DRAFT

ENVIRONMENTAL CONTAMINANT IMPACTS OF HIGHWAY RUNOFF ON FRESHWATER MUSSELS SWIFT CREEK, NASH COUNTY, NORTH CAROLINA

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MEMORANDUM

Date: December 18, 1992

From: Field Supervisor, Ecological Services, Raleigh Field Office, Raleigh, NC

Subject: ENVIRONMENTAL CONTAMINANT IMPACTS OF HIGHWAY RUNOFF ON FRESHWATER MUSSELS, SWIFT CREEK, NASH COUNTY, NORTH CAROLINA

To: Dr. Charles Facemire, Regional Environmental Contaminants Coordinator, Ecological Services, Atlanta, GA (AWE)

This memorandum documents transmittal of the enclosed draft report: "Environmental Contaminant Impacts of Highway Runoff on Freshwater Mussels, Swift Creek, Nash County, North Carolina." This study was initiated to examine one potential causative factor in the decline of freshwater mussels in North Carolina, environmental contaminants from highway runoff.

We are releasing the report in draft for a 30-day period for peer review and comment. Reviewers will include Kate Benkert, former Raleigh Field Office Contaminant Specialist, Dick Biggins, Region 4 Mussel Coordinator in the Asheville Office, and John Alderman of the North Carolina Wildlife Resources Commission who assisted with sample collection. In preparing the final report, we will address or incorporate comments from solicited reviewers. When finalized, this report will complete work performed under environmental contaminants study identifiers 90-4-066A, 90-4-066B, 90-4-066C, and 90-4-066D and contaminants catalog numbers 6271, 6272, 6361, and 6362.

Please provide us with copies of the standard report cover, mentioned in your September 18, 1992 memorandum on the subject, for the final report. Any review comments you have also are welcome. If you have any questions regarding this memorandum or the draft report, please contact Tom Augspurger of this office at (919) 856-4520.

for Mike Gantt

MEMORANDUM

TO: Tom Augspurger, Raleigh Field Office

FROM: Dick Neves, Virginia Unit

SUBJECT: Draft Report on Highway Contaminants

DATE: February 8, 1993

Many thanks for forwarding a copy of this report to me. I apologize for my tardy response, but the piles of reading materials overwhelmed me. This report is an excellent first cut at identifying contaminants likely to jeopardize mussel populations below major roadways. I learned a lot both in terms of techniques for sediment analysis and the array of contaminants that should be sought in future analyses, particularly the PAH's. If depuration occurs fairly rapidly once the aliphatic hydrocarbons decline in the water, then levels shortly after storm events would need to be measured wherever highway runoff is suspected of being a problem.

A couple points need clarification:

1. What was the size range of mussels tested?

2. Methods say "5 to 12 mussels" but Table 3 says n = 3?

3. How do the body burdens compare with toxicity levels in other organisms? Are there any red flags in Table 3 or 4?

Although sample sizes were small, I hope that you submit a manuscript for publication. There are so few data on body burdens for mussels that even preliminary studies are worthy of publication. Thanks again for sharing this report.

RJN/akb

PREFACE

This study was designed and conducted by Kathryn A. Benkert, an Environmental Contaminants Specialist formerly of the Raleigh Field Office and now with the Service's Ecological Services Office in Olympia, Washington. John Alderman and Christopher McGrath of the North Carolina Wildlife Resources Commission assisted with sample collection. Questions, comments, and suggestions related to this report are encouraged; written inquires should be directed to the Service at the following address:

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ABSTRACT

Sediments and freshwater mussels, *Elliptio complanata*, were collected upstream and downstream of the Interstate 95 (I-95) crossing of Swift Creek, Nash County, North Carolina in 1990 to identify contaminants present near highway stream crossings and to assess the impacts of highway runoff waters on freshwater mussels. Sediments were analyzed for twelve elemental contaminants, oil and grease, petroleum hydrocarbons, and polycyclic aromatic hydrocarbons (PAHs). Composite samples of the soft tissues from mussels were analyzed for elemental contaminants, petroleum hydrocarbons, and PAHs.

Total aliphatic hydrocarbons, total PAHs, oil and grease, arsenic, lead, selenium, chromium, copper, iron, manganese, nickel and zinc were elevated in sediments collected downstream of I-95 relative to upstream reference samples. All of these contaminants except nickel, chromium, and total PAHs were significantly ($p<0.05$) elevated. Average concentrations of sediment oil and grease (1060 ppm-dry weight), arsenic (3.02 ppm), lead (26.0 ppm), iron (19900 ppm), and chromium (36.5 ppm) downstream of I-95 exceeded or approached U.S. Environmental Protection Agency or National Oceanic and Atmospheric Administration sediment quality screening guidelines for contaminated sediments and biological effects.

Total aliphatic hydrocarbons, lead, and mercury were elevated in mussels collected downstream of I-95 relative to upstream reference samples; all other analytes were higher in the reference mussels. Although elevated, average concentrations of lead and mercury in mussels downstream of I-95 appear to be of minimal toxicological significance. Total aliphatic hydrocarbons (average 0.70 ppm-wet weight; maximum 1.19 ppm) were over 20 times greater in mussels downstream of I-95 relative to upstream reference specimens; levels in downstream mussels indicate chronic low level aliphatic hydrocarbon pollution. Because of the fast depuration rate of aliphatic hydrocarbons, levels well above those identified in this study may be present in mussels receiving highway runoff following rainfall.

The overall small number of samples, differing sizes of mussels from the study area and reference site, and composition of sediments from these two areas may mask or exaggerate differences in contaminant burdens up- and downstream of I-95. However, it appears from this initial reconnaissance that highway crossings of important freshwater mussel habitat are a source of sediment contamination that could lead to low level mussel contamination and stress. Recommendations for additional study include sampling a larger number of mussels and sediments for a select group of the contaminants detected in this work, and analyzing stormwater runoff and receiving water collected during storms. These studies should incorporate analysis of sediment total organic carbon and grain size, and target similar sized mussels above and below bridges, to aid in interpretation of contaminant residues.

INTRODUCTION

Stormwater runoff from highways is recognized as a source of water and sediment contaminants, including elemental contaminants, petroleum hydrocarbons, and polycyclic aromatic hydrocarbons (PAHs) (Vestal 1980; Hoffman et al. 1985; Schiffer 1989; Yousef et al. 1990). The significance of this type of pollution on receiving waters adjacent to highways depends on several factors including traffic volume, size of the watershed, paved surface area within the watershed, stormwater management practices, and rainfall volume, intensity, duration, and interval (Strecker et al. 1990). Runoff from high traffic areas through small watersheds may account for the majority of a stream's annual pollutant loading (Hoffman et al. 1985).

Contamination of water and sediments are major threats to the survival of freshwater mussels, Unionidae. Mussels are exposed through ingestion by filter feeding and direct contact with contaminated sediments onto which chemicals have adsorbed. Declines in native freshwater mussel populations have been attributed to deterioration of water quality, waterway modifications, streambed changes, commercial exploitation, and competition from exotic mollusks (Fuller 1974; Havlik and Marking 1987; U.S. Fish and Wildlife Service 1991; McGrath 1992).

In mussel surveys conducted by the North Carolina Wildlife Resources Commission, it has been determined that in Swift Creek, a tributary to the Tar River in Nash County, North Carolina, the number of mussel species decreases dramatically downstream of the Interstate 95 (I-95) bridge. At least nine species are present immediately above the bridge, while only the pollution tolerant Asiatic clam (*Corbicula* sp.) and *Elliptio complanata* are present below the bridge (J. Alderman, North Carolina Wildlife Resources Commission, personal communication). Average daily traffic along I-95 at the Swift Creek crossing is about 30,000 vehicles with a maximum daily traffic volume measured at 55,988 (North Carolina Department of Transportation 1990, 1991).

The purpose of this study is to examine one potential causative factor in the decline of freshwater mussels in North Carolina: environmental contaminants from highway runoff. The study design included two objectives both of which will assist wildlife managers and mussel recovery teams in future management decision making:

1) identify toxic contaminants present in stream sediments and mussels below highways; and,

2) assess the impacts of highway runoff waters on freshwater mussels.

METHODS

Sediment and freshwater mussels, *Elliptio complanata*, were collected upstream and downstream of the I-95 crossing of Swift Creek, Nash County, North Carolina on June 8, 1990. Average daily traffic during the sampling period was 28,800 vehicles (North Carolina Department of Transportation 1990).

Downstream of I-95, mussels were collected between the I-95 and NC 48 bridges near Gold Rock (Figure 1). Sediments were collected downstream of the NC 48 bridge due to scouring of stream sediments above the NC 48 bridge where mussels were collected. The reference site was located approximately 1/4 mile upstream of the SR 1310 bridge at Hilliardston (Figure 1). This site has a U.S. Geological Survey gauging station and is used by the North Carolina Division of Environmental Management as a reference site for their water quality monitoring programs.

Elliptio complanata were collected by hand with the assistance of SCUBA gear. Composite samples of between 5 and 12 mussels were placed on ice in the field. They were prepared for analysis the same day at the Service's Raleigh Field Office lab by opening and removing all soft tissues.

Sediments were collected using a core sampler constructed from two-inch inside diameter PVC pipe. Samples were collected by inserting the pipe to a depth of about three inches. Five cores were collected per station and mixed together in a stainless steel pan. A sub-sample of the composite sediment sample was packaged and placed on ice in the field. Those sediment samples for organic analyses were placed in chemically cleaned glass jars. Samples for metal analyses were packaged in plastic ziploc bags.

Sampling equipment and dissection tools were cleaned according to methods in the Resource Contaminant Assessment Handbook (U.S. Fish and Wildlife Service 1986). Items were washed, rinsed with tap water, acetone washed and rinsed with hexane.

Samples were stored frozen until shipment to analytical labs in July and August of 1990. Samples were shipped on dry ice to the Mississippi State Chemical Laboratory at Mississippi State University for analyses of organic compounds; elemental contaminant analyses were performed by Research Triangle Institute, Research Triangle Park, North Carolina.

Mercury concentration was determined via cold vapor reduction atomic absorption spectroscopy (AAS). Arsenic and selenium levels were determined by hydride generation AAS, and lead levels were determined by graphite furnace AAS. Other elemental contaminants reported were analyzed by inductively coupled plasma emission spectroscopy. Aliphatic hydrocarbon analyses were done by capillary column, flame ionization gas chromatography (GC); PAH analyses were performed by flame ionization GC and fluorescence high pressure liquid chromatography. Oil and grease was determined gravimetrically. Detailed description of the analytical methodologies are provided in Appendix A along with sample preparation procedures. Contaminants analyzed and method detection limits are provided in Tables 1 and 2.

Quality assurance/quality control (QA/QC) samples, including blanks, spiked samples, reference material analysis, and duplicate analyses, were performed for all analytes. Review of QA/QC samples indicates precision and accuracy were acceptable for all analytes reported except n-dodecane and napthalene. Recovery of napthalene and n-dodecane in spiked sediments was 53 percent and 52 percent respectively; therefore, results reported as below detection for these analytes may be due to method performance.

Contaminant levels in sediment samples from upstream and downstream of the I-95 crossing were compared with a t-Test (Freese 1967). The small number of mussel samples collected for this initial screening survey was not conducive to statistical interpretation.

RESULTS

The range and average contaminant concentrations in sediment are presented in Table 3. Total aliphatic hydrocarbons, total PAHs, oil and grease, arsenic, lead, selenium, chromium, copper, iron, manganese, nickel and zinc were elevated in sediments collected downstream of I-95 relative to upstream reference samples.

The range and average contaminant concentrations in mussels are presented in Table 4. Total aliphatic hydrocarbons, lead, and mercury were elevated in mussels collected downstream of I-95 relative to upstream reference samples; all other parameters were higher in the reference mussels.

DISCUSSION

Sediments

As a first approximation of the extent of sediment contamination, constituent concentrations were compared to sediment quality guidelines established by various State and Federal agencies and countries (Table 5). National Oceanic and Atmospheric Administration (NOAA) environmental effects values are used by NOAA to assess potential biological effects of contaminated sediments as part of their national monitoring program. The NOAA defines ER-L values as the low end of the range of concentrations above which adverse effects may begin or are predicted among sensitive species (Long and Morgan 1990). Although derived primarily from a database of marine systems, some freshwater toxicity information was used in the development of the environmental effects values. Because biological effects are generally expected to be more significant at a given level of contamination in freshwater versus marine environments, this approach will be somewhat less conservative than reliance on a database solely composed of freshwater toxicity information. Other guidelines offered in Table 5 were derived for screening dredged sediments proposed for freshwater disposal (Beyer 1990; Bennett and Cubbage 1991). These values are guidelines and screening measures only; there are, as yet, no promulgated State or Federal sediment quality criteria.

Although oil and grease, total aliphatic hydrocarbons, total PAHs, arsenic, lead, selenium, chromium, copper, iron, manganese, nickel, and zinc are all elevated below I-95, only oil and grease, arsenic, lead, iron, and chromium exceed or approach sediment screening values. Three of the five up-gradient samples also exceeded the chromium screening level as did one of the iron values and one arsenic value (5.16 ppm), the highest detected in this study. This may indicate low level sediment arsenic contamination at the reference location.

The sediment guidelines do not adjust or standardize the residue levels for differences in sediment grain size or organic matter content. A number of studies have illustrated the positive correlation between the organic carbon content of sediments and their capacity to adsorb contaminants (Anderson et al. 1987; Rodgers et al. 1987). The variability of organic matter content and particle size in Swift Creek sediments is not known; consequently, up- and downstream differences as well as exceedences of sediment screening guidelines should be viewed cautiously.

Raw data for elemental contaminants in sediment were manipulated in an attempt to account and adjust for normal environmental heterogeneity so that runoff induced perturbations of the system could be identified. Sediment trace metal data were analyzed via a concentration ratio technique, described in White and Tittlebaum (1984) and Smith et al. (1987), to reduce the effects of variable sediment composition (organic matter content, particle size distribution, etc.) on trace element concentrations. Whereas trace metals are naturally present in low concentrations which can be easily enhanced by anthropogenic activities, the levels of "conservative" elements, defined as metals of low environmental variability which are unlikely to be elevated in sediments from human activities, are naturally high by comparison. Even if anthropogenically elevated, conservative elements are less sensitive to change owing to their high sediment concentrations. A ratio of a sediment's trace metal content to its level of a conservative metal may normalize the samples for differences in trace element concentration between two locations due solely to environmental heterogeneity. The conservative metals chosen for this study were aluminum and manganese because their concentrations in sediments are high relative to the trace metals of interest (Smith et al. 1987) and manganese does not appear to be a major component of highway runoff (Hoffman et al. 1985). Comparison of upstream versus downstream ratios of arsenic, lead, chromium, copper, iron, and zinc to the conservative elements reveals no consistent pattern of contamination below I-95.

Mussels

Mussels have been used successfully in bioaccumulation studies to identify sources of organic and inorganic contamination (Foster and Bates 1978; Czarnezki 1987; Schmitt et al. 1987; Green et al. 1989). However, there is only limited toxicity data relevant to evaluating the significance of elevated contaminant burdens in freshwater mussels (Havlik and Marking 1987). In this study, lead and mercury were slightly elevated in mussels collected downstream of I-95 relative to upstream reference samples. Although elevated, average concentrations of these metals in mussels below I-95 are comparable to or below those reported for freshwater mussels in other studies (Schmitt et al. 1987; Eaton et al. 1991) and appear to demonstrate minimal bioaccumulation.

The most significant observation in this assessment is the greater than 20-fold elevation of total aliphatic hydrocarbons in mussels collected downstream of I-95 relative to upstream samples. Low inter-species variation in contaminant burdens of freshwater mussels (Muncaster et al. 1990) indicates that the aliphatic hydrocarbon burden of common elliptio documented in this study represents a threat to other mussels downstream of major highways.

Total aliphatic hydrocarbon concentrations in mussels were over three times those in sediments indicating significant bioaccumulation of these compounds. In a comprehensive evaluation of aliphatic hydrocarbons in the brackish water rangia clam, *Rangia cuneata*, throughout northeastern coastal North Carolina, Benkert (1992) found levels comparable to those of the common elliptio from this study only below the large industrial wastewater discharge of the pulp and paper mill at Plymouth.

In aliphatic hydrocarbon analysis of mussels, n-heptadecane, pristane, n-octadecane, phytane, n-nonadecane, and n-eicosane comprised the majority of the total aliphatic hydrocarbons. The aliphatic hydrocarbons are a class of chemicals found in petroleum and gas deposits and can enter aquatic systems from point and non-point source discharges. Point sources may include wastewater treatment plants, industrial effluent, spills, and exhaust from gasoline powered engines (Verschueren 1983). Non-point discharges occur from highway runoff and urban stormwater runoff (Vestal 1980; Hoffman et al. 1985). Aliphatic hydrocarbons can also occur naturally in aquatic systems from hydrocarbon deposit seepage (Verschueren 1983), but this source is presumed to be absent in the North Carolina Piedmont.

The lower weight aliphatic hydrocarbons ($<C_{18}$) volatilize easily. The remaining non-volatilized residue often can be metabolized by microbes. The uptake of aliphatic hydrocarbons by aquatic invertebrates is dependent upon temperature and concentration and is usually rapid. A steady-state level of aliphatic hydrocarbons in an organism is achieved relative to the concentration in the water. Depuration occurs fairly rapidly once the aliphatic hydrocarbon concentration declines in the water. The depuration process also is temperature dependent (Verschueren 1983).

Marine invertebrates in an environment of chronic aliphatic hydrocarbon exposure have residues ranging from 1 to 150 ppm-ww (Moore and Ramamoorthy 1984). Using this as a freshwater screening value, residues in mussels from Swift Creek downstream of I-95 indicate chronic low level aliphatic hydrocarbon pollution. Because of the fast depuration rate of these compounds, levels well above those identified in this study may be present in mussels receiving highway runoff following a storm event. There was no precipitation recorded at the monitoring station 6 miles southwest of Rocky Mount for the two days prior to sampling associated with this study (J. Enman, State Climatologist's Office, personal communication). There was only 0.05 inch of rainfall recorded at this station in the three days before sample collection. The importance of the "first flush" of stormwater runoff in delivering a contaminant loading is well-documented (Vestal 1980; Hoffman et al. 1985).

Research emphasizes the importance of the dissolved phase of chemicals in surface waters for organic compound accumulation by mussels (Pruell et al. 1986; Muncaster et al. 1990). Future studies at the I-95 bridge crossing should include stormwater runoff collection and analysis as well as surface water collection during storms.

The average size of mussels collected above and below I-95 may be a confounding factor in trend interpretation. Copper and polychlorinated

biphenyl concentrations in mussels have been shown to be inversely correlated with organism weight (Foster and Bates 1978; Muncaster et al. 1990) while some elemental contaminant burdens increase with increasing mussel size (Green et al. 1989). Although average mussel size from the composites used in elemental contaminant analyses for this study are comparable for collections upstream (10.2 grams) and downstream (10.5 grams) of I-95, the results are largely influenced by two individual samples. The average mussel weight in the largest composite (15.0 grams) was from below I-95 and the smallest (6.0 grams) was from the upstream reference station. The composite of larger mussels from downstream had the highest lead concentration and the composite of small specimens from upstream had the lowest lead burden. With a sample size of three at each station, these two samples greatly affect the overall average lead burdens.

CONCLUSIONS AND RECOMMENDATIONS

The overall small number of samples, differing sizes of mussels from the study area and upstream reference site, and composition of sediments from these two areas targeted in this initial reconnaissance survey may mask or exaggerate differences in contaminant burdens upstream and downstream of I-95. However, it appears from this initial reconnaissance that highway crossings of important freshwater mussel habitat are a source of sediment contamination that could lead to low level mussel contamination and stress. Before the significance of the threat to freshwater mussels can be determined, a larger number of mussels and sediments should be collected and analyzed for a select group of the contaminants detected in this work, namely aliphatic hydrocarbons and elemental contaminants. Analysis of stormwater and receiving water collected during storms is also advised. These studies should incorporate analysis of sediment total organic carbon and grain size, and target similar sized mussels above and below bridges, to aid in interpretation of contaminant residues.

Figure 1. Freshwater mussel and sediment sampling locations for the highway runoff assessment at Swift Creek, Nash County, North Carolina.

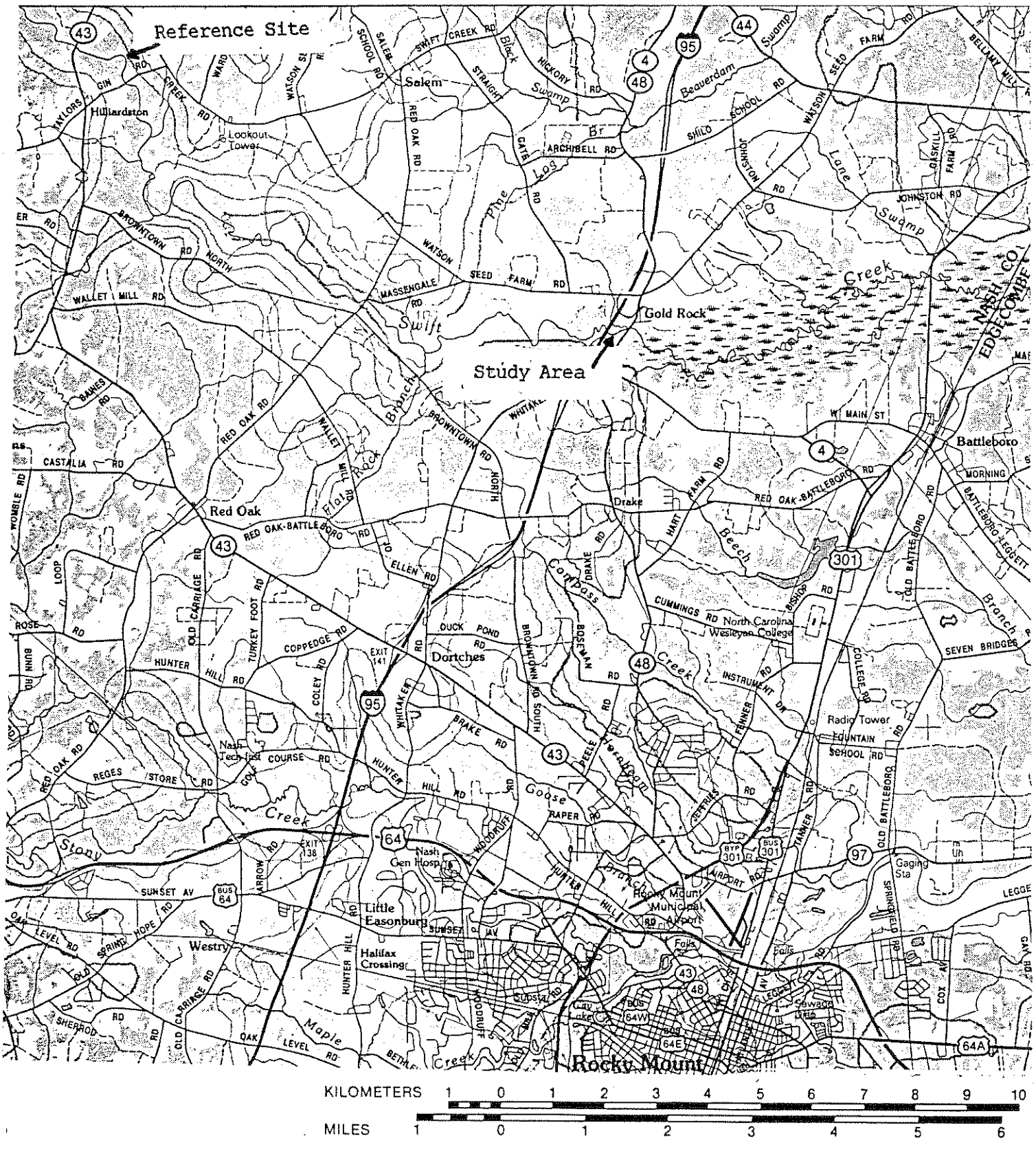

Table 1. Method detection limits of elemental contaminants analyzed in this study (µg/g dry weight).

Elemental Contaminant	Detection Limits Tissue	Sediment
mercury	0.02	0.05
arsenic	0.3	0.3
selenium	0.3	0.3
lead	0.2	0.2
aluminum	5.0	100
barium	5.0	2.5
cadmium	0.1	0.5
chromium	0.5	3.0
copper	0.5	3.0
iron	10	100
nickel	0.8	4.0
zinc	1.0	5.0

Table 2. Organic chemicals analyzed in this study and method detection limits.

Aliphatic Hydrocarbons	Polycyclic Aromatic Hydrocarbons	Miscellaneous
n-Dodecane	Naphthalene	Oil and Grease
n-Tridecane	Fluorene	
n-Tetradecane	Phenanthrene	
Octylcyclohexane	Anthracene	
n-Pentadecane	Fluoranthrene	
Nonylcyclohexane	Pyrene	
n-Hexadecane	1,2-Benzanthracene	
n-Heptadecane	Chrysene	
Pristane	Benzo(b)fluoranthrene	
n-Octadecane	Benzo(k)fluoranthrene	
Phytane	Benzo(e)pyrene	
n-Nonadecane	Benzo(a)pyrene	
n-Eicosane	1,2,5,6-Dibenzanthracene	
	Benzo(g,h,i)perylene	

Method detection limits for all compounds = 0.01 µg/g wet weight.
Method detection limit for oil and grease = 10 µg/g wet weight.

Table 3. Contaminant concentrations in sediments collected from upstream and downstream of the I-95 crossing of Swift Creek, Nash County, North Carolina.

ANALYTE	UPSTREAM (n=5)		DOWNSTREAM (n=5)	
	Average	Range	Average	Range
Organics (µg/g dry weight)				
Aliphatic Hydrocarbons*	0.15	(0.05-0.25)	0.63	(0.15-1.27)
Σ PAHs	0.06	(0.03-0.10)	0.11	(0.05-0.14)
Oil and Grease+	423	(328-594)	1060	(600-1410)
Inorganics (µg/g dry weight)				
As	2.09	(1.14-5.16)	3.02	(2.29-3.98)
Pb+	18.8	(14.9-21.4)	26.0	(22.0-29.5)
Se	----	(<0.3)	0.57	(0.30-0.71)
Hg	----	(<0.05-0.088)	----	(<0.05-0.088)
Al	11800	(2430-20000)	11600	(6220-18100)
Cd	----	(<0.50)	----	(<0.50)
Cr*	28.8	(17.4-38.9)	36.5	(27.7-47.1)
Cu*	9.72	(5.50-12.9)	14.4	(11.1-17.5)
Fe	13600	(9180-19300)	19900	(15900-26300)
Mg*	580	(100-1000)	526	(300-710)
Mn	449	(333-530)	1090	(685-1820)
Ni	12.4	(7.80-15.9)	14.8	(11.2-18.6)
Zn+	34.7	(26.1-42.0)	53.5	(44.2-64.5)

* significantly elevated down-gradient ($p<0.05$)
\+ significantly elevated down-gradient ($p<0.01$).

Table 4. Contaminant concentrations in mussels collected from upstream and downstream of the I-95 crossing of Swift Creek, Nash County, North Carolina.

ANALYTE	UPSTREAM (n=3)		DOWNSTREAM (n=3)	
	Average	Range	Average	Range
Organics (µg/g wet weight)				
Aliphatic Hydrocarbons	0.03	(0.01-0.04)	0.70	(0.04-1.19)
Σ PAHs	0.03	(0.02-0.05)	----	(<0.01-0.06)
Inorganics (µg/g dry weight)				
As	2.91	(1.90-4.32)	1.79	(1.71-1.92)
Pb	1.82	(1.39-2.09)	2.39	(1.71-3.02)
Se	2.05	(1.77-2.23)	1.96	(1.95-1.98)
Hg	0.552	(0.471-0.611)	0.687	(0.606-0.801)
Al	432	(332-509)	415	(174-857)
Cd	1.74	(1.42-2.23)	1.36	(1.28-1.50)
Cr	6.18	(3.81-8.03)	4.59	(2.52-6.32)
Cu	8.77	(7.30-10.2)	6.12	(5.45-7.42)
Fe	9860	(3990-15500)	8510	(2860-12800)
Mg	1110	(934-1270)	1050	(1030-1090)
Mn	4910	(4390-5190)	4300	(3730-4700)
Ni	1.33	(1.08-1.75)	----	(<0.8-1.81)
Zn	143	(119-163)	136	(114-164)

Table 5. Sediment quality screening guidelines (mg / kg dry weight) for contaminants elevated downstream of the I-95 crossing of Swift Creek relative to upstream reference sediments.

Parameter	As	Pb	Cr	Cu	Fe	Ni	Zn	PAHs	Oil / Grease
Swift Creek Below I-95[1]	3.02	26	36.5	14.4	19900	14.8	53.5	0.11	1060
NOAA ER-L	33	35	80	70	NA	30	120	4	NA
Great Lakes Harbors[2]	3-8	40-60	25-75	25-50	17000-25000	20-50	90-200	NA	1000-2000
Wisconsin DNR[3]	10	50	100	100	NA	100	100	NA	1000
Ontario MOE[4]	8	50	25	25	NA	25	100	NA	1500

[1] Average concentrations downstream of I-95 from this study
[2] Guidelines for the pollution classification of Great Lakes harbor sediments; range presented is described as "moderately polluted" (Beyer 1990)
[3] Wisconsin interim criteria for sediments from Great lakes harbors for disposal in water; criteria not to be exceeded (Bennett and Cubbage 1991)
[4] Ontario Ministry of the Environment guidelines for open lake disposal of sediments (Beyer 1990; Bennett and Cubbage 1991).

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PERSONAL COMMUNICATION

John Alderman, Wildlife Biologist, Division of Wildlife Management, North Carolina Wildlife Resources Commission. July 26, 1989 telephone conversation with Kate Benkert, Raleigh Field Office.

Julia Enman, Assistant State Climatologist, Office of the State Climatologist, Department of Marine, Earth, and Atmospheric Sciences, North Carolina State University. November 2, 1992 letter.

APPENDIX A

Analytical Chemistry Methods for the Highway Runoff Assessment at Swift Creek, Nash County, North Carolina.

METHODOLOGY

SEDIMENT PREPARATION

1. Homogenization. Following freeze drying, samples were ground to approximately 100 mesh using a glass mortar and pestle.

2. Digestion for Inductively Coupled Plasma Emission (ICP) Measurement. Some 0.25 to 0.5 g of sediment were placed in a 120 mL Teflon microwave vessel. One mL each of HCl, HF, and $HClO_4$, and 10 mL HNO_3 were added to the vessel. The vessel was then capped according to the manufacturer's instructions and was 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 was then filtered through Whatman 41 filter paper prior to ICP measurement. An HF resistance torch tip was used for these digests during the ICP measurement.

3. Digestion for Graphite Furnace Atomic Absorption (GFAA) Measurement. Using a CEM microwave oven, 0.25 to 0.5 g of freeze dried tissue were 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 was then diluted to 50 mL with laboratory pure water.

4. Digestion for Hg Measurement by Cold Vapor Atomic Absorption (CVAA). Some 0.25 to 0.5 g of sample were refluxed for two hours in 10 mL HNO_3 (Baker Instra-Analyzed) and diluted to 50 mL with 1% HCl.

MEASUREMENT

1. ICP. ICP measurements were made using a Leeman Labs Plasma Spec I sequential spectrometer.

2. GFAA. GFAA measurements were made using a Perkin Elmer Zeeman 3030 atomic absorption spectrophotometer with an HGA-600 graphite furnace and an AS-60 autosampler.

3. CVAA. Hg measurements were conducted using $SnCl_4$ as the reducing agent. An Instrumentation Laboratories Model 251 AA spectrophotometer was employed.

METHODOLOGY

ANIMAL TISSUE SAMPLE PREPARATION

1. **Homogenization.** These were performed using a Kitchen Aid food processor. Portions were then freeze dried for determination of moisture content and subsequent acid digestion.

2. **Preconcentration Digestion for Inductively Coupled Plasma Emission (ICP) Measurement.** Using a CEM microwave oven, 0.5 g of freeze dried tissue 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 HNO_3 adding the rinsings with 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 10 mL glass volumetric vessel and made to volume with additional laboratory pure water. The flask contents are then immediately transferred to a clean plastic centrifuge or auto sampler tube and centrifuged for 1 minute to precipitate the suspended matter. The sample is now ready for ICP analysis.

3. **Digestion for ICP Measurement.** Using a CEM microwave oven, 0.25 to 0.5 g of freeze dried tissue were 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 was then diluted to 50 mL with 5% HCl.

4. **Digestion for Graphite Furnace Atomic Absorption (GFAA) Measurement.** Using a CEM microwave oven, 0.25 to 0.5 g of freeze dried tissue were 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 was then diluted to 50 mL with laboratory pure water.

5. **Digestion for Hg Measurement by Cold Vapor Atomic Absorption (CVAA).** Some 0.25 to 0.5 g of tissue were refluxed for two hours in 10 mL HNO_3 (Baker Instra-Analyzed) and diluted to 50 mL with 1% HCl.

MEASUREMENT

1. **ICP.** ICP measurements were made using a Leeman Labs Plasma Spec I sequential spectrometer.

2. **GFAA.** GFAA measurements were made using a Perkin Elmer Zeeman 3030 atomic absorption spectrophotometer with an HGA-600 graphite furnace and an AS-60 autosampler.

3. **CVAA.** Hg measurements were conducted using $SnCl_4$ as the reducing agent. An Instrumentation Laboratories Model 251 AA spectrophotometer was employed.

Method 3. 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 °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.

Method 4. Analysis For Aliphatic and 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 for transfer 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.

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, fluoranthrene, pyrene, 1,2-benzanthracene, chrysene, benzo [b] fluoranthrene, benzo [k] fluoranthrene, 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.

Method 10. 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 tranferred with petroleum ether rinsing through a bed of sodium sulfate to 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.