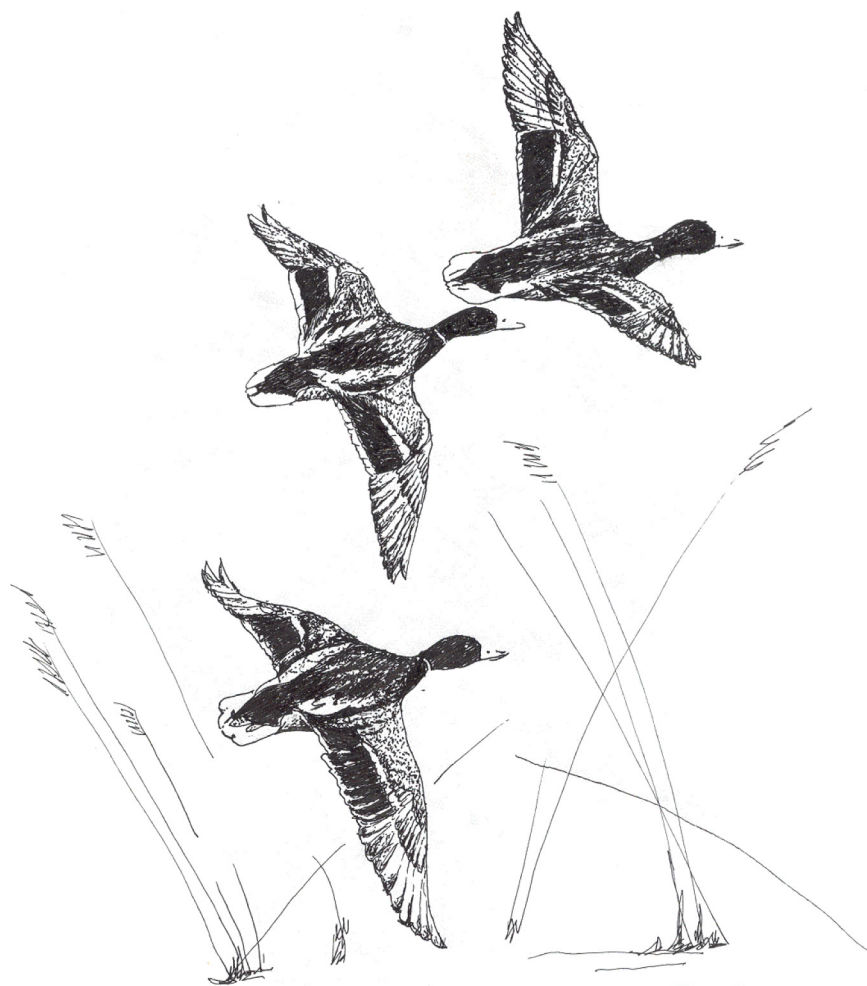


Prepared in cooperation with the
OKLAHOMA DEPARTMENT OF WILDLIFE CONSERVATION and
OKLAHOMA GEOLOGICAL SURVEY

Selected Trace Metals and Organic Compounds and Bioavailability of Selected Organic Compounds in Soils, Hackberry Flat, Tillman County, Oklahoma, 1994-95

Open-File Report 97-828



Report Documentation Page				Form Approved OMB No. 0704-0188	
Public reporting burden for the collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to a penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.					
1. REPORT DATE 1997		2. REPORT TYPE N/A		3. DATES COVERED -	
4. TITLE AND SUBTITLE Selected Trace Metals and Organic Compounds and Bioavailability of Selected Organic Compounds in Soils, Hackberry Flat, Tillman County, Oklahoma, 1994-95				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Department of the Interior 1849 C Street, NW Washington, DC 20240				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release, distribution unlimited					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT SAR	18. NUMBER OF PAGES 11	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

Cover: Art credit: Cover art drawn by Joan Bruns.



Selected Trace Metals and Organic Compounds and Bioavailability of Selected Organic Compounds in Soils, Hackberry Flat, Tillman County, Oklahoma, 1994-95

By Mark F. Becker

Prepared In Cooperation with the Oklahoma Department of Wildlife Conservation and Oklahoma Geological Survey

Open-File Report 97-828

**U.S. Department of the Interior
U.S. Geological Survey**

U.S. Department of the Interior

Bruce Babbitt, Secretary

U.S. Geological Survey

Thomas J. Casadevall, Acting Director

U.S. Geological Survey

For sale by U.S. Geological Survey, Information Services
Box 25286, Denver Federal Center
Denver, CO 80225

District Chief
U.S. Geological Survey
202 NW 66 St., Bldg. 7
Oklahoma City, OK 73116

For more information about the USGS and its products:

Telephone: 1-888-ASK-USGS

World Wide Web: <http://www.usgs.gov/>

Information about water resources in Oklahoma is available on the World Wide Web at
<http://ok.water.usgs.gov>

Any use of trade, product, or firm names in this publication is for descriptive purposes only and does not imply endorsement by the U.S. Government.

Although this report is in the public domain, it contains copyrighted materials that are noted in the text. Permission to reproduce those items must be secured from the individual copyright owners.

UNITED STATES GOVERNMENT PRINTING OFFICE: OKLAHOMA CITY 1998

Contents

Abstract	1
Introduction	1
Description of Study Area	2
Method of Data Collection and Analysis	2
Soil Sampling and Analysis.....	2
Bioavailability sampling and analysis using semipermeable membrane devices	6
Selected Trace Metals and Organic Compounds in Soils	7
Bioavailability of Selected Organic Chemicals	7
Summary	7
Selected References	11

Figures

Figures 1-3. Maps showing:

1. Location of Hackberry Flat near Frederick in Tillman County, Oklahoma.	3
2. The study area and the location of sites where soils were collected and analyzed for trace metals and organic compounds.	4
3. Boundaries of areas where soil samples were combined to form groups for assessment of bioavailable organochlorine pesticides, polychlorinated biphenyls, and polyaromatic hydrocarbons.	5

Tables

1. Total concentrations of selected trace metals of soils from Hackberry Flat, Tillman County, Oklahoma, and median concentrations for soils in the conterminous United States	8
2. Results of analyses of soil from sites H-1 to H-10 for organochlorine pesticides and polychlorinated biphenyls analyses from soils in Hackberry Flat, Tillman County, Oklahoma	9
3. Results of organochlorine pesticide, total polychlorinated biphenyl, and polyaromatic hydrocarbon analyses of dialysates from semipermeable membrane devices for groups 1-12	10

Conversion Factors and Datum

Multiply	By	To obtain
Length		
centimeter (cm)	0.3937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
meter (m)	3.281	foot (ft)
kilometer (km)	0.6214	mile (mi)
Area		
hectare (ha)	2.471	acre
Volume		
liter (L)	0.2642	gallon (gal)
Mass		
gram (g)	0.03527	ounce, avoirdupois (oz)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

$$^{\circ}\text{F} = (1.8 \times ^{\circ}\text{C}) + 32$$

Vertical coordinate information is referenced to North American Vertical Datum of 1988 (NAVD 88).

Horizontal coordinate information is referenced to North American Datum of 1983 (NAD 83).

Altitude, as used in this report, refers to distance above the vertical datum.

Sea level: In this report “sea level” refers to the National Geodetic Vertical Datum of 1929 (NGVD of 1929)—a geodetic datum derived from a general adjustment of the first-order level nets of both the United States and Canada, formerly called Sea Level Datum of 1929.

Selected Trace Metals and Organic Compounds and Bioavailability of Selected Organic Compounds in Soils, Hackberry Flat, Tillman County, Oklahoma, 1994-95

By Mark F. Becker

Abstract

In 1995 the Oklahoma Department of Wildlife Conservation acquired a drained wetland in southwest Oklahoma known as Hackberry Flat. Following restoration by Wildlife Conservation the wetland will be used by migratory birds and waterfowl. If naturally occurring trace metals and residual organic compounds from agriculture and industry were present, they may have posed a potential biohazard and were a concern for Wildlife Conservation. The U. S. Geological Survey, in cooperation with Wildlife Conservation and the Oklahoma Geological Survey, examined the soils of Hackberry Flat to determine trace metal concentrations, presence of selected organic compounds, and the bioavailability of selected organic compounds in the soils.

The purpose of this report is to present the data that establish the baseline concentrations of selected trace metals and organic compounds in the soils of Hackberry Flat prior to wetland restoration. Sampling and analysis were performed using two approaches. One was to collect soil samples and analyze the composition with standard laboratory practices. The second exposed composite soils samples to organic-free water and a semipermeable membrane device that mimics an organism and then analyzed the device. Ten soil samples were collected in 1994 to be analyzed for trace metals, organochlorine pesticides, and polychlorinated biphenyls. Soil samples tested for bioavailability of selected organic compounds were collected in 1995. Most of the 182 soil samples collected were from the center of every 40-acre quarter-quarter section owned by the Wildlife Conservation. The samples were grouped by geographical area with a maximum of 16 sample sites per group. Concentrations of most selected trace metals measured from soils in Hackberry Flat are within the range of mean concentrations measured in cultivated soils within the United States. Organochlorine pesticides, polychlorinated biphenyls, and polyaromatic hydrocarbons were not found at concentrations above the analytical detection levels and, if present, in the soil samples are at concentrations below the detection level of the analytical method used. Organochlorine pesticides, total polychlorinated biphenyls, and polyaromatic hydrocarbons were not detected in any of the semipermeable membrane devices at the analytical detection levels.

Introduction

In 1995 the Oklahoma Department of Wildlife Conservation (referred to in this report as Wildlife Conservation) acquired Hackberry Flat, a drained wetland in southwest Oklahoma. Hackberry Flat had been cultivated since the early 1900's until acquisition by Wildlife Conservation. Following restoration by Wildlife Conservation the wetland will be used by migratory birds and waterfowl.

The potential toxicity of naturally occurring trace metals and residual organic contaminants from agricultural and industrial activities, if present, was a concern for Wildlife Conservation. Prior to restoration a baseline of environmental concentrations of metals and chemicals that may endanger wildlife was needed.

The U. S. Geological Survey, in cooperation with Wildlife Conservation and the Oklahoma Geological Survey, sampled and analyzed the soils of Hackberry Flat to determine trace metal concentrations, presence and bioavailability of selected organic compounds in the soils. The data were collected during 1994-95 and analyses were completed in 1996.

The purpose of this report is to present data that documents baseline environmental conditions for trace metals and selected organic chemicals in the soils of Hackberry Flat. Soils were sampled and analyzed using typical laboratory methods and using a surrogate for fauna to determine bioavailability. Ten soil samples were collected in 1994 and analyzed for total organochlorine compounds and total trace metals. To determine organic bioavailability, 182 soil samples were collected and combined to form 12 groups ranging from 8 to 16 soil samples per group. The soil samples were saturated with organic-free water and exposed to semipermeable membrane devices (SPMD). SPMDs are in-place sampling devices that mimic chemical uptake by organisms.

The author thanks Dr. Dan Martin at the U. S. Fish and Wildlife Service for his technical assistance and James Huckins and Dr. Jim Petty from the Biological Resource Division of the U. S. Geological Survey for technical assistance with the SPMDs.

Description of Study Area

Hackberry Flat is a drained wetland located in southwest Oklahoma about 8 kilometers south-southeast of Frederick, in Tillman County (fig. 1 and 2). The wetland is a 2,700-hectare depression that may have formed by the dissolution of highly-soluble evaporite beds within the underlying Permian geologic units. Elevation changes across the wetland are less than 1 meter and indications of the depression are not evident unless the wetland is observed from the boundaries, which gently slope upward from the wetland. Changes in soil color and texture delineate the wetland. The Roscoe Clay-type soil present in Hackberry Flat (Lamar and Rhodes, 1974) is characteristic of a wetland hydric soil that has a gray color and consists largely of montmorillonite. The adjacent soils have a reddish color from the Permian geologic units from which they are derived.

The modern history of Hackberry Flat goes back to the turn of the 20th century when eyewitness accounts described a perennial lake that contained large catfish and attracted abundant waterfowl. Efforts to drain the wetland for cultivation began in 1903 and were completed in 1909 (Wagner, 1995) when a ditch was excavated through a topographic divide on the east that allows discharge of the wetland into an adjacent watershed. The farming practices within Hackberry Flat were dry-land cultivation and the principal crops were wheat, grain sorghum, and cotton.

Wildlife Conservation began acquiring the former wetlands in 1993 and restoration began in 1995. Wildlife Conservation plans to manage Hackberry Flat as a moist-soil wetland with the depth of water not to exceed 0.3 meter. Water in the wetland will come directly from precipitation upon the wetland and surface runoff. Wildlife Conservation plans to flood the wetland and control the discharge from the wetland from fall through winter (Wagner, 1995). To prevent avian botulism Wildlife Conservation plans to drain the wetland in the spring, allow it to dry, and till soils to a shallow depth.

Frederick, a city with a population of about 5,100 (oral commun., City of Frederick), is the largest city near Hackberry Flat in rural southwest Oklahoma. Agriculture is the primary industry and the principal crops are cotton and wheat. There is oil exploration and production around and in the wetland. Some light manufacturing businesses are located at the Frederick airport about four kilometers northwest of the wetland. The airport was used for flight training during World War II.

Climate of Hackberry Flat is typical of the southern Great Plains of the United States and is semiarid. Average annual precipitation is about 66 centimeters per year. The mean daily air temperature for January is 2.2° C and over 28.6° C for July. Estimates for average annual lake evaporation and evapotranspiration are 178 centimeters and 65 centimeters, respectively (Pettyjohn and others, 1983).

Method of Data Collection and Analysis

Soil Sampling and Analysis

Sampling and analysis were performed using two approaches. One was to collect soil samples and analyze the composition with standard laboratory practices. The second exposed composite soil samples to organic-free water and a semipermeable membrane device that mimics an organism and then analyzed the device.

Ten soil samples were collected in 1994 and analyzed for trace metals, organochlorine pesticides, and polychlorinated biphenyls. Due to the homogeneous soils found in the wetland a minimum of one sample was taken from the center of each of the five sections within the wetland. These samples are designated H1 through H3 and H5 and H6. Samples H4 and H8 are sediments from the ditch draining the wetland. Sample H7 also was from the ditch at the eastern boundary of the wetland and was chosen due to the red colored soil that is different from the gray color sediment found in the wetland. Sample H9 was from a small shallow drainage ditch that flowed south to the main ditch. Sample H10 was from an area that was barren of vegetation and salt encrusted. Each site was located using a global positioning system that would permit repeated sampling within one meter of the original site. Sample locations are shown in figure 3.

Soil samples collected for trace metal analysis and pesticide analysis were obtained by excavating a trench about 0.5 meter deep with a shovel. The sides of the trench were then pried off with a metal probe, to avoid any contamination by the shovel and to expose a fresh soil profile. The soil samples were pried off the newly exposed trench wall with a small stainless steel probe, placed in doubled Teflon¹ bags and sealed. The bags containing soil samples for pesticide analysis were kept chilled until shipped for analysis.

The majority of the analytical work was done by the Oklahoma Geological Survey laboratory and selective analysis of selenium and arsenic was done by the U.S. Geological Survey laboratory. Total digestion and analysis methods, as described by U.S. Geological Survey (Arbogast, 1990), were followed by both labs. Analyses of soils for organochlorine pesticides and polychlorinated biphenyls by gas chromatograph/mass spectroscopy were conducted by Quanterra Environmental Services¹, Arvada, Colorado.

Soil samples tested for bioavailability of organic compounds were collected in late August 1995. Most of the 182 soil samples collected were from the center of every 40-acre quarter-quarter section owned by the Wildlife Conservation. The samples were combined into 12 groups by geographical area (fig. 3) with a maximum of 16 sample sites per group. Nine groups are soils from cultivated land or pasture. One group of soils consisted of sediment from the ditch excavated through

¹The use of firm, trade, and brand names in this report is for identification purposes only and does not constitute endorsement by the U. S. Geological Survey.

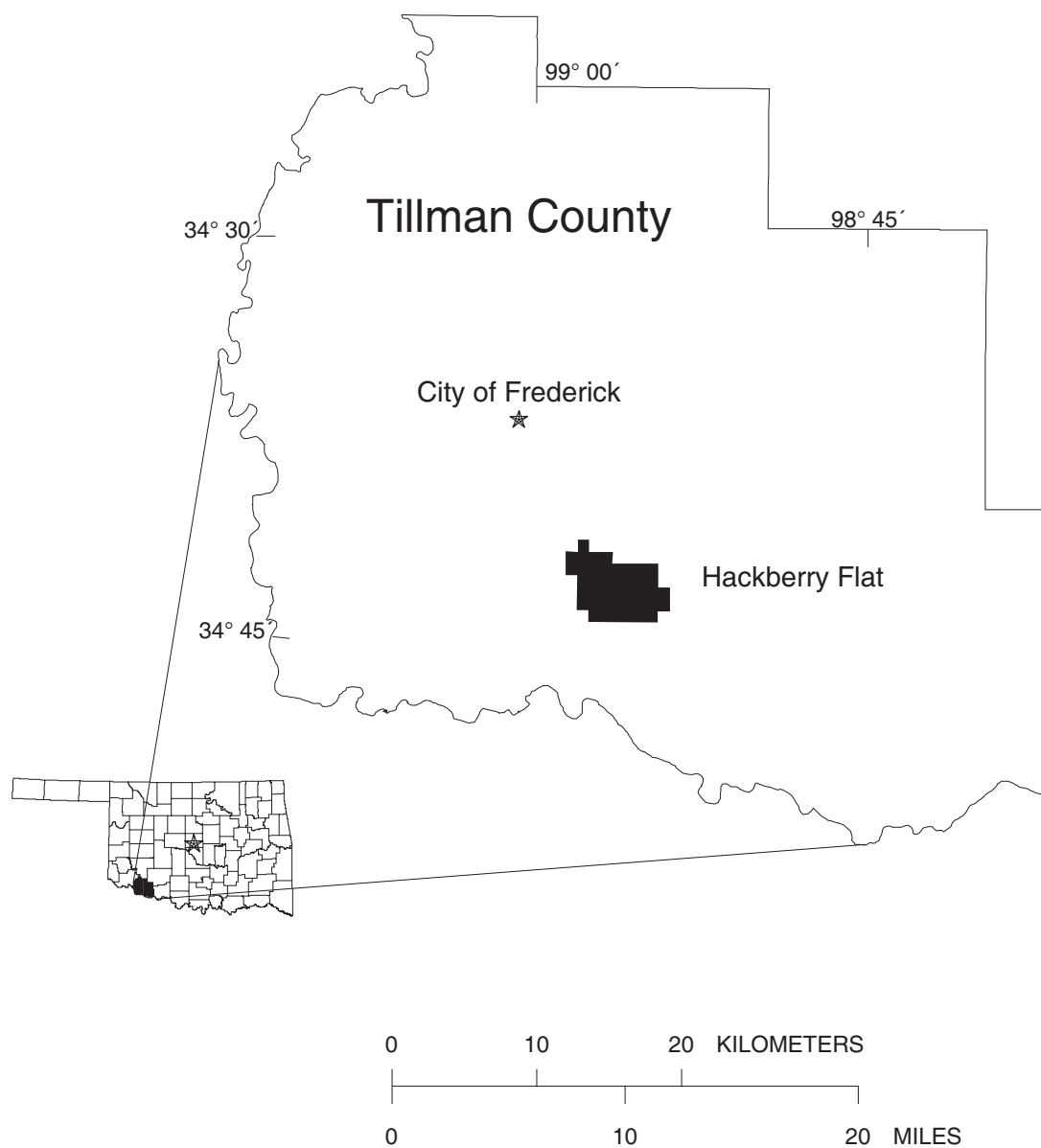


Figure 1. Location of Hackberry Flat near Frederick in Tillman County, Oklahoma.

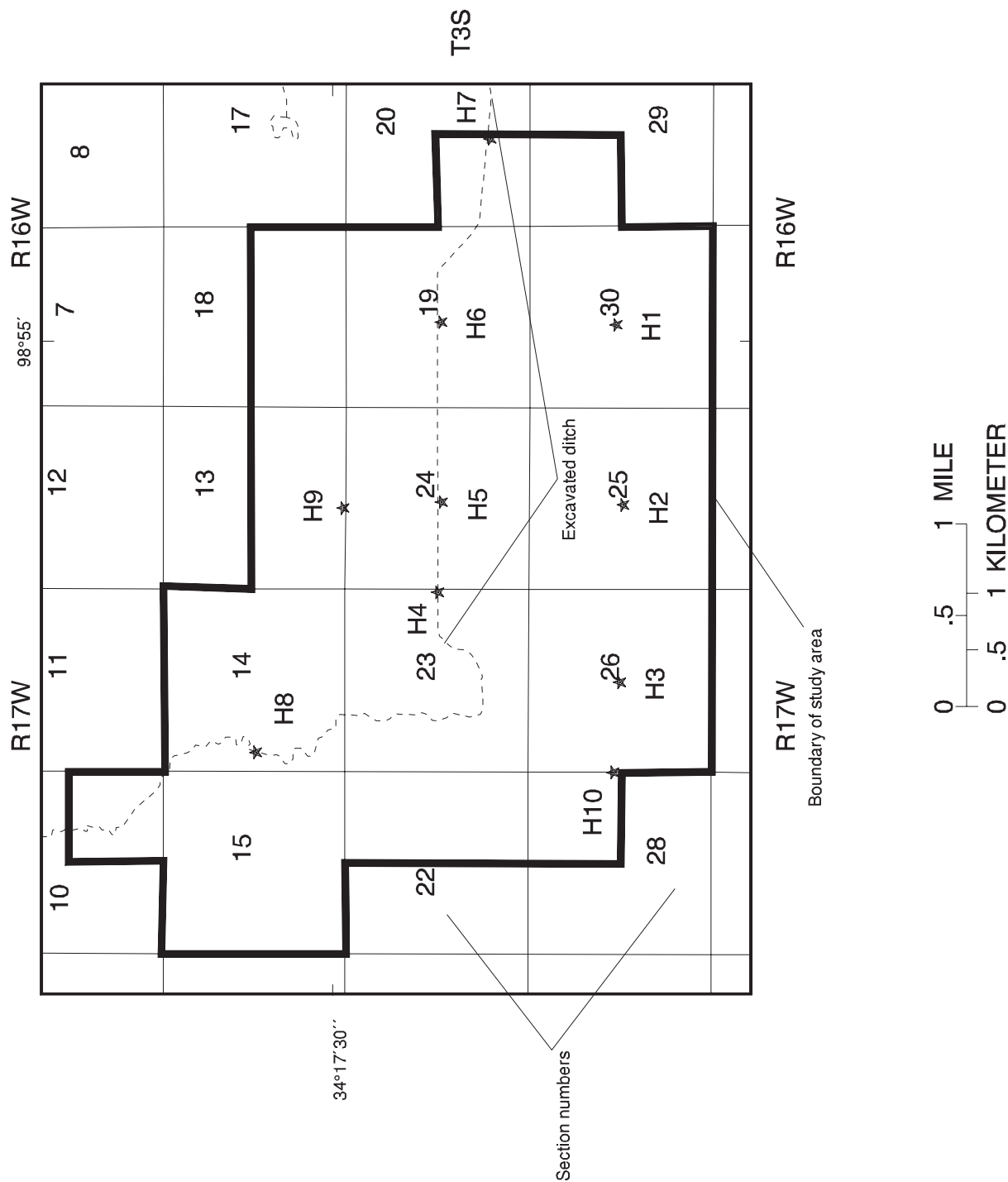


Figure 2. The study area and the location of sites where soils were collected and analyzed for trace metals and organic compounds.

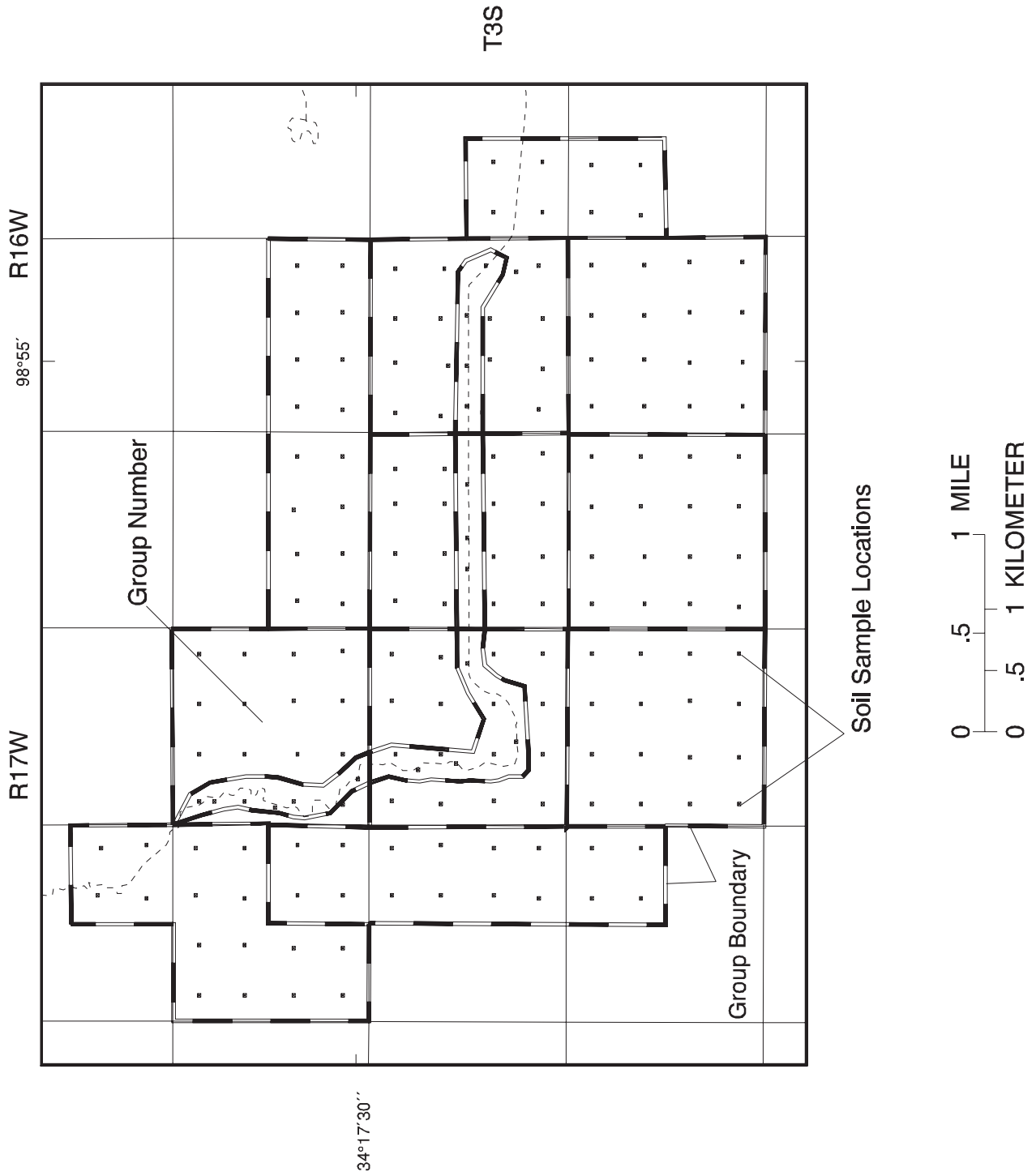


Figure 3. Boundaries of areas where soil samples were combined to form groups for assessment of bioavailable organochlorine pesticides, polychlorinated biphenyls, and polyaromatic hydrocarbons.

Hackberry Flat. The grid sampling system allowed coverage of the entire study area and allowed groups to represent the land-use distribution within the area. Most areas had been cultivated but some contained or were primarily pasture. Each site was located using a global positioning system that would permit repeated sampling within one meter of the original site.

Bioavailability sampling and analysis using semipermeable membrane devices

A portion of many organic compounds is bioavailable in natural conditions permitting uptake by organisms. These residues concentrate with time or they are metabolized in tissues and depurated. The primary interest for this study was the bioavailability of organochlorine pesticides, polychlorinated biphenyls, and polyaromatic hydrocarbons. These compounds were targeted for bioavailability testing because of their resistance to environmental degradation and the potential for these compounds to absorb into the tissues of organisms. To test bioavailability, a microcosm was created to simulate the environment that would exist once the wetland was restored and operational. The SPMD was used to mimic chemical uptake by organisms. The device sequesters lipid-soluble organic compounds that would desorb from the water-saturated soils and partition across semipermeable membranes and into the fats of exposed organisms.

The SPMD was developed at the National Fisheries Contaminant Research Center, U.S. Fish and Wildlife Service, Columbia, Missouri (Huckins and others, 1990). SPMDs contain a thin layer of triolein, a lipid found in most natural fats, enclosed in a membrane composed of layflat polyethylene tubing. The flat tubing design allows optimum exposure of the membrane to the medium sampled and can be configured into several different patterns. Only dissolved nonionic organic compounds can diffuse through the polymeric membrane because cavities in "nonporous" polymers such as polyethylene are less than 10 angstroms in cross-sectional diameter, about the size of a single organic molecule. Compounds with an octanol/water partition coefficient (K_{ow}) greater than or equal to 1 are technically lipophilic but in practice chemicals with K_{ow} values greater than 10,000 are referred to as lipophilic and tend to readily absorb into fats.

There are several advantages to using SPMDs as surrogates for fauna. A scarcity of resident organisms made it necessary to use an alternative method to sample for the bioavailable compounds of interest. The combination of SPMDs and microcosms allowed flexibility in terms of sampling density and the dimensions of the area sampled. In particular these methods allow for broad initial scans followed by a more focused approach if specific compounds are detected. Grouping soils by related subareas permitted the use of one SPMD for screening, compared to the traditional method of analyzing several soil samples over the same area. If a targeted compound is detected this method can be used to pinpoint the source by focusing on the area of interest and increasing the sampling density.

Another advantage of SPMDs is the ability to replicate the method with a changing ecosystem. The Hackberry Flat ecosystem has been fairly constant for over 85 years but will change drastically once the wetland is restored. This change will result in faunal successions that would make comparisons of environmental data of organisms difficult. The SPMD is a reproducible method to measure bioavailable compounds through time as well as under changing conditions and would allow for data that can be compared with time series stochastic analysis. Experimental control is another desirable feature of the SPMD. SPMDs enable the investigator to alter the experimental design and perform in-place sampling for the microcosm test used for this study.

The microcosms were assembled in a controlled office environment. Soil groups were brought to the U.S. Geological Survey office in Oklahoma City, and emptied into carbon-free individual 3.78 liter jars and saturated with 1.0 liter organic-free water. Soil group weights ranged from 703 to 1,920 grams. This variation was due to the number of samples per group and amount recovered at each site. An SPMD was placed in the soil-water solution and the jar was sealed. The jar was vigorously agitated every 2-4 days; after 45 days the SPMDs were removed and shipped to the appropriate laboratory for cleanup, processing, and analysis.

A quality-control program was designed to detect false positive responses caused by the efficient sorption capabilities of the SPMD. Quality control SPMD samples consisted of one field blank, two ambient water and lab blanks, and one duplicate. The ambient field blank was exposed in the field over a two week period during the soil sampling and kept in a Teflon¹ bag during transit. Two quality-control SPMDs used as ambient water blanks were placed in jars with 1.0 liter of organic-free water at the lab. Finally, a control SPMD was used with a duplicate set of soil samples from group 9. The capacity of the SPMD to absorb compounds from the environment is well documented by Huckins and others, 1993, Lebo and others, 1992, Petty and others, 1994, and Prest and others, 1992. Therefore, concerns for false negatives were not an issue.

Field procedures were designed to avoid possible sample contamination. A chrome-plated soil probe with a 17.46 millimeter inner diameter was used to obtain the bioavailability soil samples. The samples were collected to a depth of 15-20 centimeters. Samples were collected by group, combined in the field as they were collected, and stored in sealed Teflon¹ bags. Soil probes were decontaminated following collection of all the samples comprising a group. Decontamination was done by thoroughly washing the probes with soap and municipal-supply water, rinsing with organic-free water and air drying, rinsing with methanol and sealing in a Teflon¹ bag. Probes were covered with a Teflon¹ wrap and an outer cover of aluminum foil to prevent contamination while in transport.

SPMDs are available from a private vendor and require several steps of processing before analysis. SPMDs were acquired from Custom Industrial Analysis Labs¹, St. Joseph, Missouri. The SPMDs arrived in sealed metal containers filled with argon gas to protect against airborne contamination. After

exposure to the soil and water solution the SPMDs were removed from the jars, placed in the original containers, sealed and shipped overnight to Custom Industrial Analysis Labs¹. There SPMDs were cleaned and underwent a hexane dialysis recovery process. This process results in the transfer of compounds absorbed into the triolein back out into the hexane, which is followed by a further cleanup process of the dialysate prior to analysis. The purified dialysate was sent to Hazelton Environmental Services¹, Madison, Wisconsin, to be analyzed for organochlorine pesticides, total polychlorinated biphenyls, and polyaromatic hydrocarbons.

Selected Trace Metals and Organic Compounds in Soils

Most selected trace metal concentrations measured in soils in Hackberry Flat are within the range of mean concentrations measured in cultivated soils within the United States. The metals analyzed and results are listed in table 1. Included in table 1 are ranges of mean total concentrations for cultivated soils reported by Connor and Shacklette, (1975). This provides a qualitative comparison of the chemical concentrations found in Hackberry Flat soils with soils in the United States. These total concentrations of selected trace metals include metals that are bound to the organic material or associated with minerals in the soil. It would be speculative to hypothesize about the degree of bioavailability of these metals in the future wetland without knowing its geochemical conditions.

One sample, H-7, has concentrations of iron, manganese, and arsenic that exceed the comparative range of means. This sample was taken from the sediment within the ditch beyond the extent of the wetland soils and the results reflect soils and sediment containing bedrock rather than wetland soils. The Permian bedrock, that is the source of this soil, contains oxides of iron and manganese, which can contain arsenic within these minerals.

None of the organochlorine pesticides, polychlorinated biphenyls, and polyaromatic hydrocarbons (table 2) concentrations were above the analytical detection levels. If these compounds are present in the soil samples, they are at concentrations below the detection level of the analytical method used.

Bioavailability of Selected Organic Chemicals

Organochlorine pesticides, total polychlorinated biphenyls, and polyaromatic hydrocarbons were not detected in any of the SPMDs at the analytical detection levels that included the quality-control samples. Specific compounds analyzed and the analytical detection levels are listed in table 3.

Nondetection of the selected organic compounds in the SPMD could be the result of many factors. Limited use of pesticides within the area may be one factor. Another factor that might contribute to the nondetection could be the ability of the

soil to strongly partition these compounds and retard their ability to enter solution. It was beyond the scope of this project, but analysis of the microcosm sediment for particle size and organic carbon may have further validated the results.

Summary

Hackberry Flat is a drained wetland located in southwest Oklahoma about 8 kilometers south-southeast of Frederick, Tillman County (figs. 1 and 2). The wetland is a 2,700-hectare depression that probably was formed from the dissolution of highly-soluble evaporite beds within the Permian geologic units that underlie the wetland. The wetland can be delineated by the change in soil color and texture. The recorded history of Hackberry Flat goes back to the turn of the 20th century when eyewitness accounts describe a perennial lake that contained large catfish and attracted abundant waterfowl. Efforts to drain the wetland for cultivation began in 1903 and were completed in 1909 when a ditch was excavated through a topographic divide on the east that allows discharge of the wetland into an adjacent watershed. The farming practices within Hackberry Flat were dry-land cultivation and the principal crops were wheat, grain sorghum, and cotton. Wildlife Conservation plans to manage Hackberry Flat as a moist-soil wetland with the depth of water not to exceed 0.3 meter. Water in the wetland will come directly from precipitation upon the wetland and surface runoff.

Sampling and analysis were performed using two approaches. One was to collect soil samples and analyze the composition with standard laboratory practices. The second exposed composite soils samples to organic-free water and a semipermeable membrane device that mimics an organism and then analyzed the device. Ten soil samples were collected in 1994 to be analyzed for trace metal, organochlorine pesticide, and polychlorinated biphenyls. Due to the homogeneous soils found in the wetland at least one sample was taken from the center of each of the five sections within the wetland. Soil samples collected for trace metal analysis and pesticide analyses were obtained by excavating a trench about 0.5 meter deep with a shovel. Soil samples tested for bioavailability of organic compounds were collected in 1995. Most of the 182 soil samples collected were from the center of every 40-acre quarter-quarter section owned by Wildlife Conservation. The samples were grouped by geographical area with a maximum of 16 sample sites per group. Soil bioavailability samples were obtained using a 17.46 millimeter inner diameter chrome-plated soil probe. The samples were collected to a depth of 15-20 centimeters. A scarcity of resident organisms made it necessary to use an alternative method to sample for the bioavailable compounds of interest. Semipermeable membrane devices, SPMDs, used as an alternative organism in a wetlands microcosm, allowed control of sampling density and the ability to control the dimensions of the area sampled. An SPMD was placed in the soil-water solution and the jar was sealed. The jar was vigorously agitated every 2-4 days, after 45 days the SPMDs were

Table 1. Total concentrations of selected trace metals of soils from Hackberry Flat, Tillman County, Oklahoma, and median concentrations for soils in the conterminous United States

[mg/kg, milligrams per kilogram; <, indicates the concentration is less than the analytical detection level shown; percent, indicates the percent of the total volume analyzed]

Sample site	Location	Hg mg/kg	Cd mg/kg	Cr mg/kg	Cu mg/kg	Fe mg/kg	Mn mg/kg	Pb mg/kg	V mg/kg	Se ¹ mg/kg	As ¹ mg/kg
H-1	16W-03S-30	<0.04	<4.0	60	40	2.84	460	<20	80	0.1	8.2
H-2	17W-03S-25	<0.04	<4.0	60	30	2.70	430	<20	70	0.1	8.0
H-3	17W-03S-26	<0.04	<4.0	50	30	2.44	440	<20	70	0.1	7.5
H-4	17W-03S-23	<0.04	<4.0	50	20	2.37	390	<20	60	0.1	6.2
H-5	17W-03S-24	<0.04	<4.0	60	20	2.95	520	<20	80	0.2	8.5
H-6	16W-03S-19	<0.04	<4.0	60	20	2.91	530	<20	80	0.1	9.5
H-7	16W-03S-20	<0.04	<4.0	60	30	3.13	1,370	<20	80	0.6	14.0
H-8	17W-03S-14	<0.04	<4.0	60	30	2.90	410	<20	80	0.3	11.0
H-9	17W-03S-13	<0.04	<4.0	50	30	2.42	570	<20	60	0.4	7.9
H-10	17W-03S-26	<0.04	<4.0	40	20	1.99	420	<20	50	0.2	7.5
		² 0.030-0.096	² <1	² 31-70	² 9.9-39.0	² 1.4-2.8	² 99-740	² 2.6-31.0	² 20-93	² 28-.62	² 5.4-12.0
		³ 0.15/1.3	³ 5.0/9.0	³ 80/145	³ 70/390	⁴ 2.0/4.0	⁴ 460/1100	³ 35/110	ND	ND	³ 33/85

All results are average values for duplicates.

¹Analyzed by USGS, all others analyzed by the Oklahoma Geological Survey laboratory.

²Values represent the range of average total concentrations measured for cultivated soils in the conterminous United States (Connor and Shacklette, 1975).

Table 2. Results of analyses of soil from sites H-1 to H-10 for organochlorine pesticides and polychlorinated biphenyls analyses from soils in Hackberry Flat, Tillman County, Oklahoma

[mg/kg, milligrams per kilogram; <, indicates the concentration is less than the analytical detection limit shown]

Constituent	Detection level Reported in mg/kg
Aldrin	<0.01
Alpha-BHC	<0.01
beta-BHC	<0.01
delta-BHC	<0.01
gamma-BHC (Lindane)	<0.01
Chlordane	<0.1
4,4,'-DDD	<0.02
4,4,'-DDE	<0.02
4,4,'-DDT	<0.02
Dieldrin	<0.02
Endrin	<0.02
Endrin aldehyde	<0.02
Endosulfan I	<0.02
Endosulfan II	<0.02
Endosulfan sulfate	<0.02
Heptachlor	<0.02
Heptachlor epoxide	<0.02
Toxaphene	<0.02
Aroclor 1016	<0.1
Aroclor 1221	<0.1
Aroclor 1232	<0.1
Aroclor 1242	<0.1
Aroclor 1248	<0.1
Aroclor 1254	<0.1
Aroclor 1268	<0.1
Methoxychlor	<0.1

Table 3. Results of organochlorine pesticide, total polychlorinated biphenyl, and polyaromatic hydrocarbon analyses of dialysates from semipermeable membrane devices for groups 1-12

[µg/ml, micrograms per milliliter; <, indicates the concentration is less than the analytical detection level shown]

Parameter	Detection Level (µg/ml)
Hexachlorobenzene	<0.02
Alpha-BHC	<0.02
Beta-BHC	<0.02
Gamma-BHC (Lindane)	<0.02
Oxychlordane	<0.02
Gamma-Chlordane	<0.02
Alpha-Chlordane	<0.02
4,4'-DDD	<0.02
4,4'-DDE	<0.02
4,4'-DDT	<0.02
Endrin	<0.02
o,p'-DDD	<0.02
o,p'-DDE	<0.02
o,p'-DDT	<0.02
Heptachlor epoxide	<0.02
Dieldrin	<0.02
Mirex	<0.02
Trans-Nonachlor	<0.02
Toxaphene	<0.2
Total PCB's	<0.2
Naphthalene	<0.54
Acenaphthylene	<1.1
1-Methyl naphthalene	<0.54
2-Methyl naphthalene	<0.54
Acenaphthene	<0.54
Fluoranthene	<0.11
Phenanthrene	<0.05
Anthracene	<0.05
Fluoranthene	<0.11
Pyrene	<0.05
Benzo (a) anthracene	<0.05
Chrysene	<0.05
Benzo (b) fluoranthrene	<0.11
Benzo (k) fluoranthene	<0.05
Benzo (a) pyrene	<0.05
Dibenzo (a,h) anthracene	<0.11
Benzo (ghi) perylene	<0.11
Indeno (1,2,3-cd) pyrene	<0.05

removed and shipped for cleanup, processing, and analysis. A quality-control program was used to detect false positive responses caused by the efficient sorption capabilities of the SPMD.

Most concentrations of selected trace metals measured in soils in Hackberry Flat are within the range of mean concentrations measured in cultivated soils within the United States. This provides a qualitative comparison of the concentrations found in Hackberry Flat soils that shows they are similar to the means of total concentrations of cultivated soils found through the United States. There were no detectable organochlorine pesticides and polychlorinated biphenyls in the soils when analyzed by gas chromatograph/mass spectroscopy. There were no detectable organochlorine pesticides, total polychlorinated biphenyls, and polyaromatic hydrocarbons accumulated in the SPMD, exposed to the soil groups or in the quality-control samples.

Selected References

- Arbogast, Belinda F., ed., 1990, Quality assurance manual for the Branch of Geochemistry, U.S. Geological Survey: U.S. Geological Survey Open-File Report 90-668, p. 184.
- Connor, J. J. and Shacklette, H. T., 1975, Background geochemistry of some soils, plants, and vegetables in the conterminous United States: U. S. Geological Survey Professional Paper 574-F, 164 p.
- Huckins, J. N., Tubergen, M. W., Manuweera, G. K., 1990, Semipermeable membrane devices containing model lipid: A new approach to monitoring the bioavailability of lipophilic contaminants and estimating their bioconcentration potential: Great Britain, *Chemosphere*, V. 20 p. 533-552.
- Huckins, J. N., Manuweera, G. K., Petty, J. D., MacKay, D., Lebo, J. A., 1993, Lipid-containing semipermeable membrane devices for monitoring organic contaminants in water: *Environmental Science and Technology*, V. 27, p. 2489-2496.
- Lamar, Otho W. and Rhodes, Clifford E., 1974, Soil survey of Tillman County, Oklahoma: United States Department of Agriculture, Soil Conservation Service, p. 21.
- Lebo, J. A., Zajicek, J. L., Huckins, J. N., Petty, J. D., and Peterman, P. H., 1992, Use of semipermeable membrane devices for *in situ* monitoring of polycyclic aromatic hydrocarbons in aquatic environments: Great Britain, *Chemosphere*, V. 25, No. 5, p. 697-718.
- Petty, J. D., Huckins, J. N., Orazio, C. E., Lebo, J. A., Clark, R. C., and Gibson, V.L., 1994, Laboratory studies of the use of semipermeable membrane devices (SPMDs) as passive water samplers of polyaromatic hydrocarbons (PAH) priority pollutants: Midwest Science Center, Columbia, MO, National Biological Survey Technical Report.
- Petty, J. D., Huckins, J. N., Martin, D. B., and Adornato, T. G., 1995, Use of semipermeable membrane devices (SPMDs) to determine bioavailable organochlorine pesticide residues in streams receiving irrigation drain water: Great Britain, *Chemosphere*, V. 30, p. 1891-1903.
- Pettyjohn, Wayne A., White, Hal, and Dunn, Shari, 1983, Water atlas of Oklahoma: Stillwater, Oklahoma, University Center for Water Research, Oklahoma State University, Stillwater, p. 71.
- Prest, H.F., Jarman, W.M., Burns, S.A., Weismuller, M. Martin, and Huckins, J.N., 1992, Passive water sampling via semipermeable membrane devices (SPMDs) in concert with bivalves in the Sacramento/San Joaquin River Delta: Great Britain, *Chemosphere*, V. 25, no. 12, p. 1811-1823.
- Wagner, Steve, 1995, From wheatlands to wetlands: Oklahoma City, Oklahoma, Oklahoma Department of Wildlife Conservation, *in* Outdoor Oklahoma, March/April.