.255). Each is concentrated to appropriate volume for quantification of residues by megabore column, electron capture gas chromatography. HCB and Mirex are in SAI. PCBs are found in SAII. The rest of the compounds are in SAIII. GC determinations were run on a Varian 3600 GC with a Varian Star Data System and a Varian 8200 Autosampler. All GCs were equipped with dual DB-608 (0.83u film thickness, J & W Scientific # 125-1730) and DB-5 (1.5u film thickness, J & W Scientific # 125-5032) 30M megabore columns. All compounds were calculated using a three point standard curve forced through the origin using external standards (SOP 1.267).

PCB's were determined by shooting SAII fractions on a Varian 3400 GC with a Varian Star Data System and a Varian 8200 Autosampler. This GC is equipped with a 60M DB-5 0.25 ID capillary column. Another 3400 equipped with a 60M DB-XLB 0.25 ID capillary column is also used as a second system for PCB's. The compounds were calculated in the following manner. All the aroclor standards are at 0.5 ng/ul with one ul shot.

Starting with Aroclor 1260, 4 peaks that are unique to this mixture are located. The areas of the standards are summed and the same peaks located in the sample and also summed. Aroclor 1260 is calculated by the following formula to obtain PPM 1260.

(Area sample) (weight of std shot in ng)
(Area 1260 std) (basis shot in mg)

Aroclor 1254 is calculated by locating the major peaks in the mixture that are normally found in samples. The areas of these peaks are summed. Because some of this area comes from Aroclor 1260 and not all from Aroclor 1254, the contribution from the 1260 has to be subtracted from the total area. Aroclor 1254 is calculated by using the formula:

- {(Area sample)-[((PPM 1260) (basis) (area from 1260))/ng 1260 std]} (wt 1254 std in ng) (Area 1254 std) (Basis shot in mg)

Results are in PPM.

Aroclor 1248 and Aroclor 1242 are calculated in a similar fashion, subtracting the contribution from 1254 in the 1248 and the 1248 in the 1242.

Total PCBs are calculated by adding the sum of Aroclor 1242, 1248, 1254, and 1260.

Basis = (weight of the sample mg/final volume of sample ul)(ul of sample shot)

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 051

Analysis For Organochlorines Pesticides and PCBs, Aliphatic and Polynuclear Aromatic Hydrocarbons In Soil and Sediment, GPC cleanup

Weigh a ten-gram soil or sediment samples into a PRQ (SOP 1.105) centrifuge bottle. Add 50-ml acetone, and shake for 1 minute every 15 minutes, for 11/2 hours. Add 50-ml petroleum ether and repeat the shaking. Centrifuge the sample and decant the liquid into a 500-ml separatory funnel containing 200-ml PRQ water (SOP 1.255) and 15-ml PRQ saturated sodium chloride (SOP 1.255). Extract the sample again for 1 minute with 50 ml 1:1 acetone/petroleum ether (SOP 1.255), centrifuge and decant the liquid into the separatory funnel. The separatory funnel is shaken vigorously for one minute and the layers allowed to separate. The pet ether is removed, and the water fraction extracted again with 50-ml pet ether. The combined pet ether is washed twice with 50-ml water and concentrated by Kuderna-Danish (SOP 1.261) to appropriate volume. The sample is dissolved in 4 ml of methylene chloride and 2 ml (5 gram equalivant) injected into a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). This fraction is analyzed for PAHs. The remainder of the sample is diluted to 4 ml with methylene chloride, and 1.6 ml (2grams) shot on the Waters high pressure GPC. This fraction is analyzed for OCs.

PAH

The fraction is concentrated by Turbovap and then exchanged into hexane. The sample is transferred to a column containing 20 grams of 1% deactivated silica gel (SOP 1.255) column (silica gel is added to the column in a pet ether slurry) topped with five grams neutral alumina (SOP 1.255). Aliphatic and polynuclear aromatic hydrocarbon residues are fractionated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) followed by elution of aromatics using first, 100 ml 40% methylene chloride (SOP 1.255)/60% petroleum ether, then 50 ml methylene chloride (Combined elutes, Fraction II). The silica gel Fraction II containing aromatic hydrocarbons is concentrated, reconstituted in methylene chloride to a known volume, and quantified by gas chromatography - mass spec (SOP 1.276) on a Varian Ion Trap GC/MS with a 30M DB-5MS capillary column. Fraction I is concentrated and shot on a Varian 3400 GC with a 30M RXT-1 megabore column and a FID detector. Aliphatics from C-10 to C-34 are identified.

OCs

The 2-gram aliquot of the extract is transferred to a 1.6 gram Florisil mini-column topped with 1.6-gram sodium sulfate. Residues are eluted from the column in two elution fractions. Fraction I consists of 12 milliliters hexane followed by 12 milliliters of 1% methanol in hexane, and Fraction II consists of an additional 24 milliliters of 1% methanol in hexane. If additional cleanup is required to separate PCBs from other organochlorines in Fraction I, further chromatography on a Silicic acid column

is performed. Quantification of residues in the two Florisil fractions and three Silicic acid fractions is by megabore column, electron capture gas chromatography.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 052

Benomyl in Water

Midwest Research Institute Pesticide Analysis Method Manufacturing Site: DuPont, Texas Pesticide Class: Urea (Benomyl)

Benomyl is known to be slowly hydrolyzed to carbendazim in water. Since neither the rate nor the extent of conversion is predictable, it was decided to develop the method around the total conversion to carbendazim. This method involves a procedure to complete the hydrolysis process and an analysis for Benomyl as carbendazim using the method previously developed for carbendazim. Included in this method are the parameters developed for the determination of carbendazim.

1. Application: Analysis of wastewater samples for Benomyl

2. Chemistry

- CAS Nomenclature: Methyl [1-[(butyl- Amino) carbonyl]-1H-benzimidazol-2-yl]carbamate
- Trade Names: Benlate, Tersan
- Molecular Weight: 290
- Molecular Weight carbendazim = 191.2
- Conversion to benomyl: ppm x 290/191.2 or ppm x 1.52

3. Apparatus

a. Instrumentation: Waters Associates dual Model 6000/A pumps, Model 660 solvent programmer, U6K injector and Model 440 absorbance detector.

b. Operating Parameters:

(1) Detector: Set at a wavelength of 280 nm.

(2) Column: Waters Associates uBondapak C18 (10 um, 4 mm x 30 cm ID)

(3) Guard Column: Whatman CO:PELL ODS (30-38 um, 4 mm x 7 cm ID)

c. Glassware:

- Kuderna-Danish (K-D) 3 Ball Snyder column 250 ml evaporator 10 ml receiver
- Chromatographic column: 2.5 x 15 cm
- Sample filtration apparatus:
- 10 ml. Syringe, Luer slip;
- Stainless steel Swinney, 13 mm;
- Millipore Fluoropore filter, 13 mm
- Separatory funnel: 2 liters
- Solvent filtration apparatus:
- 1 liter suction flask
- Millipore filter holder, 47 mm
- Millipore Fluoropore filter, 47 mm
- Syringes: 10 1, 25 1, 50 1, 100 1
- Screw cap vials: 7 ml

d. Chemicals:

- Carbendazim Standard: Supplied by EPA/HERL, No. 1071
- Benomyl Standard: Supplied by EPA/HERL, No. 0500
- Distilled in Glass Solvents: Supplied by Burdick and Jackson
- methylene chloride, hexane, petroleum ether, diethyl ether, acetonitrile, methanol.
- Florisil (PR)
- Sodium sulfate, anhydrous

4. Standards

a. Stock: A 25 mg/25 ml stock solution was prepared in acetonitrile.

b. Working: The stock was diluted to prepare a 100 ug/ml standard. The 100 ug/ml standard diluted to prepare 20, 10, 5, 1 and 0.5 ug/ml standards.

5. Procedures

a. Sample handling: One liter of neutral wastewater and 10 ml of HC1 are stirred for 24 hr to assure the complete hydrolysis of Benomyl to carbendazim. The pH was then raised to 7 for sample extraction.

b. Extraction: One hundred milliliter portions of each sample were extracted with three 35 ml volumes of methylene chloride. The sample was shaken each time for 2 min. The methylene chloride was passed through a 5 cm long column of anhydrous sodium sulfate and drained into a 250 ml K-D. A 60 ml methylene chloride final wash of the sodium sulfate column was added to the K-D evaporator.

The extract was concentrated to 10 ml in a K-D evaporator on a steam bath. The solvent remaining was evaporated under a stream of nitrogen and the residue redissolved into 5 ml acetonitrile.

c. High Pressure Liquid Chromatography: The sample extracts and carbendazim standards were analyzed using as a mobile phase: 80/19.8/0.2 acetonitrile/methanol/acetic acid.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 053

Ammonia Procedure

Samples were analyzed using a meter (capable of reading millivolts) equipped with an ion-selective electrode designed to measure the amount of ammonia as N in water and wastewater samples. Samples were analyzed without a pre-distillation step and were evaluated against a standard curve obtained from standards prepared from a stock solution of standard Ammonium Chloride. Samples and standards were analyzed by immersing the electrode in 100 mL of sample to which 1 mL of 10N sodium hydroxide was added. All standards and samples were continuously stirred at a slow rate with a magnetic stirrer during measurement readings.

Samples were analyzed on an Orion meter, research microprocessor ionalyzer model 901, equipped with a Corning Combination Ammonia Electrode. The detection limit of the electrode is 0.14 mg/L and has a working range up to 140 mg/L. The amount of ammonia as N for each sample was reported as ammonia by multiplying by a conversion factor of 17/14.

Reference:

Method 4500-NH3 F. Ammonia Selective Electrode Method: Standard Methods for the Examination of Water and Wastewater (1992) 18th Edition, pp 4-81 to 4-82, Edited by Arnold E. Greenberg, Lenore S. Clesceri and Andrew D. Eaton.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 054

Nitrite and Nitrate Procedure

Samples were analyzed by ion chromatography using suppressed conductivity detection. Equipment includes a Dionex Ion Chromatograph, model DX-100, an autosampler, an advanced computer interface, and a computer operating with Windows 95 and employing the PeakNet (version 5.1) Ion Chromatography software by Dionex. Columns used are the IonPac AS4A-SC analytical and AG4A-SC guard

columns. Eluent used is 1.8 mM sodium carbonate/1.7 mM sodium bicarbonate with a flow rate of 2.00 mL/minute.

Detection is achieved with suppressed conductivity using the ASRS-ULTRA 4 mm self-regenerating suppressor by Dionex. Normal operating conditions yield detection limits of 0.1 g/L.

Samples were evaluated against a 4-point standard curve generated from multi-ion standards (20, 10, 5 and 1 mg/L) containing each of the following: Fluoride, Chloride, Nitrite, Nitrate, Phosphate and Sulfate. Upon calibration, samples were analyzed and diluted accordingly to fall within the calibration range.

References:

EPA Method 300.0: The Determination of Inorganic Anions in Water by Ion Chromatography (1991), U.S. Environmental Protection Agency, Environmental Monitoring and Systems Laboratory, Cincinnati, OH, Authors: John D. Pfaff, Carol A. Brockhoff and James W. O'Dell.

Method 4110 B. Ion Chromatography with Chemical Suppression of Eluent Conductivity: tandard Methods for the Examination of Water and Wastewater (1992) 18th Edition, pp 4-1 to 4-5, Edited by Arnold E. Greenberg, Lenore S. Clesceri and Andrew D. Eaton.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 056

Analysis For Organophosphate In Soil and Sediment, GPC cleanup.

Weigh a ten-gram soil or sediment samples into a PRQ (SOP 1.105) centrifuge bottle. Add 50-ml acetone, and shake for 1 minute every 15 minutes, for 1-1/2 hours. Add 50-ml petroleum ether and repeat the shaking. Centrifuge the sample and decant the liquid into a 500-ml separatory funnel containing 200-ml PRQ water (SOP 1.255) and 15-ml PRQ saturated sodium chloride SOP 1.255). Extract the sample again for 1 minute with 50 ml 1:1 acetone/petroleum ether (SOP 1.255), centrifuge and decant the liquid into the separatory funnel. The separatory funnel is shaken vigorously for one minute and the layers allowed to separate. The pet ether is removed, and the water fraction extracted again with 50-ml pet ether. The combined pet ether is washed twice with 50-ml water and concentrated by Kuderna-Danish (SOP 1.261) to appropriate volume. The sample is dissolved in 4 ml of methylene chloride and 2 ml (5 gram equalivant) injected into a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A).

The extract is concentrated, and exchanged into hexane. The sample is shot on a Varian 3400 GC with a 30M megabore column and a Thermionic Specific Detector (N and P detector) and on a Varian 3600 GC with dual 30M megabore DB-608 and DB-5 columns and electron capture etectors.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 057

Analysis for TPH (total petroleum hydrocarbon) in Soil and Sediment

1. Weigh 20 -g soil into a PRQ centrifuge bottle (SOP 1.105). (Add 10 ml PRQ H2O (SOP 1.255) to dry samples) Adjust pH to < 2 with PRQ 12N sulfuric acid (SOP 1.255). Add 50 -ml acetone (SOP 1.255) and shake 6 times over a one and one-half hour period (about every 15-min). Add 50 ml of a 1:1 petroleum ether (SOP 1.81)/ ethyl ether (SOP 1.255) mixture and repeat shaking. Centrifuge (SOP 1.100) and decant liquid into a 500 -ml separatory funnel containing 200 -ml PRQ water and 15-ml PRQ saturated NaCl.

Re-extract soil by shaking one minute with 50 ml 1:1 petroleum ether: ethyl ether (may need to add 10 ml H2O & adjust to pH < 2), then centrifuge and decant liquid into separatory funnel.

2. Using PRQ 6N KOH (SOP 1.255), adjust contents of separatory funnel to pH >12. Shake vigorously 2 min, then allow to stand 30 min. with intermittent shaking. Drain water layer and reserve ether layer. Re-extract H2O layer with 100 ml 1:1 petroleum ether: ethyl ether. Cap and reserve combined ether extracts. (This contains Total Petroleum Hydrocarbons.)

3. Concentrate the extract with Kuderna-Danish evaporators (SOP 1.261) and reduce volume to adequate size for column clean up.

4. Column Clean-up: The concentrated extract for TPH determination representing 20 grams of sample is transferred to a 20 gram 1% deactivated silica gel (SOP 1.255) column (silica gel is added to the column in a pet ether slurry) topped with five grams neutral alumina (SOP 1.255). TPH residues are fractionated by eluting the aliphatics from the column with 100 ml petroleum ether. The extract is concentrated and a known aliquot is air dried in an aluminum weighing dish to determine the weight of oil.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 059

Analysis for Paraquat and Diquat in Water

A known volume of water is made acidic by the addition of 15ml concentrated (18M) sulfuric acid. The entire sample is percolated through a cation - exchange resin (Dowex-50W, 3.5g) that retains the Paraquat and Diquat. The herbicides are eluted with saturated ammonium chloride at a flow rate of one ml/min. Fifty mls of eluent are collected in a 50ml volumetric flask.

20 ml of the eluent is pipetted into a 25ml test tube. 4ml of 0.2% sodium dithionate solution is added to develop the color. The paraquat and diquat are determined by using a HP 8453 UV-Vis spectrophotometer which is operated in the 2nd derivative. Paraquat is determined at 396 nm and 1.2 ug/ml of Paraquat and 0.001 to 0.2 ug/ml for Diquat.

Calculations:

- Volume of eluent(ml) X concentration in eluent(ug/ml) = ppm
- Original Volume of sample(ml)

Reference:

Modification of: The Determination of Residues of Paraquat in Crops -- A spectrophotometric method; Company method from ICI Plant Protection Division, Residue Analytical method No. 1B.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 060

Analysis for Aliphatic and Polynuclear Aromatic Hydrocarbons in Animal Tissue

Extraction

A sample of appropriate size (i.e. 5 grams animal tissue, 2 grams adipose, 5 grams eggs) is digested for 24 hours in 25 ml 6N aqueous potassium hydroxide (SOP 1.255). Shake once an hour at least 7 times during the digestion process. Cool digestate thoroughly in an ice bath and carefully neutralize with 15 ml glacial acetic acid, agitate slowly to cool. Add 100 ml PRQ H20 to a 500-ml separatory funnel. Pour sample into a separatory funnel, rinse 3 times with a total of 100 ml PRQ H20, then rinse the sample bottle with 25 ml CH2Cl2. Shake separatory funnel vigorously for 1 minute. Let the sample settle, then drain the CH2Cl2 into a PRQed 250-ml wide

mouth French square bottle. Centrifuge if necessary. Add 25 ml CH2Cl2 to the separatory funnel again; shake vigorously for 1 minute and drain. Repeat this procedure once more, so that 75 ml of extract has been collected in the sample jar.

Backwash

Discard the H20 in the separatory funnel, rinse funnel with tap water, then with PRQ H20. Add 100 ml PRQ H20 to the separatory funnel, and then add 50 ml of 6N potassium hydroxide to the separatory funnel resulting in 150 ml of 2N KOH. Shake to mix. Add the 75 ml of extract to the funnel, rinsing the collection jar 3 times with CH2Cl2. Shake the sample gently for 1 minute. After settling, drain the sample and add 25 ml CH2Cl2 to the funnel. Shake gently for 1 minute, drain again, and repeat this procedure twice more, until the expected 150 ml of sample extract has been collected in the sample jar.

KD

Pour sample from the jar to the KD flask and rinse 3 times using hexane, then add 10 ml hexane to each flask. KD the samples.

N-evap

Evaporate the samples gently, until the solvent level reaches approximately 3mls. The samples should be in hexane at this point. If you smell methylene chloride in the samples, add 5 ml more of hexane, mix and blow down again to about 3 ml.

Silica Gel

PRQ silica gel columns. Pack with 20 grams 1% deactivated silica gel by making a slurry with 50-ml petroleum ether and washing the silica gel into the column with an additional 50ml pet ether. Top each column with 5-ml of neutral alumina (SOP 1.255). Rinse the columns with 100 ml more Pet Ether if aliphatic fraction is to be analyzed. Transfer the sample to the column with a Pasteur pepette, rinsing the tube 3 times with petroleum ether. Pour 100mls of petroleum ether into the column, adjusting the drip rate to 5mls a minute (SG-1, aliphatics). When the first fraction is complete, change the sample bottle and pour 100-ml of 40% CH2Cl2/PE (methylene chloride/petroleum ether) onto the column. Just before the column goes dry, add 50-ml of CH2Cl2 and collect in the same sample bottle as 40% (SG- 2, PAH).

Concentration

SG-1 is transferred to the KD flask using petroleum ether to rinse the collection jars. SG-2 is transferred to the KD flask using CH2Cl2. The samples are concentrated to about 5 ml. The samples are further concentrated by n-evap. The samples are transferred to calibrated tubes so that the final concentration is 5g/ml using the above solvents. Do NOT let these samples go dry.

Analysis

Both fractions are analyzed using a Varian Saturn 2000 GC/MS ion trap with a 30M DB-5MS or equivalent 0.25mm capillary column. Methylated aromatics are calculated against the unsubstituted parent compound, or against a named methylated compound in this list. C1-naphthalenes are the sum of the 2-methyl and 1-methyl naphthalene. C2-naphthalenes are calculated against 2,6-dimethylnaphthalene. C3 and C4-naphthalenes are calculated against 2,3,5-trimethylnaphthalene. Methylated phenanthrenes are calculated against 1-methyl phenanthrene. C1-fluoranthenes + C1-pyrenes are calculated against fluoranthene. All other methylated aromatics are calculated against their unsubstituted parent compound.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 061

Analysis for Atrazine metabolites in Water

1. Record sample volume and pH. Add 1500 ml of sample to a 2000-ml separatory funnel. Add 200 grams of sodium chloride and 100 mls phosphate buffer to the funnel and shake to mix.

2. Adjust to pH 8 by adding PRQ 6N KOH (SOP 1.255) or 6N sulfuric acid to sample, and shake vigorously 30 sec. Allow sample to stand one hour at pH 8. Rinse the sample container with 50-ml CH_2Cl_2 (SOP 1.255) and add this to the funnel. Pour the remainder of the sample back into the sample container and rinse the graduated cylinder 2 X 50 ml CH_2Cl_2.

3. Shake 2 min. with periodic venting. Drain CH_2Cl_2 through a funnel of CH_2Cl_2 washed sodium sulfate into a 1000 ml French Square bottle if there is no emulsion. Otherwise, drain lower layer into centrifuge bottle and centrifuge to separate layers. Repeat 2 X 100 ml CH_2Cl_2. This combined extract contains the compounds of interest.

4. Concentrate the extract by turbo-vap.

5. Exchange sample extract into hexane and adjust to 2 ml.

6. Shoot the extract on two columns, a 60m 0.25mm x 0.25u DB-XLB and a 60m 0.25mm x 0.25u DB-5 with electron capture detector.

- Phosphate Buffer: 29.6 ml 0.1N hydrochloric acid & 50 ml 0.1M dipotassium phosphate
  - 0.1N hydrochloric acid = 2.5 ml HCl in 296 ml H_2O
  - 0.1M dipotassium phosphate = 8.7g dipotassium phosphate to 500 ml H_2O (This makes approximately 800 ml buffer.

References:

- E.P.A. Method 507
- E.P.A. Method 508
- E.P.A. National Survey of Pesticides in Drinking Water Wells (11/90) Method 4

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 062

Analysis of Water for Glyphosate

The samples were analyzed using the method of Moye,A. et.al., Validation of An Analytical Residue Method for Glyphosate and metabolite: An Interlaboratory Study. J. Agri. & Food Chem., 1986. 34:955-960.

Measure 100 ml water

Adjust pH to 2.0 plus-minus 0.4 with 6N HCL Add sample to the following chromatography columns: (2 columns)

1. Pour sample through a Chelex column. Elute with 15ml 6N HCL. Collect HCL, and make up to 25ml with concentrated HCL and add to next column.

2. AG-1X8 column , catch sample then elute with 6N HCL (2ml then 8ml) for a total of 35 ml. Concentrate by rotovap and re-dissolve in 10ml water

Inject on HPLC

Conditions:

- Fluorescence detector Emission wavelength 455 Excitation wavelength 339
- Mobile Phase: 0.05% Phosphoric acid in Deionized Water 1.0 ml/min
- Oxidative solution 0.3 ml/min
- Derivatization solution (OPA-Borate) 0.4ml/min
- Post-column derivatization at 38 degrees C.
- Waters IC-Pac Ion-Exclusion 7 micrometers 7.8x150mm column.

If Glyphosate is found, the confirmation column a Hamilton PRP X400: 7 micrometer 4. 1X250mm column

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 063

Toxicity Characteristic Leaching Procedure (TCLP)

Summary: Solid waste is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase.

Apparatus: Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 +-2 rpm.

Bottle extraction Vessel: For a nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel. It is recommended that borosilicate glass bottles be used.

Filtration device: Any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Wastes should be filtered using positive pressure filtration. Filters should have an effective pore size of 0.6 to 0.8 um.

Reagents: Extraction fluid.

Add 5.7 ml of glacial acetic acid to 500 ml of reagent water, add 64.3 ml of 1N sodium hydroxide and dilute to 1 liter. The pH of this fluid is 4.93 +-0.05.

Procedure:

Weigh out a subsample of the waste (100 gram minimum). Transfer the solid material into the extractor bottle. Slowly add 20 times the weight of sample in extraction fluid (2000 ml for a 100 gram sample) to the extractor vessel. Close the extractor bottle tightly, secure in the rotary agitation device, and rotate at 30 rpm for 18 +- 2 hours at an ambient temperature of 23 C. Following the extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a glass fiber filter in a pressure filter. The liquid obtained is the TCLP extract.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 064

Analysis for Organophosphate Pesticides and Triazine Herbicides in Animal Tissue using ASE/GPC

Four-gram samples or two-gram fat samples are weighed into a 150-ml beaker then thoroughly mixed with 4-grams of Hydromatrix. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. The sample can be stored in a desiccator over night. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 22-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spa tula, and beaker with no more than 4-ml total of hexane and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 100% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 1:1 hexane: acetone (SOP 1.255).

The extract is concentrated by Turbovap; transferred to a tarred test tube through a Pasteur pipette containing sodium sulfate using methylene chloride, and further concentrated to dryness for lipid determination (SOP 1.264). The weighed lipid sample is dissolved in 4 ml of methylene chloride and the fat removed by injecting 2 ml on a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into hexane.

Quantification is by Varian 3400 GC with a 30M RTX-200 megabore column and a TSD (thermionic specific detector), to detect N and P containing compounds.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 065

Analysis for Pirate and Metabolites Tissue using ASE/GPC.

Ten-gram samples are weighed into a 150-ml beaker then thoroughly mixed with 6-grams of Hydromatrix. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 33-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 4-ml total of hexane and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min

static cycles, 60% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 1:1 hexane: acetone (SOP 1.255).

The extract is concentrated by Turbovap; transferred to a tarred test tube through a Pasteur pipette containing sodium sulfate using methylene chloride, and further concentrated to dryness for lipid determination (SOP 1.264). The weighed lipid sample is dissolved in 4 ml of methylene chloride and the fat removed by injecting 2 ml on a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into hexane. A drop of methylene chloride is added to the sample.

The sample is transferred to a 300 ml glass chromatographic column (Kontes # 420280-0242) containing 20 grams of Florisil (SOP 1.255) topped with 1 cm of sodium sulfate and the sample tube rinsed three times with about 2 ml pet ether. The column is eluted with 200 ml 6% diethyl ether (SOP 1.255)/94% petroleum ether (Fraction I) followed by 250 ml 15% diethyl ether/85% petroleum ether (Fraction II). The diethyl ether used in this analysis contains 2 % ethanol (SOP 1.255). Fractions II contains the Pirate and metabolites. This fraction is concentrated to an appropriate volume for quantification of residues.

GC determinations were run on a Varian 3400 GC with a Varian Star Data System and a Varian 8200 Autosampler. The GC is equipped with a 60m DB-XLB (0.25u film thickness) capillary column. All compounds were calculated using a three point standard curve forced through the origin using external standards (SOP 1.267).

- Pirate, also known as chlorfenapyr, CAS # 122453-73-0

Metabolites

- CL303267, CAS# 122454-23-3
- CL303268, CAS# 122454-29-9

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 066

Analysis for Organochlorine Pesticides and PCBs in Animal Tissue using ASE/GPC.

Two-gram samples are weighed into a 150-ml beaker then thoroughly mixed with 4-grams of Hydromatrix. Stir the sample with a PRQ spatula until the mixture becomes a flowable powder and leaves the sides of the beaker clean. The sample can be stored in a desiccator over night. Pour the sample through a PRQ powder funnel into a PRQ Accelerated Solvent Extractor (ASE) 22-ml cell with a 2-cm glass fiber filter in the bottom cell cap. Tap the ASE cell to settle the sample and add more Hydromatrix

if needed to fill the cell. Rinse the funnel, spatula, and beaker with no more than 4-ml total of methylene chloride and add the rinses to the cell. Place the top cap on the cell and tighten both caps hand tight. Record the cell number, the position number, and the number of the sample placed in the cell in the ASE logbook as the cell is placed in the ASE for extraction. Place the sample number on the collection vial. Operate the ASE according to SOP 1.260 (EPA method 3545) with the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 100% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 100% methylene chloride. (SOP 1.255).

The extract is concentrated by Turbovap to about 20 ml. The water is removed by pouring the sample through a Horizon drydisk apparatus. The extract is transferred to a tarred test tube using methylene chloride, and further concentrated to dryness for lipid determination (SOP 1.264). The weighed lipid sample is dissolved in 4 ml of methylene chloride and the fat removed by injecting 2 ml on a Waters high pressure GPC (Gel Permeation Chromatography)(EPA Method 3640A). The fraction is concentrated by Turbovap and then exchanged into hexane.

The sample is transferred to a 300 ml glass chromatographic column (Kontes # 420280-0242) containing 20 grams of Florisil (SOP 1.255) topped with 1 cm of sodium sulfate and the sample tube rinsed three times with about 2 ml pet ether. The column is eluted with 200 ml 6% diethyl ether (SOP 1.255)/94% petroleum ether (Fraction I) followed by 200 ml 15% diethyl ether/85% petroleum ether (Fraction II). If Endosulfan II and/or Endosulfan Sulfate analysis is required, then 200 ml of 50% diethyl ether/petroleum ether (Fraction III) is required. The diethyl ether used in this analysis contains 2% ethanol (SOP 1.255). Fractions II and III are concentrated to an appropriate volume for quantification of residues by megabore column electron capture gas chromatography (SOP 1.265)(DB-608 and DB-5 dual columns) Dieldrin and Endrin are in Fraction II, and part of the delta BHC.

Fraction I is concentrated to 5 ml and transferred to a Silicic acid (SOP 1.255) chromatographic column (custom columns 1 cm OD x 40 cm with a 100 ml reservoir on top, Tutor Glass) for additional cleanup required for separation of PCBs from other organochlorines. Five grams of hot silicic acid is put into the column, which already has a glass wool plug and about 3-mm sodium sulfate in the bottom. The Silicic acid is topped with 10-mm sodium sulfate and prewashed with 10-ml hexane. Three fractions are eluted from the silicic acid column. The sample in 5-ml solvent is added to the column and rinsed into the column with 3,1,1-ml hexane. Then the sample is eluted with 20-ml pet ether (fraction SAI). Fraction SAII is 150-ml pet ether. Fraction SAIII is 20 ml of a mixed solvent consisting of 1 part acetonitrile, 19 parts hexane and 80 parts methylene chloride (SOP 1.255).

Each is concentrated to appropriate volume for quantification of residues by megabore column, electron capture gas chromatography. HCB and Mirex are in SAI. PCBs are found in SAII. The rest of the compounds are in SAIII.

GC determinations were run on a Varian 3600 GC with a Varian Star Data System and a Varian 8200 Autosampler. All GCs were equipped with dual DB-608 (0.83u

film thickness, J & W Scientific # 125-1730) and DB-5 (1.5u film thickness, J & W Scientific # 125-5032) 30M megabore columns. All compounds were calculated using a three point standard curve forced through the origin using external standards (SOP 1.267).

PCB's were determined by shooting SAII fractions on a Varian 3400 GC with a Varian Star Data System and a Varian 8200 Autosampler. This GC is equipped with a 60M DB-5 0.25 ID capillary column. Another 3400 equipped with a 60M DB-XLB 0.25 ID capillary column is also used as a second system for PCB's. The compounds were calculated in the following manner. All the aroclor standards are at 0.5 ng/ul with one ul shot.

Starting with Aroclor 1260, 4 peaks that are unique to this mixture are located. The areas of the standards are summed and the same peaks located in the sample and also summed. Aroclor 1260 is calculated by the following formula to obtain PPM 1260.

(Area sample) (weight of std shot in ng)
(Area 1260 std) (basis shot in mg)

Aroclor 1254 is calculated by locating the major peaks in the mixture that are normally found in samples. The areas of these peaks are summed. Because some of this area comes from Aroclor 1260 and not all from Aroclor 1254, the contribution from the 1260 has to be subtracted from the total area. Aroclor 1254 is calculated by using the formula:

$$\frac{\text{(Area sample)} - \dfrac{\text{(PPM 1260)(Basis shot in mg)(area from 1260)}}{\text{ng 1260 std}}}{\text{(Area 1254 std)(Basis shot in mg)}} \text{(ng 1254 std)}$$

Results are in PPM.

Aroclor 1248 is calculated in a similar fashion, subtracting the contribution from 1254 in the 1248. Aroclor 1242 is calculated using the area of five early peaks.

Total PCBs are calculated by adding the sum of Aroclor 1242, 1248, 1254, and 1260.

Basis = (weight of the sample mg/final volume of sample ul)(ul of sample shot)

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 067

Analysis for Organochlorine Pesticides and PCBs, Aliphatic and Polynuclear Aromatic Hydrocarbons in Soil and Sediment

A 10-gram soil or sediment sample is weighed into a PRQ (SOP 1.105) centrifuge bottle. If the sample is less than 10% moisture, then 10- ml water (SOP 1.255) is added. Fifty-ml acetone (SOP 1.255) is added and the sample shaken well six times over a ninety-minute period (about every 15 minutes). Fifty mls petroleum ether (SOP 1.81) is added to the sample and the shaking repeated. The sample is centrifuged (SOP 1.100) and the liquid decanted into a 500-ml separatory funnel containing 200 ml PRQ water and 15 ml saturated sodium chloride solution (SOP 1.255). Fifty mls of a 1:1 acetone/pet ether mixture is added to the sample and it is shaken, centrifuged, and the liquid added to the separatory funnel. The separatory funnel is shaken vigorously for two minutes and the layers allowed to separate. The pet ether is removed, and the water fraction extracted again with 50-ml pet ether. The combined pet ether is washed twice with water and concentrated by Kuderna-Danish (SOP 1.261) to appropriate volume. An aliquot of the concentrated extract representing 2 grams of sample is transferred to a 1.6 gram Florisil (SOP 1.255) mini-column topped with 1.6 grams sodium sulfate (SOP 1.255) for pesticide determination. A Kontes #7 column is used for this. Residues are eluted from the column in two elution fractions. Fraction I consists of 12 milliliters hexane (SOP 1.255) followed by 12 milliliters of 1% methanol (SOP 1.255) in hexane, and Fraction II consists of an additional 24 milliliters of 1% methanol in hexane. If additional clean up is required to separate PCBs from other organochlorines in Fraction I, further chromatography on a Silicic acid (SOP 1.255) column is performed. Quantification of residues in the two Florisil fractions and three Silicic acid fractions is by dual megabore column (DB-608 and DB-5), electron capture gas chromatography (SOP 1.265).

A second aliquot of the concentrated extract for hydrocarbon determination representing 8 grams of sample is transferred to a 20 gram 1% deactivated silica gel (SOP 1.255) column (silica gel is added to the column in a pet ether slurry) topped with five grams neutral alumina (SOP 1.255). Aliphatic and polynuclear aromatic hydrocarbon residues are fractionated by eluting aliphatics from the column with 100 ml petroleum ether (Fraction I) followed by elution of aromatics using first, 100 ml 40% methylene chloride (SOP 1.255)/60% petroleum ether, then 50 ml methylene chloride (Combined elutes, Fraction II). Both fractions are concentrated, reconst chromatography - mass spec (SOP 1.276) using a Varian Saturn 2000 ion trap mass spec with a 30M DB-5MS capillary column.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 068

Analysis for Organochlorine Pesticides and PCBs in Soil and Sediment, ASE, Dridisk.

Extract a two-gram dry weight samples using a Dionex Accelerated Solvent

Extractor (ASE) with 22-ml cells under the following extraction conditions: 5-min heating cycle, 2x2-min static cycles, 100% solvent flush, 60 sec purge cycle, 100 degrees C @ 1500psi, 100% methylene chloride.

Concentrate the extract by Turbovap to about 10 ml. Remove the water by pouring the sample through a Horizon drydisk apparatus. Exchange the sample into hexane and transfer to a 7mm ID column containing 1.6 gram Florisil topped with 1.6 grams sodium sulfate.

Elute the residues from the column in two fractions. Fraction I consists of 12 milliliters hexane followed by 12 milliliters of 1% methanol in hexane, and Fraction II consists of an additional 24 milliliters of 1% methanol in hexane. If additional cleanup is required to separate PCBs from other organochlorines in Fraction I, further chromatography on a Silicic acid column is performed. Adjust the samples to an appropriate volume. Quantification of residues in the two Florisil fractions and three Silicic acid fractions is by dual megabore column (DB-608 and DB-5), electron capture gas chromatography (SOP 1.265).

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 069

Imadacloprid in Tissue

ASE

- 100% methylene chloride
- 22 ml cells-100% flush
- 5 min heat 2X 2 min static cycle
- 100 degrees C
- 4 grams Hydromatrix

PROCEDURE

- Weigh 2g of homogenized tissue into beaker.
- Add 4g of Hydromatrix and mix to a flowable powder.
- Load ASE cells and extract.
- Turbovap to ~ 15 mls at 40 degrees C

Dry Disk

- Turbovap and transfer to tube with methylenechloride
- N-evap to dryness and pipet 2 mls of 20/80 acetonitrile/water into tube
- Filter into HPLC vial and inject

Reference

S. Baskaran, R.S. Kookana, R. Naidu, J. Chromatography, A, 787 (1997) 271-275Bayer-Environmental Fate Group, Analytical method

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 070

Imidacloprid in Soil and Sediment

Solvent

- 80/20 acetonitrile/water
- 20/80 acetonitrile/water
- Glassware and Other Materials
- Soil shake bottle
- Thin tipped funnel

ASE tube

- 10 ml calibrated tube
- Pipette
- Glass wool

Procedure

- Weigh 10g soil into shake bottle
- Add 20ml 80/20 acetonitrile/water
- Shake 15 min
- Centrifuge 5 min
- Decant thru funnel plugged with glass wool into ASE tube
- Put extract in Turbo and concentrate
- Repeat 2 more time putting extract in same ASE tube Put concentrated extract in10 ml tube and adjust with 20/80 acetonitrile/water
- Shoot on HPLC

Reference

S. Baskaran, R.S. Kookana, R. Naidu, J. Chromatography, A, 787 (1997) 271-275Bayer-Environmental Fate Group, Analytical method

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 071

Anticoagulant Rodenticides in Blood and Liver

EQUIPMENT

1) HPLC with absorbance and fluorescence detectors

2) Tissue grinder (e.g., Tissue-mizer)

3) Centrifuge

4) Silica and C18 Sep-Paks (Waters)

5) Disposable syringes, Monoject

6) Filter papers, ½ in, Schleicher and Schuell (S & S)

7) C18 HPLC column, 4.6 mm X 15 cm; e.g. Vydac or Luna

8) Glass Luer-Lok syringe, 25 or 50 ml

9) Syringe adapters (e.g., Baker)

10) 16 X 100 test tubes

11) Large test tubes (e.g., 25 X 150)

12) HPLC sample vials

REAGENTS AND CHEMICALS

1) Standards (from EPA or other sources): HYDROXYCOUMARINS: Racumin (coumatetralyl), warfarin, bromodiolone, difenacoum and brodifacoum; INDANEDIONES: pival (Pindone), diphacinone and chlorophacinone and potentially others;

2) Acetic acid, ACS grade

3) Phosphoric acid, ACS grade

4) Aluminum oxide, basic (50-200 mcm), Brockman activity grade I

5) Acetonitrile, HPLC grade

6) Methanol, HPLC grade (mobile phase A)

7) Methylene chloride, HPLC grade

8) Florisil, 60-100 mesh

9) Potassium phosphate, ACS grade

10) Triethylamine

11) Formic acid (0.25%) in methylene chloride

12) Distilled water

13) Helium

14) Triethylamine

15) Acetic acid

16) Ammonium acetate

17) Mobile phase B, for hydroxycoumarins: 752 ml water, 2 ml triethylamine, 2 ml acetic acid and 2.89 g ammonium acetate, all mixed thoroughly; plus 250 ml acetonitrile; degas with helium

18) 0.05 M phosphate buffer, pH = 6.9

19) Mobile phase B for indanediones: 500 ml phosphate buffer (0.05 M, pH = 6.9) plus 500 ml methanol; degas with helium

PREPARATION OF STANDARDS

1) For each compound, prepare 1,000 ppm stock solutions in acetonitrile

2) Prepare dilutions as necessary for standard curves, spiking, etc.

CONTROL SAMPLES

1) Prepare duplicate extractions of serum or liver and spike to 5 ppm with the appropriate stock

2) Extract as regular samples

PREPARATION OF CLEAN UP COLUMNS

1) Aluminum oxide/ Florisil clean-up column: Place 1 S&S filter in a 6 ml syringe barrel. Add 1 ml (ca.0.5 g) of Florisil, 1 S&S filter and then 2 ml (ca. 2 g) basic aluminum oxide and 2 S&S filters Before use wash with 4 ml each of methanol, water and acetonitrile, in that order.

2) Aluminum oxide/ C18 clean-up column: Place 1 S&S filter in a 6 ml syringe, add 3 g (ca. 3 ml) basic Alumina and 2 S&S filters.

Connect a C18 Sep-Pak to the outlet. Before use wash with 4 ml each methanol, water and acetonitrile, in that order.

SERUM EXTRACTION

1) Place 2 ml serum in test tube

2) Add 4 ml acetonitrile

3) Vortex

4) Centrifuge at medium speed for 10 min

5) Decant supernatant into 2nd test tube

6) Wash protein precipitate with additional 4 ml acetonitrile

7) Combine wash with 1st supernatant

8) Attach glass syringe to a washed Florisil/ aluminum oxide clean-up column with an adapter.

9) Pass extract through clean-up column, collecting eluate in a large test tube

10) Wash clean-up column with 4 ml acetonitrile, combining wash with 1st eluate

11) Dry eluates at ca. 55 C under nitrogen

12) Dissolve residue in 0.15 ml methanol and add 0.05 ml water

13) Transfer sample extract to HPLC sample vial

14) Proceed to HPLC analysis of hydroxycoumarins or indanediones

LIVER EXTRACTION: HYDROXYCOUMARINS

1) Weigh 2 g liver into a large test tube

2) Add 6 ml acetonitrile

3) Homogenize with tissue grinder

4) Centrifuge at medium speed

5) Decant supernatant into a 2nd large test tube

6) Wash pellet with additional 6 ml acetonitrile

7) Combine wash with initial supernatant

8) Attach washed aluminum oxide/ C18 clean-up column to a glass syringe with an adapter

9) Pass extract through the clean-up column, collecting eluate in a 2nd large test tube

10) Wash clean-up column with additional 6 ml acetonitrile, combining eluates

11) Dry at ca. 55 C under nitrogen

12) Dissolve residue in 0.15 ml methanol and add 0.05 ml water

13) Transfer extract to an HPLC vial

14) Proceed to HPLC of hydroxycoumarins

ADDITIONAL LIVER EXTRACTION CLEAN-UP FOR INDANEDIONES

1) Attach glass syringe to silica Sep-Pak

2) Apply 0.1 ml liver extract

3) Wash Sep-Pak with 3 ml methylene chloride, discarding wash

4) Elute indanediones into a large test tube with 30 ml 0.25% formic acid in methylene chloride

5) Dry at ca. 55 C under nitrogen

6) Dissolve residue in 0.15 ml methanol and add 0.05 ml water

7) Transfer extract to an HPLC vial

8) Proceed to HPLC of indanediones

HPLC OF HYDROXYCOUMARINS (FLUORESCENCE DETECTION)

1) Equilibrate HPLC column with 30% A (methanol) and 70% B (for hydroxycoumarins), total flow rate = 1.5 ml/ min

2) Fluorescence detection: EX = 318 nm and EM = 390 nm

3) Inject 0.01 ml or more of extract or standard

4) Ramp rate = 2.5 %/ min

5) Integrate area under peaks

6) Proceed to calculations

HPLC OF INDANEDIONES

1) Equilibrate C18 column with 55% A and 45% B

2) Absorbance detection at 285 nm

3) Inject 25 ml extract or standard

4) Ramp rate = 2.5 %/ min

5) Integrate area under peaks

6) Proceed to calculations: Method Code 071

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 072

Analysis of Water for Imazapic

Record sample volume and pH. Add 300 ml of sample to a 500-ml separatory funnel. Add 25 grams salt and 15 ml phosphate buffer. Adjust pH to less than 2 with drop wise addition of PRQ 18N H 2SO 4 (About 8 drops) (SOP 1.255).

Extract water with 50 ml methylene chloride by shaking two minutes. Drain the lower organic layer from the separatory funnel through acid sodium sulfate into a 200-ml turbovap cup. Repeat 2 x 50 ml methylene chloride. This combined extract contains imazapic.

Imazapic - Evaporate samples just to dryness. Reconstitute the samples in 20% acetonitrile / 80% water (HPLC grade) at a 100 ml/ml concentration. Samples are injected on a HPLC for quantitation.

HPLC conditions:

Waters 2690 HPLC system with a Waters 2487 UV detector
Column: Phenomenex Lichrosphere 5 RP-Select-B 4.0mm x 250mm
UV wavelength 240
Flow rate 1 ml/min

Gradient:	min	%A
	0	80
	14	60
	15	60
	16	80
	20	80

Mobile phase:

A Water (0.1% Acetic acid)
B Acetonitrile

This procedure was developed for water samples that had no sediment. Dirty water samples might require clean-up.

mazapic CAS # 104098-48-8

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 073

Analysis for TPH (total petroleum hydrocarbon) in Water

1. Mark the level of the water in the bottles. After extraction determine the volume of the samples.
2. Pour the entire sample into a separatory funnel. Rinse the sample bottle with 1:1 petroleum ether: ethyl ether using 100 ml of solvent per 500 ml of sample. Add to the separatory funnel. Shake 2 minutes. Drain water layer and reserve ether layer. Re-extract H 2O layer with 100 ml 1:1 petroleum ether: ethyl ether. Combine ether extracts. (This contains Total Petroleum Hydrocarbons.)
3. Concentrate the extract by KD and reduce volume to adequate size for column clean up (3 to 5 ml).
4. Column Clean-up: The concentrated extract is transferred to a 20 gram 1% deactivated silica gel column (silica gel is added to the column in a pet ether

slurry) topped with five grams neutral alumina. TPH residues are fractionated by eluting the aliphatics from the column with 100 ml petroleum ether. The extract is concentrated by KD and air dried in an aluminum weighing dish to determine the weight of oil (weighed to 5 places).

Spike: Use 1 ml of a 5 mg/ml TPH std in 1000 ml of water. Make a co-spike of 1 ml of the solution in a weighed Al dish.

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Lab Name: Mississippi State Chemical Laboratory (MSCL) Laboratory
Method Code 099

% Moisture

Performed at other laboratory.

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Research Triangle Institute (RTI) Laboratory Methods

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053	Determination of Sulfide as Hydrogen Sulfide By Gas Phase Molecular AS
054	Organotin
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056	AVS
057	Total Dissolved Solids

058	Total Suspended Solids
060	Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption (EPA Method 7473)

Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 001

Homogenization

Tissue samples are prehomogenized using a food processor. A portion of the tissue sample (or sediment) is then freeze dried for determination of moisture content and ground to 100 mesh with a mill.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 002

Preconcentration Digestion for Inductively Coupled Plasma Emission (ICP) Measurement

Using a CEM microwave oven, 0.5 g of freeze dried tissue is heated in a capped 120 mL Teflon vessel in the presence of 5 mL of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and 35 minutes at 450 watts. The vessel contents are then allowed to cool and the cap is removed and rinsed carefully with 3 ml of HNO3 adding the rinsings to the vessels contents. The uncapped vessel is then returned to the microwave oven and heated until the vessel contents are less than 1 mL in volume. The contents are carefully rinsed with laboratory pure water into a 5 ml glass volumetric vessel and made to volume with additional laboratory pure water. The flask contents are then immediately transferred to a clean plastic centrigure or auto sampler tube and centrifuged for 1 minute to precipitate the suspended matter. The sample is now ready for ICP analysis.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 003

Digest for ICP Measurement

Using a CEM microwave oven, 0.25 to 0.5 g. of freeze dried sample is heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and fifteen minutes at 450 watts. The residue is then diluted to 50 ml with D.I. water.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 004

Digestion for Graphite Furnace and Cold Vapor Atomic Absorption (GFAA) Measurement.

Using a CEM microwave oven, 0.25 to 0.5 g of freeze dried sample is heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and fifteen minutes at 450 watts. The residue is then diluted to 50 ml with laboratory pure water.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 005

Digestion for Hg Measurement by Cold Vapor Atomic Absorption (CVAA)

Some 0.25 to 0.5 g of tissue is refluxed for two hours in 10 ml HNO3 (Baker Instra-Analyzed) and diluted to 50 ml with 1% HCl.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 006

ICP

ICP measurements are made using a Leeman Labs Plasma Spec I sequential or ES2000 simultaneous spectrometer.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 007

Graphite Furnace Atomic Absorption (GFAA)

GFAA measurements are made using a Perkin-Elmer Zeeman 3030 or 4100ZL atomic absorption spectrometer.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 008

Cold Vapor Atomic Absorption (CVAA)

Hg measurements are conducted using SnC14 as the reducing agent. A Leeman PS200 Hg Analyzer is employed.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 009

Homogenization Following freeze drying samples

Homogenization Following freeze drying samples are ground to approximately 100 mesh using a mill.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 010

Digestion for Inductively Coupled Plasma Emission (ICP) Measurement

Some 0.25 to 0.5 g of sediment is placed in a 120 ml Teflon microwave vessel. One ml each of HC1, HF, and HC104, and 10 ml HNO3 are added to the vessel. The vessel is then capped according to the manufacturer's instructions and heated in a CEM microwave oven for two minutes at 120 watts, three minutes at 180 watts, and ten minutes at 600 watts. The resulting residue is diluted to 100 ml with 5% HCl. This solution is then filtered through Whatman 41 filter paper prior to ICP measurement. An HF resistance torch tip is used for these digests during the ICP measurement.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 011

Preconcentration Digestion for Inductively Coupled Plasma Emission (ICP) Measurement

Using a CEM microwave oven, 50 ml of water sample are heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and 35 minutes at 450 watts. The vessel contents are then allowed to cool and the cap is removed and rinsed carefully with 3 ml of HNO3 adding the rinse to the vessel contents. The uncapped vessel is then returned to the microwave oven and heated until the vessel contents are less than 1 ml in volume. The contents are carefully rinsed with laboratory pure water into

a 5 ml glass volumetric vessel and made to volume with additional laboratory pure water. The sample is now ready for ICP measurement.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 012

Digestion for Graphite Furnace Atomic Absorption (GFAA) Measurement

Using a CEM microwave oven, 50 ml of water sample are heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for fifteen minutes at 300 watts. The sample is then diluted to 50 ml with laboratory pure water.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 013

Digestion for Hg Measurement by Cold Vapor Atomic Absorption (CVAA)

Ten ml of water sample were refluxed for two hours in 10 ml HNO3 (Baker Instra-Analyzed) and diluted to 50 ml with 1% HCl.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 014

Summary of EPA Method 3010 for wastewaters, etc.

A mixture of HNO3 and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of HNO3 until the digestate is light in color or until its color is stabilized. After the digestate has been brought to a low volume, it is refluxed with HCl and brought up to volume. If the sample should go to dryness, it must be discarded and the sample reprepared.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 015

Toxicity Characteristic Leaching Procedure (TCLP) analyses are performed according to EPA Method 1311, 40 CFR, Chapter 1 (7-1-91 Edition) Part.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 016

Ultrasonic Extraction with IC Measurement.

0.5 g of sediment is sonicated in 30 mL of DI water for 30 minutes. The final volume of extract is adjusted to 50 mL with DI water. Anions are measured by ion chromatography.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 017

IC MEASUREMENT OF WATER SAMPLES

Anions in water are measured by ion chromatography.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 018

Determination of Radium - 226, 228

METHOD SUMMARY

This method is applicable to determination of radium-226, 228 activity in aqueous solutions.

Radium isotopes are collected by co-precipitation with barium and lead sulfates from an alkaline citrate solution, and purified by re- precipitation from EDTA solution. Both radium-226 and radium-228 are collected in this manner. For radium-226 determination, radium is co-precipitated on barium sulfate and counted by alpha spectrometry. The Ra-226 yield is determined through initial addition of a Ba-133 tracer. Ra-228 is also co-precipitated on barium sulfate, then after a 36-hour ingrowth of neptunium 228 from radium-228, the actinium-228 is carried with yttrium, purified, precipitated as yttrium oxalate and beta counted. The Ra-228 yield is determined gravimetrically from the yttrium oxalate.

INTERFERENCES

Samples containing barium in excess of 1 mg per aliquot may cause interference when counting the Ra-226 by alpha spectroscopy.

Performance studies of the EPA 904.0 method indicate that the presence of Sr-90 in the sample gives a positive bias to any measured radium- 228 activity because the Sr-90 is not completely separated from the Ac-228.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 019

Determination of Strontium-90

METHOD SUMMARY

This method is applicable to determination of strontium-90 activity in aqueous solutions and may be used in conjunction with sample preparation procedures which are designed to dissolve non-aqueous samples containing strontium-90 into an aqueous matrix.

Strontium-90 along with stable strontium carrier are precipitated from aqueous samples as insoluble carbonates. Interferences from calcium and other radionuclides are removed by one or more precipitations of the strontium carrier as strontium nitrate. Barium and radium are removed as chromate. The yttrium-90 daughter of strontium-90 is removed from the initial sample by a hydroxide precipitation step. The yttrium-90 daughter is permitted to grow in again and is then separated with stable yttrium carrier as hydroxide and finally precipitated as the oxalate and beta counted. The strontium-90 concentration is determined by the yttrium-90 activity.

INTERFERENCES

Samples that contain significant amounts of stable strontium may cause errors in the recovery of the added strontium carrier. Hard waters containing large quantities of calcium which will precipitate with the strontium in the initial carbonate precipitation may cause errors in the strontium recovery. Repeated precipitations of the strontium nitrate will minimize this interference but may not eliminate it.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 020

Total organic carbon (TOC)

Total organic carbon (TOC) analyses are performed according to EPA Method 9060 "Text Methods for Evaluating Solid Waste: Physical/Chemical Methods; SW-846 3rd Ed. 1986.

Measurement of the TOC as CO2 is performed by IR using a Dohrman DC-80 TOC analyzer. Analyses will be performed either in-house or though purchase order agreement with Chemical and Environmental Technology, Inc. (Gary, NC).

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 021

Analysis by Ion Selective Electrode.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 022

Cr(VI) by IC-PCR - Hexavalent Chromium

Cr(VI) by IC-PCR - Hexavalent Chromium is separated from other anions and cations by ion chromatography followed by post column reaction with diphenyl carbazide (DPC). The Cr(VI)-DPC complex is measured colorimetrically at 530 nm.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 023

Hexavalent Chromium

Hexavalent Chromium species are extracted from 10 grams of soil using 100 mL 0.1N NaOH for 30 minutes using ultrasonication. After filtering 100 mL the resulting mixture, the filterate is suitable for det ermination of Cr(VI) by ion chromatography (IC-PCR).

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 025

Method for Total Cyanide Analysis in Soil/Sediment Cyanide, Total (in Sediments)

Method 335.2 CLP.M (Titrimetric; Manual Spectrophotometric; Semi-Automated Spectrophotometric)

SCOPE AND APPLICATION

This method is applicable to the determination of cyanide in sediments and other solids. The detection limit is dependent upon the weight of sample taken for analysis.

SUMMARY OF METHOD

The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric filtration or colorimetrically.

In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone or pyridine- barbituric acid regent. The absorbance is read at 620 nm when using pyridine-pyrazolone and at 578 nm when using pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 030

Methods for Determination of Grain Size

Grain size analysis will be performed to determine the relative proportion of the different grain sizes of unconsolidated soils or sediments. The general classification of grain sizes includes gravel (grain size > 2mm), sand (grain size between 0.0625 mm and 22 mm), silt (grain size between 0.0039 mm and 0.0625 mm) and clay (grain size fractions will be dependent on the nature of the sample. If the sample contains very little silt and clay, it will be dried, weighed, and sieved through a stacked column of sieves of the desired mesh sizes with the bottom sieve having a mesh size of 0.0625 mm (U.S. Standard Sieve Mesh No. 230). The material caught in the collection pan will represent the size fraction <0.0625 mm or the silt-clay fraction. If the sample contains a significant silt-clay fraction, it will be moistened with a dispersing agent and wet-sieved. Again, the material caught in the collection pan represents the silt-clay fraction. Pipette analysis will be used to determine the relative proportion of silt to clay; the silt- clay fraction is dispersed in 1000 mL of distilled water containing dispersant. After the water and sample are vigorously

agitated, measured volumes will be pipetted from the container at specified time intervals at a measured withdrawal depth. Since settling velocity is proportional to the square of the particle diameter, the particle size diameter can be computed given the parameters of time and depth. The pipetted samples are then dried and weighed to determine the weight percent fraction of the total sample.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 035

Nitric Acid Digestion of Blood and Soft Tissues

Nitric Acid Digestion of Blood and Soft Tissues at 85 C Using a Water Bath Followed by Microwave Concentration

PURPOSE: This procedure is a wet chemical digestion procedure for biological samples using nitric acid. Digestion is accomplished using concentrated nitric acid and heating in a water bath. The resulting digest is suitable for the atomic spectroscopic determination of nitric acid soluble metals.

INSTRUMENT DESCRIPTION: See operator's manual for a complete and detailed description of the water bath (1). The basic instrument features are presented here for the water bath and digestion system.

1. Water bath - Model No. 1235 PC by VWR Company. Hz = 50/60; Volts 120 phase single. Watts 600, Serial No. 1100493 - Located in Room 220A, Building 6.

2. CEM MDS-2000 Microwave Digestion System.

PROCEDURE: The following procedure describes preparation of biological materials for the atomic spectroscopic determination of copper and other metals.

1. Precleaning of 15 mL centrifuge tubes. After the normal nitric acid wash and deionized water rinse of the centrifuge tubes used for digestion, soak each tube for 30 minutes with 25% nitric acid. Discard this solution and rinse three (3) times with deionized water.

2. Digestion of Biological Samples. An aliquot of wet tissue (0.1 grams to 1.5 grams) is weighed into a graduated 15 mL plastic centrifuge tube. Two (2) mL of concentrated nitric acid (Baker Instra-analyzed Reagent for trace metals analysis, 70.0-71.0% HNO3) are added to each tube. The tubes are capped tightly and are placed into plastic racks and the racks are placed in the water bath at a water temperature of 85 C + 2 C for 45 minutes. Be sure that the bath water level is at least up to the level of the sample digestion solutions in each centrifuge tube. Cool and remove the caps, add 1 mL of hydrogen peroxide and reheat for an additional 15 minutes. Cool and place in a CEM MDS-2000 microwave oven. With caps

removed, heat the rack of samples at 20-40% power (120-240 watts) CAREFULLY until 0.5 mL of residue remains. All samples are then brought up to the 5 mL mark on the centrifuge tubes with deionized water and mixed well. If other measurements are needed the final volume may need to be increased. Documentany other volumes used on the digestion sheet.

3. Calibration. At least monthly, monitor the bath temperature with an NIST traceable thermometer that reads between approximately 20 and 100 C. The temperature should be within 2 C of the temperature display on the bath. If not, adjust the bath thermostat until 85 + 2 C is reached, according to the operators' manual.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 037

FAT IN FOODS CHLOROFORM-METHENOL EXTRACTION METHOD

Method is applicable to composite foods and foods for which methods of analysis for fat or lipids are not specified. Method is for lipids, not for fats (triglycerides and other ether-soluble materials).

REFERENCE

JAOAC 66, 927 (1983).

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 038

Perchloric Acid Digestion Procedure

1. Weigh 0.25 grams of sample directly into a glass 50 mL florence flash.

2. Add 4 ml of Instra analyzed nitric acid 70.0-71.0% to each flask.

3. Place the flasks in a rack and microwave for 5 minutes at 75% power.

4. Remove the rack from the microwave and allow to cool completely in the fume hood.

5. Add 2.5 ml of perchloric acid to each flask.

6. Place the rack of flasks in the microwave and heat gently to dense white fumes. Take the sample volume down very carefully to approximately 1 ml.

7. Remove the rack of flasks from the microwave and place in the fume hood. Allow to cool completely.

8. Quantitatively transfer the contents of each flask to a 15 ml plastic centrifuge tube. Bring all samples to a final volume of 14 ml with deionized water.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 039

MEASUREMENT BY ICP-MS

MEASUREMENT BY ICP-MS

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 040

Nitric Acid Digestion of Water Sample at 85 C Using a Water Bath

1. Place 44-45 ml of the water sample in a 50 ml polypropylene centrifuge tube.

2. Add 5 ml of Baker Instra-analyzed HNO3.

3. Heat samples in a water bath at 85 C for 1 hour.

4. Dilute to 50 ml volume with DI water.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 041

HYDRIDE GENERATION WITH ICP MEASUREMENT

HYDRIDE GENERATION WITH ICP MEASUREMENT

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 042

MERCURY IN LIQUID WASTE (MANUAL COLD-VAPOR TECHNIQUE) EPA SW846, METHOD 7470

1.0 SCOPE AND APPLICATION

1.1 Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility- procedure extracts, aqueous wastes, and ground waters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

2.2 Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.3 The typical detection limit for this method is 0.0002 mg/L.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 043

AOAC Method 942.05 for % Ash

One gram of sample is weighed into a porcelain crucible and placed in a muffle furnace at 600 C for 2 hours. The crucible is then placed in a desiccator, cooled, and weighed.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 044

The Elemental Analyzer EA1108

The Elemental Analyzer EA1108 (Figure 1) is an instrument designed for the micro, semi-micro and macro determination of total carbon, hydrogen, nitrogen, sulphur and oxygen (C, H, N, S, O) present in a wide range of organic and inorganic samples such as: organic chemicals, pharmaceuticals, fine chemicals, fuels, gasolines, oils, coal, coke, graphite, metal powders, steel, nitrides, carbides, polymers, rubbers, catalysts, soils and sediments, ceramics, carbon fibers and many others.

The original analytical method is based on the complete and instantaneous oxidation of the sample by "flash combustion" which converts all organic and inorganic substances into combustion products. The resulting combustion gases pass through a reduction furnace and are swept into the chromatographic column by the carrier gas (helium) where they are separated and detected by a thermal conductivity detector (TCD) which gives an output signal proportional to the concentration of the individual components of the mixture.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 045

Flame Atomic Absorption

Flame Atomic Absorption -- Perkin Elmer 603

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 046

Fluoride Determination

Approximately 70mg of sample is placed in a 7ml teflon digestion vessel with 3ml of 3M nitric acid. The vessel is tightly capped and placed in a 90 C drying oven for 1 hour. The sample is then diluted to an appropriate volume with deionized water.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 047

Salinity Calculation

N. Fofonoff and R. Millard -- "Algorithms for Calculation of Fundatmental Properties of Seawater" from UNESCO Technical Papers in Marine Science, No 44.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 048

Methylmercury

Extraction of methylmercury. The sample was treated with 10 ml of 5 mol/l HCI to liberate methylmercury (plus any other organomercury species), which was then extracted into 3 x 20 ml aliquots of toluene. The combined toluene aliquots was diluted to 100 ml with toluene. This solution was amenable to gas chromatography. For the other techniques which accept only aqueous samples, methylmercury was extracted from the toluene solution with a cysteine acetate solution, 4/1 v/v. To prepare the cysteine acetate solution, 0.5 g of cysteine hydrochloride monohydrate, 0.34 g of sodium acetate and 6.25 g of anhydrous sodium sulphate was dissolved in 50 ml of DDW.

Cold vapor atomic absorption spectrometry. Digestion: EPA method 7470 (nitric, sulfuric acids permangante, persulfate). After digestion the resulting solution was then subjected to CVAAS. An automated mercury analyzer, the Leeman Labs PS200, was used for measurement. The reducing agent was a solution of stannous chloride and hydroxylamine hydrochloride (2/1).

Reference:

Marine Biological Reference Materials for Methylmercury: Analytical Methodologies Used in Certification. Fresenius Z Anal. Chem. (1989) 333:641-644.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 049

Total Kjeldahl Nitrogen (as N) (TKN): EPA Method 351.3

Summary of method: The sample is heated in the presence of conc. sulfuric acid, K2SO4 and CuSO4 and evaporated until SO3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and is treated and made alkaline with a hydroxide-thiosulfate solution. The ammonia is distilled and determined by titration.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 050

Determination of Inorganic Anions by Ion Chromatography: EPA Method 300.0

Summary of method: A small volume of sample, typically 2 to 3 ml, is introduced in to an ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 051

Total Phosphorus (as P) - EPA Method 365.3

Ammonium molybdate and antimony potassium tartrate are added to the sample in an acid medium to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue colored complex by ascorbic acid. The color is proportional to the phosphorus concentration. Since only orthophosphate forms a blue color in this test, sulfuric acid and ammonium persulfate are added to convert polyphosphates and organic phosphorus compounds to the orthophosphate form.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 053

Determination of Sulfide as Hydrogen Sulfide By Gas Phase Molecular AS

This is a molecular absorption method which uses a sharp-line irradiation source for the determination of sulfide in water and sludge samples. Sulfide is transformed into hydrogen sulfide by adding sulfuric acid and then the absorption of H2S is measured at 196.0 nm using the selenium atomic line.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 054

Organotin

Approximately 0.02 grams of the sample was transferred into a 50 mL beaker. 10 mLs of acetonitrile with 0.1% acetic acid was added to the beaker. The beaker was covered with parafilm and placed in the ultrasonic bath for approximately 30 minutes. The contents of the beaker was transferred into a centrifuge tube. The sample was then centrifuged for 20 minutes at about 2000 rpm. The supernatant was decanted

into the sample cup and analyzed for Sn by GFAA with a calibration curve generated by standards made from tributyltin chloride.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 055

Sulfur

Preparation: Weigh sample to nearest 0.01 mg. Weigh the sample directly into a sample boat tared on electronic balance. The weight is automatically transferred to SC432 database. Cover the sample with V2O6 combustion accelerator as called for by sample type. Place liquids in a capsule and crimp.

Instrument: LECO SC 432DR Sulfur Analyzer

Calibration: Three conditioners of 4-12 mg Cystine. One each of NIST traceable sulphanilic acid at 18, 15, 12, 9, 6, and 3 mg, plus 50 mg of NIST non-fat milk powder for initial calibration. Calibration is generated internally using a quadratic regressed curve.

Control: 6-1625 Cystine (26.69% S)

Determination: Combustion in O2 atmosphere at 1350 +/- 50 C. An infrared detector quantitates the resulting SO2.

Detection limit: 1 %.

Precision and Accuracy:RSD RE

1.56 % -0.20%

Calculations: Internal, quadratic regression.

References: Leco Sulfur Analyzer Systems Manual: 602-500, Version 1.0

ASTM D4239-83, Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods, Method C: Annual Book of ASTM Methods, Vol. 05:05, 1992.

ASTM D 1552-95: Test Method for Sulfur in Petroleum Products (High- Temperature Method); Annual Book of ASTM Methods, Vol. 05:01, 1997.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 056

AVS

An aliquot of acid is added to a fixed weight of waste in a closed system. The generated gas is swept into a scrubber and the sulfide is then quantified using Method 9034 which is a titrimetric procedure.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 057

Total Dissolved Solids

A well mixed water sample is filtered through a standard glass fiber filter and the filtrate is evaporated in a weighed dish and dried to a constant weight at 180 C. The increase in dish weight represents the total dissolved solids.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 058

Total Suspended Solids

A well mised water sample is filtered through a weighed standard glass fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105 C. The increase in weight of the filter represents the total suspended solids.

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Lab Name: Research Triangle Institute (RTI) Laboratory Method Code 060

Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption (EPA Method 7473)

Approximately 0.25 g of sample is introduced into a Milestone DMA-80 Mercury Analyzer. The sample is dried, then chemically decomposed at 750oC under an oxygen atmosphere, liberating the mercury content in the sample. The mercury vapor is then collected selectively on a gold amalgam surface. The amalgamator is then rapidly heated to 800oC and the oxygen flow carries the mercury vapor though an absorption cell where the Hg absorbance (as a function of Hg concentration) is

measured at 253.7nm. The detection limit for sediments and tissues is approximately 0.01 æg/g.

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TDI Brooks International, Inc. (TDI) Laboratory Methods

Select one of the links below to display the method descriptions associated with TDI or <HERE> to go to the Start Page.

Method Code	Method Title
001	Tissue Extraction Method (PAH and OCs)
002	Aromatic Hydrocarbon Determination by Selected Ion Monitoring (SIM) Gas Chromatography/Mass Spectrometry (GC/MS)
003	Chlorinated Hydrocarbon Determination by Gas Chromatography/Electron Capture Detection (GC/ECD)
004	Confirmation of Analytes
005	Sediment Extraction Method (PAH and OCs)
006	Total Organic and Inorganic Carbon is Soils and Sediments
007	Determination of Particle Size Distribution in Sediments
008	Determination of Total Petroleum Hydrocarbons in Soil/Sediment
009	Determination of Aliphatic Hydrocarbons in Soil/Sediment
010	Volatile Organic Compounds (VOC) Determination by Purge and Trap Analysis with Gas Chromatography/Mass Spectrometry (GC/MS)
011	Water Extraction Method (PAH and OC)

Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 001

Tissue Extraction Method (PAH and OCs)

Tissue samples are either immediately processed or stored frozen (-20°C) until processing. Tissues are processed as appropriate for the tissue type (e.g. dissection, shucking). Processed tissues are homogenized using a variety of mechanical methods (Waring blender, Hobart meat grinder or Tissumisser), depending upon the tissue amount and type. After homogenization, an approximate 1 g aliquot is removed and dried in an oven at 105°C to a constant weight to determine % moisture. The remaining samples are stored in certified pre-cleaned jars frozen (- 20°C) until analysis. Prior to extraction, tissue samples may be lypholized or a wet aliquot is chemically dried using a dessicant such as Hydromatrix or sodium sulfate. Samples are then extracted using a Dionex ASE200 Accelerated Solvent Extractor (ASE). The dried sample or the sample and dessicant material is loaded into 22 or 33 mL stainless steel ASE extraction tubes. The extraction are performed using 100% dicholormethane at 100°C and 2000 psi. The extracted organics dissolved in the solvent are collected in 60 mL glass vials. The extract is concentrated to approximately 10 mL in the collection vials and then transferred to 25 mL Kurdena-Danish (KD) concentrator tubes. The sample extract is concentrated to 3 mL in a water bath at 55-60°C. If lipid weight is required, a 100 mL aliquot is removed and weighed using a microbalance. Interfering non-contaminant organic material (primarily lipids) must be removed prior to instrument analyses.

The extract is processed through silica gel/alumina chromatography columns and High Performance Liquid Chromatography (HPLC). The remaining 2.9 mL of sample extracted are loaded on top of 300 mm x 19 mm glass liquid chromatography columns packed with 10 g of deactivated alumina and 20 g of deactivated silica gel. The columns are loaded in 100 % dichloromethane. The dichloromethane is replaced by adding 40 mL of pentane. The extract is carefully added to the top of the chromatography columns. The column is flushed at a rate of 1-2 mL per minute using 200 mL of 50:50 pentane/dichloromethane and collected into 250 mL flasks. The eluent collected in the 250 mL flask is evaporated to 2 mL using a waterbath at 55-50°C. The samples is transferred into 4 mL amber vials. Extracts subsequently processed by HPLC to further remove lipid interferences. Lipid removal is accomplished by flushing samples with dichlormethane through size exclusion Phenogel 10 m GPC 100 A columns. Approximately 40 mL is collected using a fraction collector, which is concentrated to 0.5 mL using a water bath at 55-60C. The concentrated extract is then analyzed by GC/MS for polynuclear aromatic hydrocarbons (PAHs) or GC/ECD for selected organochlorines (OCs).

Additional column chromatography is required to separate PCBs from toxaphene/pesticides when toxphene analysis is
required and to separate planar PCBs. If toxaphene analyses is required, an aliquot of the extract prior to HPLC clean up is processed through a 3% deactivated silica gel column. The column is packed in dicloromethane which is then flushed with 50 mL of pentane. The sample extract is transferred to the top of the column and

flushed with 100 mL of pentane. The fraction contains PCBs and DDTs. The column is then flushed with 120 mL of 50:50 pentane/dichloromethane. This fraction contains toxaphene and chlorinated pesticides. Both fractions are reduced to 1 mL using a water bath at 55-60°C. The extracts are then ready for instrument analysis.

If planar PCB analyses are required, the PCB/DDT fraction prepared by 3% silica gel column is further processed by column chromatography packed with 2 g of 1:19 (5% by weight) mixture of activated carbon/Celite. The column and flushed with 25 mL of 1:4 dichloromethane/cyclohexane mixture. The sample is added to the top of the column and flushed with 50 mL of 1:4 dichloromethane/cyclohexane mixture, followed by 30 mL of 9:1 dichloromethane/toluene. This is followed by the addition of 40 mL of toluene. The toluene fraction contains the planar PCBs and is concentrated to 1 mL in a Zymark TurboVap II concentrator at 42°C and 20 psi. The sample is ready for instrument analysis.

REFERENCES:

Lauenstein, G.G. and A.Y. Cantillo, ed. (1993). Sampling Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984-1992; Volume IV: Comprehensive Descriptions of Trace Organic Analytical Methods. NOAA Technical memorandum NOS ORCA 71, Silver Spring, MD.

U.S. Environmental Protection Agency. 2001. National Coastal Assessment Quality Assurance Project Plan 2001-2004. United States Environmental Protection Agency, Office of Research and Development, National Health and Environmental Effects Research Laboratory, Gulf Ecology Division, Gulf Breeze, FL. EPA/6 20/R-01/002.

Environmental Protection Agency, "Method 3545: Pressurized Fluid Extraction (PFE)," in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods EPA SW-846 [Version 2 (December 1997), Integrated Manual through Update III] Washington DC, U.S. Environmental Protection Agency (1997)

Zuloaga, O.; Etxebarria, N.; Fernandez L. A.;Madariaga, J.M.; Optimization and comparison of MAE, ASE and Soxhlet extraction for the determination of HCH isomers in soil samples. Fresenius J Anal Chem, 2000, 367, 733-737.

Schantz, M.; Nichols, J. J.; Wise, S. A.; Evaluation of Pressurized Fluid Extraction for the Extraction of Environmental Matrix Reference Material, Anal. Chem., 1997, 69, 4210-4219.

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 002

Aromatic Hydrocarbon Determination by Selected Ion Monitoring (SIM) Gas Chromatography/Mass Spectrometry (GC/MS)

Polycyclic aromatic hydrocarbons (PAH) and their alkylated homologues are analyzed in sample extracts by a HewlettPackard, model 5890 GS and model 5972 MS operated in SIM using a capillary column. The GC is operated in splitless mode and the capillary column is an Agilent Technologies HP-5MS (60 m x 0.25 mm ID and 0.25 mm film thickness). The carrier gas is helium at a flow rate of 1 mL/minute. The temperature of the injection port is 300°C and transfer line is 290°C. The initial oven temperature is 60°C, the ramp rate is 7°C/minutes to a final oven temperature of 310°C and held for 20 minutes. For analyte identification, the extracted ion current profiles of the primary m/z and the confirmatory ion for each analyte must be at a maximum in the same scan or within one scan of each other and the retention time must fall with 5 seconds of the retention time of the authentic standard or alkyl homologue grouping. The pattern of alkylated PAH homologue groupings is established by analysis of reference oil standards. The relative peak heights of the primary mass ion compared to the confirmation or secondary mass ion must fall within 30 % of the relative intensities of these masses in a reference mass spectrum.

REFERENCES:

Lauenstein, G.G. and A.Y. Cantillo, ed. (1993). Sampling Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984-1992; Volume IV: Comprehensive Descriptions of Trace Organic Analytical Methods. NOAA Technical memorandum NOS ORCA 71, Silver Spring, MD.

U.S. Environmental Protection Agency. 2001. National Coastal Assessment Quality Assurance Project Plan 2001-2004. United States Environmental Protection Agency, Office of Research and Development, National Health and Environmental Effects Research Laboratory, Gulf Ecology Division, Gulf Breeze, FL. EPA/620/R-01/002.

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 003

Chlorinated Hydrocarbon Determination by Gas Chromatography/Electron Capture Detection (GC/ECD)

Chlorinated hydrocarbons are determined in samples by GC/ECD. Samples are extracted as previously described and analyzed on a HewletPackard (HP), model 5890 GC equipped with an ECD. Between 1 to 5 mL of sample is injected using an HP, model 7673A autosampler. The instrument is set up with dual columns. The primary capillary column is a J&W DB-5 (30 m x 24 mm ID and 0.25 mm film thickness). The second column, a confirmation column, is a J&W DB-17HT (30 m x

0.25 mm ID and 0.15 mm film thickness). The inlet system is splitless and the carrier gas is helium at a flow rate of 1 mL/min. For the analysis of standard halogenated hydrocarbons, the temperature of the injection port is 275°C and the detector is 325°C. The initial oven temperature is 100°C with a hold time of 1 minute. The ramp rate is 5°C/minute to 140°C with a hold time of 1 minute, followed by a ramp rate of 1.5°C /minute to 250C with a hold time of 1 minute and finally a ramp rate of 10°C/minutes to 300°C with a final hold time of 5 minutes. For planar PCBs the instrument is operated in the splitless mode withhelium as the carrier gas with a flow rate of 1 mL/minute. The temperature of the injection port is 275°C and the detector is 325°C. The initial oven temperature is 100°C, which is held for 1 minute. The ramp rate is 10°C/minute to 150°C, followed by a ramp rate of 6.0°C/minute to 270°C with a hold time of 3 minutes. The retention time of sample analytes must fall within 15 seconds of the retention time of analytes in a calibration standard or a retention index solutions. The levels of aroclors and toxophene are determined using retention index solutions of both complex mixtures. Arochlors are determined in a similar method to that described in EPA SW-846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Method 8082 (1997).

REFERENCES:

Lauenstein, G.G. and A.Y. Cantillo, ed. (1993). Sampling Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984-1992; Volume IV: Comprehensive Descriptions of Trace Organic Analytical Methods. NOAA Technical memorandum NOS ORCA 71, Silver Spring, MD.

U.S. Environmental Protection Agency. 2001. National Coastal Assessment Quality Assurance Project Plan 2001-2004. United States Environmental Protection Agency, Office of Research and Development, National Health and Environmental Effects Research Laboratory, Gulf Ecology Division, Gulf Breeze, FL. EPA/620/R-01/002.

Environmental Protection Agency, "Method 8082: Polychlorinated Biphenls (PCBs) by Gas Chromatography," in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods EPA SW-846 [Version 2 (December 1997), Integrated Manual through Update III] Washington DC, U.S. Environmental Protection Agency (1997)

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 004

Confirmation of Analytes

The presence of pesticides and PCBs is confirmed by gas chromatography/mass spectrometry (GC/MS) in either full scan or selection ion monitoring mode (SIM).

Samples are extracted as previously described. Samples are initially screened by GC/ECD and GC/MS to confirm the presence of specific analytes in a sample. When analytes are detected at 10 x the SIM limit of detection they may be confirmed by SIM GC/MS. The samples are analyzed on a HewlettPackard 5890GC/ 5972MS. The GC is temperature- programmed and operated in splitless mode. The analytical column is an Aglient Technologies HP5MS (60 m x 0.25 mm ID and with a 0.25 mm film thickness). The carrier gas is helium with a flow rate of 1mL/min. The temperature of the injection port is 300°C and the transfer line is 290°C. The oven is at an initial temperature of 60°C with a ramp time of 7°C/minute. Analytes are "confirmed" when the spectrum contains at least 3 of the major ions. The chromatographic peaks must be at least 3 x the background noise and must be within once scan of each other and match the retention time of the standard run under the same conditions to be "confirmed"

REFERENCES:

Environmental Protection Agency, "Method 8270C: Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)" in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods EPA SW-846 [Version 2 (December 1997), Integrated Manual through Update III] Washington DC, U.S. Environmental Protection Agency (1997)

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 005

Sediment Extraction Method (PAH and OCs)

Sediment samples are either immediately processed or stored frozen (- 20°C) until processing. A sediment aliquot is dried in a convection oven at 40°C. After the sediment is dry is it thoroughly homogenized using a ceramic motar and pestle. An additional aliquot of approximately 1 g of wet sediment is removed and dried in an oven at 105°C to a constant weight to determine % moisture. Samples are extracted using a Dionex ASE200 Accelerated Solvent Extractor (ASE). The dried sample is loaded into 22 or 33 mL stainless steel ASE extraction tubes. The extractions are performed using 100% dicholormethane at 100 °C and 2000 psi. The extracted organics dissolved in the solvent are collected in 60 mL glass vials. The extract is concentrated to approximately 10 mL in the collection vials and then transferred to 25 mL Kurdena-Danish (KD) concentrator tubes. The sample extract is concentrated to 3 mL in a water bath at 55-60°C. If extractable organic material weight is required, a 100 mL aliquot is removed and weighed using a microbalance. Interfering non-contaminant organic materials must be removed prior to instrument analyses.

The extract is processed through silica gel/alumina chromatography columns. The sample extract is loaded on top of 300 mm x 19 mm glass liquid chromatography

columns packed with 10 g of deactivated alumina and 20 g of deactivated silica gel. The columns are loaded in 100 % dichloromethane. The dichloromethane is replaced by adding 40 mL of pentane. The extract is carefully added to the top of the chromatography columns. The column is flushed at a rate of 1-2 mL per minute using 200 mL of 50:50 pentane/dichloromethane and collected into 250 mL flasks. The eluent collected in the 250 mL flask is evaporated to 2 mL using a waterbath at 55-50°C. The samples is transferred into 2 mL amber vials. The concentrated extract is then analyzed by GC/MS for polynuclear aromatic hydrocarbons (PAHs) or GC/ECD for selected organochlorines (OCs).

Additional column chromatography is required to separate PCBs from toxaphene/pesticides when toxphene analysis is required and to separate planar PCBs. If toxaphene analyses is required, an aliquot of the extract after silica/alumina clean-up is processed through a 3% deactivated silica gel column. The column is packed in dicloromethane which is then flushed with 50 mL of pentane. The sample extract is transferred to the top of the column and flushed with 100 mL of pentane. The fraction contains PCBs and DDTs. The column is then flushed with 120 mL of 50:50 pentane/dichloromethane. This fraction contains toxaphene and chlorinated pesticides. Both fractions are reduced to 1 mL using a water bath at 55-60°C. The extracts are then ready for instrument analysis.

If planar PCB analyses are required, the PCB/DDT fraction prepared by 3% silica gel column is further processed by column chromatography packed with 2 g of 1:19 (5% by weight) mixture of activated carbon/Celite. The column and flushed with 25 mL of 1:4 dichloromethane/cyclohexane mixture. The sample is added to the top of the column and flushed with 50 mL of 1:4 dichloromethane/cyclohexane mixture, followed by 30 mL of 9:1 dichloromethane/toluene. This is followed by the addition of 40 mL of toluene. The toluene fraction contains the planar PCBs and is concentrated to 1 mL in a Zymark TurboVap II concentrator at 42°C and 20 psi. The sample is ready for instrument analysis.

References:

Lauenstein, G.G. and A.Y. Cantillo, ed. (1993). Sampling Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984-1992; Volume IV: Comprehensive Descriptions of Trace Organic Analytical Methods. NOAA Technical memorandum NOS ORCA 71, Silver Spring, MD.

U.S. Environmental Protection Agency. 2001. National Coastal Assessment Quality Assurance Project Plan 2001-2004. United States Environmental Protection Agency, Office of Research and Development, National Health and Environmental Effects Research Laboratory, Gulf Ecology Division, Gulf Breeze, FL. EPA/620/R- 01/002.

Environmental Protection Agency, "Method 3545: Pressurized Fluid Extraction (PFE)," in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods EPA SW-846 [Version 2 (December 1997), Integrated Manual through Update III] Washington DC, U.S. Environmental Protection Agency (1997)

Zuloaga, O.; Etxebarria, N.; Fernandez L. A.;Madariaga, J. M.; Optimization and comparison of MAE, ASE and Soxhlet extraction for the determination of HCH isomers in soil samples. Fresenius J Anal Chem, 200
0, 367, 733-737.

Schantz, M.; Nichols, J. J.; Wise, S. A.; Evaluation of Pressurized Fluid Extraction for the Extraction of Environmental Matrix Reference Material, Anal. Chem., 1997, 69, 4210-4219.

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 006

Total Organic and Inorganic Carbon is Soils and Sediments

Sediments samples are either immediately processed or stored frozen (- 20°C) until processing. A sediment aliquot is dried in a convection oven at 105°C. For total carbon analysis an aliquot of approximately 350 mg is placed in a clean, carbon-free combustion boat. The sample boats are loaded into a LECO autosampler rack assembly. The dried sample is combusted at 1350°C under an oxygen atmosphere in a LECO CR-412 Carbon Analyzer. Carbon present in the samples is oxidized to form CO2 gas. The gaseous sample flows through a nondispersive infrared (NIDR) detection cell. The mass of carbon dioxide is measured and converted to a percentage value with respect to sample weight. Percent total organic carbon is determined by pre- treating dry samples with 1:1 phosphoric acid to remove all inorganic carbon. Approximately 350 mg of treated sample is loaded into a clean, carbon-free combustion boat. The sample boats are loaded into a LECO autosampler rack assembly. The dried sample is combusted at 1350°C under an oxygen atmosphere in a LECO CR-412 Carbon Analyzer. Carbon present in the samples is oxidized to form CO2 gas. The gaseous sample flows through a nondispersive infrared (NIDR) detection cell. The mass of carbon dioxide is measured and converted to a percentage value with respect to sample weight.

References:

Kahn, Lloyd. 1988. Determination of total organic carbon is sediment. USEPA.

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 007

Determination of Particle Size Distribution in Sediments

Sediment samples are stored refrigerated at 4°C until processing. Samples are thoroughly mixed and an aliquot of approximately 25 to 50 g is weighed and placed into 750 mL wide-mouth jars. Approximately 250 mL of deflocculent solution (2.5 g/L sodium hexametaphosphate in DI water) is added to the jar. The jar is sealed and shaken until the sample is disaggregated. Once shaken the sample and deflocculent solution is poured through a 63 mm sieve into 1000 mL graduated cylinder. The coarse sediment left on top of the 63 mm sieve is concentrated into a 150 mL beaker. Deflocculent is added to 975 mL in the graduate cylinder. The coarse sediment in the beaker is dried in an oven at 70°C to 90°C. The weight is noted and then transferred to the top sieve (-1 phi) in a sieve stack that is arranged in descending order (-1 phi and +4 phi). The sieve stack is covered and shaken for 15 minutes on a sieve shaker. Empty the material from the top sieve onto a large piece of clean paper and weigh. The material from the next sieve is emptied onto the large piece of clean paper and weighed. Any material that passes through both sieves is added to the graduated cylinder. The phi fraction represents the gravel size and the + 4 phi represents the sand size. The silt/clay fraction is determined by filling the corresponding graduated cylinder to 1000 mL with deflocculent solution. After allowing the cylinder to stand for 24 hours at 24°C, the cylinder is thoroughly mixed. After 20 seconds a 25 mL aliquot is withdrawn from a depth of 20 cm (representing the silt fraction). The aliquot is emptied into a tared 50 mL beaker. A second 25 mL aliquot is withdrawn from the graduated cylinder froma depth of 10 cm at an interval of 2 hours and 3 minutes (representing the clay fraction) and placed into a tared 50 mL beaker. The 50 mL beakers are dried in an oven at 70°C to 90°C until dry. The dried samples are then weighed.

References:

APHA. 1989. Standard Methods for the Examination of Water and Wastewater. Clesceri, Greenberg, and Trussel, eds. American Public Health Association, 17th edition.

Folk, R.L. 1974. Petrology of Sedimentary Rocks. Hemphill Publishing Co., Austin, TX 184 pp.

Plumb, R.H. 1981. Procedure for Handling and Chemical Analysis of Sediment and Water Samples. Technical Report EPA/CE 81-1, prepared by Great Lakes Laboratory, State University College at Buffalo NY for the U.S. EPA/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material. Published by the U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

Tetra Tech. 1986. Quality Assurance and Quality Control (QA/QC for 301 (h) Monitoring Programs. Guidance on Field and Laboratory Methods. USEPA, TC-3953-04, Final Report, 267 pp.

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 008

Determination of Total Petroleum Hydrocarbons in Soil/Sediment

Sediment samples are extracted as described in method 005. Total petroleum hydrocarbons (TPH) are determined by quantifying the TPH with gas chromatography/flame ionization detection (GC/FID)

TPH are analyzed using a HewelettPackard, model 5890 Gas Chromatograph (GC) with a Flame Ionization Detector (FID) operated in a splitless mode. A HP-1MS capillary column (30m x 0.25 mm ID and 0.25 mm film thickness) is used to resolve peaks. The carrier gas is helium at a flow rate of 1.5 mL/min. The temperature of the injection port is 300°C and transfer line is 300C. The initial oven temperature is 60°C, the ramp rate is 12°C/min to a final oven temperature of 180°C. For analytes of interest, a response factor relative to the internal standard is determined at each calibration level. All 5 response factors are averaged for a mean relative response factor. Data are surrogate corrected. TPH is determined by straight line integration between the retention times for n-C10 and n-C34.

Environmental Protection Agency, "Method 8100/8015. Polynuclear Aromatic Hydrocarbons/Nonhalogenated Organics using GC/FID"," in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods EPA SW-846 [Version 2 (December 1997), Integrated Manual through Update III] Washington DC, U.S. Environmental Protection Agency (1997)

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 009

Determination of Aliphatic Hydrocarbons in Soil/Sediment

Sediment samples are extracted as described in method 005. Aliphatic hydrocarbons are determined by quantifying target analytes with a gas chromatography/flame ionization detection (GC/FID)

Aliphatic hydrocarbons are analyzed using a HewelettPackard, model 5890 Gas Chromatograph (GC) with a Flame Ionization Detector (FID) operated in a splitless mode. A HP-1MS capillary column (30m x 0.25 mm ID and 0.25 mm film thickness) is used to resolve peaks. The carrier gas is helium at a flow rate of 1.5 mL/min. The temperature of the injection port is 300°C and transfer line is 300C. The initial oven temperature is 60°C, the ramp rate is 12°C/min to a final oven temperature of 180°C. Normal alkanes with 10 to 34 carbons and the isoprenoids pristine and phytane are determined using this procedure. For analytes of interest, a response factor relative

to the internal standard is determined at each calibration level. All 5 response factors are averaged for a mean relative response factor. Data are surrogate corrected.

Environmental Protection Agency, "Method 8100/8015. Polynuclear Aromatic Hydrocarbons/Nonhalogenated Organics using GC/FID"," in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods EPA SW-846 [Version 2 (December 1997), Integrated Manual through Update III] Washington DC, U.S. Environmental Protection Agency (1997).

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 010

Volatile Organic Compounds (VOC) Determination by Purge and Trap Analysis with Gas Chromatography/Mass Spectrometry (GC/MS)

Volatile organic compounds (VOC) are introduced into a gas chromatograph by a purge-and-trap method (OI Corp Model 4560 with a DPM 16 Autosampler). The samples are purged with purified helium for 12 minutes and capturing VOC on a Tenax trap. The analytes are introduced directly to a narrow-bore capillary column by flash evaporation. The capillary column is temperature-programmed to separate the analytes. Resolved analytes are detected with a Thermo- Finnigan PolarisQ ion-trap mass spectrometer (MS) interfaced to the Thermo-Finnigan TRACE gas chromatograph (GC). The GC is operated in splitless mode and the capillary column is a J&W Scientific/Agilent Technologies DB-1 (60 m x 0.25-mm ID and 1.0 mm film thickness). The carrier gas is helium at a flow rate of 2 mL/minute. The temperature of the injection port is 175°C and transfer line is 290°C. The initial oven temperature is 45°C for 2 minutes, the ramp rate is 5°C/minutes to an oven temperature of 120°C and held for 0 minutes, then ramped at a rate of 10°C/minutes to a final oven temperature of 220°C and held for 5 minutes. For analyte identification, the extracted ion current profiles of the primary m/z and the confirmatory ion for each analyte must be at a maximum in the same scan or within one scan of each other and the retention time must fall with 5 seconds of the retention time of the authentic standard. The relative peak heights of the primary mass ion compared to the confirmation or secondary mass ion must fall within 30 % of the relative intensities of these masses in a reference mass spectrum.

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Lab Name: TDI Brooks International, Inc. (TDI) Laboratory
Method Code 011

Water Extraction Method (PAH and OC)

Water samples are either immediately processed or stored refrigerated (4 degrees C) until processing. The pH of samples is adjusted to <2 by the addition of 1 mL of hydrochloric acid. Samples are serially extracted using separatory funnels and dichloromethane. For a 1 L sample, 100 mL of dichloromethane is added. The sample is then shaken for three minutes. The organic layer is allowed to separate from the aqueous phase. The organic layer is drained into a 500 mL round bottom flask through a glass funnel containing a glass fiber filter and combusted sodium sulfate. This process is repeated twice more. The extract is concentrated to approximately 10 mL in the 500 mL round bottom flasks in a water bath at 55-60 degrees C and then transferred to 25 mL Kurdena-Danish (KD) concentrator tubes. The sample extract is concentrated to 3 mL in a water bath at 55-60 degrees C. If extractable organic material weight is required, a 100 micro litter aliquot is removed and weighed using a microbalance. Interfering non-contaminant organic materials may be removed prior to instrument analyses.

If the extract is colored then sample clean up may be required. The extract is processed through silica gel/alumina chromatography columns. The sample extract is loaded on top of 300 mm x 19 mm glass liquid chromatography columns packed with 10 g of deactivated alumina and 20 g of deactivated silica gel. The columns are loaded in 100 % dichloromethane. The dichloromethane is replaced by adding 40 mL of pentane. The extract is carefully added to the top of the chromatography columns. The column is flushed at a rate of 1-2 mL per minute using 200 mL of 50:50 pentane/dichloromethane and collected into 250 mL flasks. The eluent collected in the 250 mL flask is evaporated to 2 mL using a waterbath at 55-50 degrees C. The samples is transferred into 2 mL amber vials. The concentrated extract is then analyzed by GC/MS for polynuclear aromatic hydrocarbons (PAHs) or GC/ECD for selected organochlorines (OCs).

Additional column chromatography may be required to separate PCBs from toxaphene/pesticides when toxphene analysis is required and to separate planar PCBs. If toxaphene analyses is required, an aliquot of the extract after silica/alumina clean-up is processed through a 3% deactivated silica gel column. The column is packed in dicloromethane which is then flushed with 50 mL of pentane. The sample extract is transferred to the top of the column and flushed with 100 mL of pentane. The fraction contains PCBs and DDTs. The column is then flushed with 120 mL of 50:50 pentane/dichloromethane. This fraction contains toxaphene and chlorinated pesticides. Both fractions are reduced to 1 mL using a water bath at 55-60 degrees C. The extracts are then ready for instrument analysis.

If planar PCB analyses are required, the PCB/DDT fraction prepared by 3% silica gel column is further processed by column chromatography packed with 2 g of 1:19 (5% by weight) mixture of activated carbon/Celite. The column and flushed with 25 mL of 1:4 dichloromethane/cyclohexane mixture. The sample is added to the top of the column and flushed with 50 mL of 1:4 dichloromethane/cyclohexane mixture, followed by 30 mL of 9:1 dichloromethane/toluene. This is followed by the addition of 40 mL of toluene. The toluene fraction contains the planar PCBs and is concentrated to 1 mL in a Zymark TurboVap II concentrator at 42 degrees C and 20 psi. The sample is ready for instrument analysis.

References:

Lauenstein, G.G. and A.Y. Cantillo, ed. (1993). Sampling Analytical Methods of the National Status and Trends Program National Benthic Surveillance and Mussel Watch Projects 1984-1992; Volume IV: Comprehensive Descriptions of Trace Organic Analytical Methods. NOAA Technical memorandum NOS ORCA 71, Silver Spring , MD.

U.S. Environmental Protection Agency. 2001. National Coastal Assessment Quality Assurance Project Plan 2001-2004. United States Environmental Protection Agency, Office of Research and Development, National Health and Environmental Effects Research Laboratory, Gulf Ecology Division, Gulf Breeze, FL. EPA/620/R-01/002.

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Trace Element Research Laboratory (TERL)
Analytical Methods

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002	Digestion of Soil and Sediment
003	Digestion of Water, Soil, Sediment, and Biological Tissue for Mercury Analysis
004	Digestion of Water Samples for "Total Recoverable" Metals (Other than Mercury)
005	Analysis of Trace Metals by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP)
006	Analysis of Trace Metals by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)
007	Analysis of Mercury by Cold-Vapor Atomic Absorption Spectroscopy (CVAAS)
008	Analysis of Trace Metals by Flame Atomic Absorption Spectroscopy (FAAS)
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010	Analysis of Trace Metals in Biological Tissue, Soils, and Sediments by Instrumental Neutron Activation Analysis (INAA)
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017 Analysis of Inorganic Arsenic (As) in Tissue and Sediment Samples

018 Analysis of Inorganic Species and Organic Arsenic in Tissues and Sediment Samples

019 Preconcentration of Water Samples for Trace Element Analysis

020 Determination of Total Suspended Solids

021 Nitrogen

022 Digestion of Biological Tissue in Microwave Oven Under Pressure

023 Digestion of Biological Tissue in Microwave Oven Under Ambient Pressure

024 Determination of Mercury in Tissue and Sediment

025 Determination of Mercury in Water

026 Determination of Methyl Mercury in Water

027 Determination of Perchlorate in Water

028 Determination of Anions in Water by Wet Chemistry Procedures

029 Determination of carbon, nitrogen, and/or stable C and N isotopes by isotope ratio mass spectroscopy

Lab Name: *Trace Element Research Laboratory* Method Code: *001*

Digestion of Biological Tissue

Liquid or solid biological tissue samples are wet digested with nitric acid and converted into acidic digest solutions for analysis by various atomic spectroscopy methods. When possible, tissue is freeze dried in order to minimize loss of analytes and to facilitate subsequent sample preparation steps, and then homogenized to a fine powder by ball-milling in plastic containers. Approximately 0.20 to 0.25 g of powdered tissue is weighed into a Teflon reaction vessel and 3 ml of HNO3 are added. The closed reaction vessel is heated in a 130 C oven until digestion is complete. Samples are then diluted to a final volume of 20 ml with quartz distilled water and stored in 1 oz. polyethylene bottles for later analysis by instrumental techniques.

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Lab Name: *Trace Element Research Laboratory* Method Code: *002*

Digestion of Soil and Sediment

Soil and sediment samples are wet digested with nitric and hydrochloric acids and converted into acidic digest solutions for analysis by various atomic spectroscopy methods. Wet sediment is homogenized in its container, and an aliquot is freeze dried and homogenized to a fine powder. Approximately 0.5 g of powdered sediment is weighed into a tall form beaker and 10 ml of Aqua Regia (1:4v:v HNO3:HCl) are added. The vessel is heated on a hot plate for 2 hrs., with swirling, repositioning, and rinsing of the beaker walls (if necessary) at 1 hr. Samples are then transferred into centrifuge tubes and diluted to a final volume of 30 ml with distilled deionized water. They are spun to settle particles and transferred to 1 oz polyethylene bottles for storage until analysis. The samples are then diluted as necessary and analyzed for trace metals by various TERL analytical methods.

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Lab Name: *Trace Element Research Laboratory* Method Code: *003*

Digestion of Water, Soil, Sediment, and Biological Tissue for Mercury Analysis

Before samples are analyzed by the CVAAS method in use in this laboratory, the mercury is converted to the Hg2+ form. Mercury is digested by a modified version of EPA method 245.5 and 245.6. Sediment and tissue samples can be analyzed either freeze dried or on a wet basis. Sediment samples are homogenized by mixing before subsampling, while tissue samples are homogenized in the original sample containers either after freeze drying or with a Tekmar Tissumizer and subsampled. Samples are digested with nitric acid, sulfuric acid, potassium permanganate, and

potassium persulfate in polypropylene tubes in a water bath at 90-95 C. Before analysis, hydroxylamine hydrochloride is added to reduce excess permanganate and the samples are brought to volume with distilled-deionized water.

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Lab Name: *Trace Element Research Laboratory* Method Code: *004*

Digestion of Water Samples for
"Total Recoverable" Metals (Other than Mercury)

Water samples are digested for two hours at 85 degrees Centigrade in polyethylene containers with ultrapure nitric and hydrochloric acids. Acid strength, on a vol:vol basis, is 1% HCl and 0.5% HNO3. Sample aliquots for digestion are taken after vigorous shaking to assure suspension of solids that may have settled. The original sample must have had preservative added (usually HNO3) in order to ensure that metals do not adhere to the walls of the container.

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Lab Name: *Trace Element Research Laboratory* Method Code: *005*

Analysis of Trace Metals by
Inductively Coupled Plasma Optical Emission Spectroscopy (ICP)

Liquid samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a inductively coupled argon plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes or solid state detectors. Samples are quantitated by comparison with external standards. One or more internal standards may be incorporated to compensate for physical effects resulting from viscosity and varying levels of total dissolved solids in the samples. Background correction is required and is measured adjacent to analyte lines on samples during analysis.

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Lab Name: *Trace Element Research Laboratory* Method Code: *006*

Analysis of Trace Metals by
Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)

Liquid samples requiring lower detection limits than are available by ICP are Analyzed by GFAAS, in which electrical resistance heating evaporates solvent (water), removes interfering species, and finally atomizes the analyte into the light

path of an atomic absorption spectrophotometer. The method is subject to numerous chemical and physical interferences, and requires the use of matrix modifiers to produce accurate and reliable data in real-world sediment, tissue, and water digest solutions.

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Lab Name: *Trace Element Research Laboratory* Method Code: *007*

Analysis of Mercury by
Cold-Vapor Atomic Absorption Spectroscopy (CVAAS)

In this procedure, divalent mercury (Hg++) in aqueous samples (digests of water, tissue or sediment samples) is reduced to the elemental state (Hgo) by a strong reducing agent (stannous chloride). Gaseous Hgo enters the sweep gas and is introduced into an atomic absorption cell, where light produced by a mercury vapor lamp is absorbed by the free Hg atoms. Mercury in the sample is determined by comparing light absorption of the sample with that of external calibration standards.

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Lab Name: *Trace Element Research Laboratory* Method Code: *008*

Analysis of Trace Metals by
Flame Atomic Absorption Spectroscopy (FAAS)

Flame atomic absorption spectrophotometry relies on a flame to provide the heat necessary to evaporate the solvent (water) and break molecular bonds in order to produce a cloud of free atoms in the path of an atomic absorption spectrophotometer. In general, the method is rapid, sensitive, and free from inter-element interferences. Prior to analysis, samples must be digested using a method that is appropriate for the matrix. Depending upon element, the fuel (acetylene) is burned with either air or nitrous oxide as the oxidant. Background correction is used to compensate for molecular absorption and light scattering by solids.

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Lab Name: *Trace Element Research Laboratory* Method Code: *009*

Analysis of Trace Metals by
Atomic Fluorescence Spectroscopy (AFS)

Aqueous samples (including sample digests) are analyzed for mercury and hydride-forming elements (antimony, arsenic, and selenium) by atomic fluorescence. Analytes are introduced to the gas phase by reaction with a strong reducing agent

(e.g. stannous chloride for mercury and sodium borohydride for the other elements), and free atoms are bombarded with light of element-specific wavelengths. Light that is released via atomic fluorsecence is measured by a detector set at a right angle to the source. Because of the low background signal, AFS is extremely sensitive and is appropriate when other methods (e.g. GFAAS) lack the sensitivity to determine ambient concentrations. Spectral interferences are few, but the method is subject to chemical and matrix interferences that may impact the cold-vapor and hydride generation steps.

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Lab Name: *Trace Element Research Laboratory* Method Code: *010*

Analysis of Trace Metals in Biological Tissue, Soils, and Sediments by Instrumental Neutron Activation Analysis (INAA)

Biological tissue, soil, and sediment samples are freeze dried, powdered, weighed into poly vials and exposed to thermal neutrons. Depending upon target analyte and level of background radiation from interfering nuclides, samples are allowed to cool for a period of time before counting on high-resolution gamma ray detectors. Concentrations are determined by comparison of counts with those of external calibration standards. This method avoids sample digestion problems that may occur with certain analytes and sample matrices.

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Lab Name: *Trace Element Research Laboratory* Method Code: *011*

Analysis of Grain Size Distribution in Soil and Sediment Samples (GS)

This method incorporates wet-sieving and the pipette method to separate soil and sediment samples into sand, silt, and clay size fractions. A 0.0625 mm sieve is used to collect the sand fraction, which is dried and weighed. Material passing the sieve is diluted to volume and at given times, small volumes of suspension are withdrawn, evaporated, and the residue weighed. Values are expressed as percent of the dry sample on a weight:weight basis.

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Lab Name: *Trace Element Research Laboratory* Method Code: *012*

Analysis of Soils and Sediments for Total Organic Carbon (TOC)

Total organic carbon concentrations are determined on freeze-dried (or oven-dried at 40 to 50 C) soil and sediment by combustion in an oxygen atmosphere. The carbon

dioxide that is produced is swept out of the furnace's combustion chamber, passes through a series of filters, traps, and catalysts, and finally is measured by an infrared detector. Samples are quantitated by comparison of peak area with that of external calibration standards. Prior to analysis, inorganic carbon is removed by acidification. TOC is reported as percent of the dry sample on a weight:weight basis.

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Lab Name: *Trace Element Research Laboratory* Method Code: *013*

Analysis of Acid-Volatile Sulfide in Soils and Sediments (AVS)

Determination of the quantity of acid volatile sulfide is part of a technique that has been developed to predict the bioavailability of metals in sediment. This approach is based on the recognition that many metal sulfides are extremely insoluble, and that formation of solid-phase sulfides in anoxic sediments may control the availability of metals to organisms. AVS, defined as sulfides that are converted to H2S upon exposure to 1N HCl at room temperature for 1 hour, is determined by releasing sulfide from soil and sediment particles with HCl, trapping hydrogen sulfide gas in a base, and measuring it by colorimetry. It is critical that samples to be analyzed for AVS are collected without a headspace and subsampled under an inert atmosphere in order to prevent oxidation of AVS by atmospheric oxygen.

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Lab Name: *Trace Element Research Laboratory* Method Code: *014*

Moisture Content of Sediment, Soil, and Tissue Samples

Moisture content is determined by weight loss upon freeze-drying, and is expressed as weight percent of the original wet sample. Depending upon sample size, either the whole sample or a representative aliquot is frozen and then dried under vacuum until a constant weight is attained. Samples are prepared and dried using plastic materials, whenever possible, in order to minimize potential contamination artifacts that might impact subsequent trace element analysis.

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Lab Name: *Trace Element Research Laboratory* Method Code: *015*

Preparation and Analysis of Tissue and Sediment Samples for Methyl Mercury (MeHg)

Methyl mercury and other alkyl mercury compounds are of concern because of their toxicities, and because they are commonly found in the environment.

Bioaccumulation results in elevated concentrations in higher trophic levels, especially when lower trophic levels include fish. The procedure used to extract these compounds in the Trace Element Research Laboratory follows the method of Uthe et al. (JAOAC 55: 583-589, 1972), and measures the sum of all organo-mercury species extracted into the solvent. This determination is essentially equivalent to the GC method for analyzing MeHg in fish muscle tissue (where almost all of the organo mercury is present as MeHg). In other organs, such as kidneys, much of the organic mercury may be present as a form other than MeHg, and may not be measured by methods that employ detectors that are specific for halogenated compounds. Samples are analyzed either wet or after freeze-drying. Homogenized aliquots are extracted in to an organic solvent with potassium bromide and copper sulfate added to improve partitioning between phases. The organic phase is digested in combusted glass vials, using nitric and sulfuric acids and potassium permanganate, in order to convert all mercury species to ionic mercury and to remove traces of organic solvent that would otherwise impact the measurement. Analysis is based upon the cold vapor atomic absorption method, although cold vapor atomic fluorescence can be used when lower detection limits are required.

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Lab Name: *Trace Element Research Laboratory* Method Code: *016*

Analysis of Trace Metals in Water Samples by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS)

Concentrations of trace elements in water samples are determined with an atomic spectroscopy method that relies on ionization of sample constituents in a high temperature argon plasma and separation of positively-charged ions on the basis of their mass:charge ratios (m:z) by a quadrupole mass spectrometer. The method offers extremely low detection limits but is subject to interferences from atomic and molecular ions having values within 1 AMU of the target ions. Sample preconcentration and matrix elimination can sometimes eliminate these problems, along with those resulting from high total dissolved solids.

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Lab Name: *Trace Element Research Laboratory* Method Code: *017*

Analysis of Inorganic Arsenic (As) in Tissue and Sediment Samples

Inorganic As species [As(III) and As(V)] are extracted from homogenized samples using the method of Willie (Spectrochim. Acta Part B, 51:1781-1790; 1996) and analyzed by hydride-generation atomic fluorescence spectrometry (HGAFS). A single result is reported (inorgAs) since the analysis involves a reduction of As(V) to As(III) prior to hydride generation. Small sample sizes can be accommodated by modifying extraction and analysis solution volumes. Development of reference

materials certified for metal species is not yet complete, so spiked samples provide the best estimates of accuracy at this time.

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Lab Name: *Trace Element Research Laboratory* Method Code: *018*

Analysis of Inorganic Species and Organic Arsenic in Tissues and Sediment Samples

Inorganic As species [As(III) and As(V)] are extracted from freeze- dried tissue with methanol-water and from freeze-dried sediment with 0.3 M phosphoric acid. An anion exchange resin is used to separate As(III) from As(V), and the inorganic species are analyzed by hydride generation atomic fluorescence spectroscopy. Organic As is calculated as the difference in total As and the sum of the inorganic forms [As(III) + As(V)].

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Lab Name: *Trace Element Research Laboratory* Method Code: *019*

Preconcentration of Water Samples for Trace Element Analysis

Water samples with severe matrix interferences and/or requiring detection limits below instrument DL's can be preconcentrated by one of several methods. Ideally, this separates analytes from matrix components while increasing analyte concentrations in solution. Prior to preconcentration, samples are digested with nitric acid and uV irridation. Depending upon analyte requirements, digested samples are preconcentrated by ion exchange, reductive coprecipitation, iron hydroxide precipitation, or chelation/solvent extraction. Resulting solutions are generally analyzed by either GFAAS, ICP, or ICP-MS.

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Lab Name: *Trace Element Research Laboratory* Method Code: *020*

Determination of Total Suspended Solids

Total suspended solids (TSS) in water samples is determined gravimetrically. Samples are vacuum-filtered through preweighed 0.45 micrometer pore size membrane filters, salts are rinsed from the filters with reagent water, and filters are reweighed after drying. TSS is reported in ppm (mg/liter).

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Lab Name: *Trace Element Research Laboratory* Method Code: *021*

Nitrogen

Analysis of nitrogen in tissue, soil, and sediment samples. Nitrogen (N) is determined colorimetrically after digestion of freeze dried samples. Results are reported in parts per million (ppm) on a dry weight basis.

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Lab Name: *Trace Element Research Laboratory* Method Code: *022*

Digestion of Biological Tissue in Microwave Oven Under Pressure

Liquid or solid biological tissue samples are wet digested with nitric acid and hydrogen peroxide and then converted into acidic digest solutions for analysis by various atomic spectroscopy methods. When possible, tissue is freeze dried in order to minimize loss of analytes and to facilitate subsequent sample preparation steps, and then homogenized to a fine powder by ball-milling in plastic containers. Approximately 0.20 to 0.25 g of powdered tissue is weighed into a Teflon reaction vessel and HNO3 (3 ml) is added. The closed reaction vessel is heated in a microwave oven at 160 psi and approximately 180 degrees C until digestion is complete. H2O2 (2 ml) is added to the sample and it is heated gently at ambient pressure to digest residual lipids. The samples is then diluted to a final volume of 20 ml with deionized water and stored in a 30 ml polyethylene bottle for later analysis by instrumental techniques.

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Lab Name: *Trace Element Research Laboratory* Method Code: *023*

Digestion of Biological Tissue in Microwave Oven Under Ambient Pressure

Liquid or solid biological tissue samples are wet digested with nitric acid and hydrogen peroxide and then converted into acidic digest solutions for analysis by various atomic spectroscopy methods. When possible, tissue is freeze dried in order to minimize loss of analytes and to facilitate subsequent sample preparation steps, and then homogenized to a fine powder by ball-milling in plastic containers. Approximately 0.2 g of powdered tissue is weighed into a polypropylene tube and HNO3 (2 ml) is added. The tube is capped with a vented lid and allowed to stand overnight to solubilize the sample. The sample is heated in a microwave oven at 100 degrees C for 20 minutes and then allowed to cool. H2O2 (0.25 ml) is added and after standing again overnight the sample is heated at 100 degrees C for 1 hour. Digest volume is determined gravimetrically and the sample is diluted as necessary with deionized water for analysis by instrumental techniques.

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Lab Name: *Trace Element Research Laboratory* Method Code: *024*

Determination of Mercury in Tissue and Sediment

Determination of mercury in tissue and sediment samples by decomposition, trapping, and atomic absorption. Total mercury is determined in wet or dry samples by combustion in a stream of oxygen, trapping on a gold column, release by electrothermal heating, and analysis by atomic absorption. Mercury is reported in ppm on either a wet or dry weight basis.

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Lab Name: *Trace Element Research Laboratory* Method Code: *025*

Determination of Mercury in Water

Determination of mercury in water by purging, trapping, and atomic fluorescence. Total mercury is determined in water by oxidation with BrCl followed by reduction of Hg (II) to Hg(0) with SnCl2. Hg(0)g is purged from the aqueous sample with argon and trapped on a gold column. The trapped Hg is released by heating and then analyzed by atomic fluorescence.

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Lab Name: *Trace Element Research Laboratory* Method Code: *026*

Determination of Methyl Mercury in Water

Determination of methyl mercury in water by distillation, ethylation, trapping, gas chromatography, and atomic fluorescence. Methyl mercury in water is distilled to separate it from interfering species and then ethylated with sodium tetraethyl borate. Methyl ethyl mercury is trapped on a Tenax column and then separated on an isothermal GC column. Following pyrolysis of the separated species, Hg is detected by atomic fluorescence.

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Lab Name: *Trace Element Research Laboratory* Method Code: *027*

Determination of Perchlorate in Water

The perchlorate anion is determined in water by ion chromatography. Perchlorate is separated and measured, using a system comprised of an ion chromatographic pump, sample injection valve, guard column, analytical column, suppressor device, and conductivity detector.

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Lab Name: *Trace Element Research Laboratory* Method Code: *028*

Determination of Anions in Water by Wet Chemistry Procedures

Anions are determined by classical wet chemistry procedures. Chloride ion is measured by titrating the sample with a standardized silver nitrate solution. Bicarbonate is determined by electrochemical titration to a pH of 4.5. Sulfate is determined turbidimetrically following formation of a barium sulfate precipitate.

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Lab Name: *Trace Element Research Laboratory* Method Code: *029*

Determination of carbon, nitrogen, and/or stable C and N isotopes by isotope ratio mass spectroscopy

Stable istopes of C and N, as well as C and N content, are determined by isotope ratio mass spectroscopy. Samples are combusted under oxygen, converting C to CO2 gas and N to N2 gas. Ratios of the CO2 and N2 species are determined on a multicollector isotope ratio mass spectrometer. Values are reported as "delta 13C" and "delta 15N", in "per mil" (parts per thousand", relative to the PDB standard for C and air for N. Inorganic species can be removed by vapor acidification prior to analysis in order to measure only organic species.

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