South Dakota Water Research Institute Annual Technical Report FY 2004

Introduction

South Dakota's Water Resources Research Institutes program is administered through the College of Agricultural and Biological Sciences at South Dakota State University (SDSU). Dr. Van Kelley has been the Director since August 1, 2000. Dr. Kelley is also the head of the Agricultural and Biological Sciences Engineering Department. The annual base grant from the United States Geological Survey (USGS) and a legislative appropriation of \$92,412 form the core of the SDWRRI budget. The core budget is supplemented by research grants from a variety of funding agencies as well as private organizations interested in specific water issues.

The mission of the South Dakota Water Resources Institute is to address the current and future water needs of people, agriculture, and industry through research, education, and service. This report is a summary of activities conducted during Fiscal Year 2004 to accomplish this important mission.

Research Program

During FY 2004, the South Dakota Water Resources Institute (SD WRI) used its 104B Grant Program funds to conduct research of local, state, regional, and national importance addressing a variety of water problems in the state. The Advisory Committee reviews grant applications and recommends projects for funding that address research priorities, have a good chance of success, and that increase our scientific knowledge. Principal investigators and graduate students studied issues like runoff and nutrient transport; hydrologic interactions among runoff, the vadose zone, shallow groundwater and its influence on crop growth, groundwater quality characterization, and aquatic habitats and communities within them.

The subject of nutrient transport caused by runoff from manured fields has been an active research area since a literature review conducted in 2000 generated statewide interest in transport of phosphorus from soils high in phosphorus. Information is needed to support regulations to protect the water quality of lakes and streams without placing an undue burden on livestock producers. Livestock production plays a major role in agriculture, the states number one industry. Current research is exploring the upper limits of phosphorus concentration in soils before phosphorus in runoff causes major surface water quality degradation. One soil series was evaluated in 2002, three additional soils were completed in 2003, and two more soil series were evaluated in 2004.

Non-point source pollution from agricultural land is an important issue in South Dakota. Runoff models could be used for estimating impacts of non-point source pollution in eastern South Dakota but they require validation with measured field data to be credible. A research project was started in 2004 to measure runoff rate, drain line flow, and volume that calibrated a runoff model that was previously written. Two sites were established in small agricultural watersheds and were used for this study. Additional data collected included precipitation at the sites, drain line flow rate, and water table elevations. Evapotranspiration was estimated with measured weather data. One MS-level graduate student

was funded through this project.

In 2004 a groundwater research project funded with the 104B program focused on dissolved organic carbon (DOC) concentrations in the Big Sioux Aquifer. It has been discovered that wetlands, lakes, and streams that are hydrologically connected to the Big Sioux Aquifer have DOC concentrations as much as 30 times greater than the DOC concentrations of the aquifer itself. The low concentration of DOC in the aquifer may indicate that DOC can be used as a sensitive indicator of groundwater quality. However, the organic geochemistry of the Big Sioux hydrologic system must be understood before DOC can confidently be used as an indicator.

In 2004 a related study was conducted to better understand the coupled carbon-nitrogen cycle in a prairie pothole wetland. The study site is unique because it rapidly becomes anoxic after the water surface freezes in the winter, and rapidly becomes oxic in the spring, and stays oxic, because it is well-mixed by the prevailing winds. It thus represents a convenient closed chemical reactor to study these reactions. This is the first year of what will be a three-year project. The overall project has the following objectives: 1. Determine the annual variation in the redox potential of the pothole. 2. Create a nitrogen and carbon mass balance for the pothole. 3. Determine the form of nitrogen present in the dissolved organic matter. 4. Using 15N and 13C labeled compounds, trace the coupled C-N transformations in the pothole under oxic and anoxic conditions.

South Dakota relies heavily on ground water resources to fulfill its domestic needs. Petroleum compounds such as benzene, toluene, ethylbenzene, and xylene (BTEX) have been implicated as significant sources of ground water contamination in South Dakota. Soil organic matter (SOM) has the ability to remove (sorb) potentially hazardous organic chemicals that have been introduced, either deliberately or inadvertently, into the environment. Glomalin, a glycoprotein in SOM, is noted for its ameliorative effects on soil structure; however, its molecular character and sorptive capacity for organic contaminants are unknown. Through a series of sorption experiments, and solid-state nuclear magnetic resonance and various chemical analyses, the molecular and sorptive character of Glomalin was studied. The objectives of this study are to 1) evaluate the role of glomalin and its fractions in the sorption of hydrophobic organic contaminants, and 2) elucidate a molecular understanding of glomalin through its extraction, fractionation, and characterization. Glomalin may prove to be a very useful index in predicting the fate of organic contaminates introduced into the environment.

Finally, a lake habitat research project was continued in 2003. The potential invasion of exotic species such as zebra mussel and Eurasian water milfoil has spurred study of two lake habitats to document the natural populations of macroinvertebrates and aquatic plants and monitor their water quality. Monitoring of these habitats continued in FY04 with emphasis on collecting and identifying the many adult chronomids that emerge from both lakes each summer.

Hydraulic Calibration of the Upper Soil Layers in a Glacial Till System

Basic Information

Title:	Hydraulic Calibration of the Upper Soil Layers in a Glacial Till System
Project Number:	2004SD18B
Start Date:	3/1/2004
End Date:	2/28/2006
Funding Source:	104B
Congressional District:	First
Research Category:	Not Applicable
Focus Category:	Agriculture, Hydrology, Non Point Pollution
Descriptors:	
Principal Investigators:	Todd P. Trooien, Hal D. Werner

Publication

Annual Progress Report

State Water Resources Institute Program (SWRIP) March 2004 to February 2005

Title: Hydrology model calibration in a glacial till system

Investigators: Todd P Trooien and Hal D Werner, Agricultural and Biosystems Engineering Department, South Dakota State University, Brookings, SD 57007. Phone: 605-688-5141.

Introduction

Runoff models could be used for estimating impacts in eastern South Dakota but they require validation with measured field data to be credible. This project will measure runoff rate and volume and be used to calibrate a runoff model such as HEC. Two sites have been established in small agricultural watersheds and will be used for this study. Additional data to be collected include precipitation at the sites, drain line flow rate, and water table elevations. Evapotranspiration will be estimated with measured weather data. One MS-level graduate student is funded with this project.

Current project

Previous research at these two sites and simulation of the hydrology of the sites have shown that adding subsurface drainage to a cropped waterway can increase the long-term average crop yield by eliminating catastrophic wet events in years of high rainfall (Kathol, 2003). The current project was established to validate the water balance model and to calibrate standard models such as DRINAMOD and HEC for these two sites.

Progress in 2003-4

Piezometers and monitoring wells were installed at both research sites in the fall of 2003. Two monitoring wells were installed at both sites. Fourteen piezometers were installed at each site.

Instrumentation was installed to continuously measure depth to water in monitoring wells and piezometers at both research sites. The ultrasonic sensors and associated data collection and transmission equipment were purchased from AgSense LLC, Huron, SD. The systems (Fig. 1) consist of ultrasonic signal generator/sensor units that mount in the PVC cap at the top of the well, wire to a control module, short-range telemetry to a shortterm data storage module, then long-range telemetry to an internet access point. Power is provided by a small batter at each control module; a solar panel keeps the small battery charged. The data are retrieved remotely via WWW. The tipping bucket rain gauge is connected to the long-range telemetry so rainfall data are also retrieved via the WWW. Checking the rainfall data remotely can alert people on campus to rainfall amounts and intensity and provide an indication of whether or not runoff was generated. If runoff was generated, the runoff sampler will be visited and the samples collected and returned to the analytical lab on campus.

The final data collection procedure is currently being refined. Occasional manual readings of water levels have been collected.

The early spring of 2005 was dry and resulted in no sampled runoff events. Early summer has brought more rainfall and the artificial drains are now flowing. Samples have been collected and laboratory chemical analyses are pending. A dry 2004 resulted in few runoff and drain flow samples.



Figure 1. Ultrasonic sensors atop two piezometer tubes, wire to the control module, and solar panel. The additional wire leads to other sensors (not shown).

The piezometric surfaces and hydraulic gradients to the artificial drain, water table elevations, weather data, runoff rates and volumes, and drain flow rates and volumes will be used to refine simulation models of the system. The models will include the spreadsheet water balance model generated in a previous project (Kathol, 2003), DRAINMOD, and HEC.

The remainder of CY2005 and early 2006 will be used to integrate the field data into the simulation models. Field data collection will continue through 2005 and into 2006. We expect that two MS theses will be generated in 2006. One thesis will be supported by funds from this project. The other will be supported by assistantship funds from the Agricultural Experiment Station and using the data generated in this project.

References

Kathol, J. P. 2003. Simulated corn yield responses in drained and undrained waterways. MS Thesis, South Dakota State University.

Evaluating Glomalin and Its Role in the Sorption of Organic Contaminants

Basic Information

Title:	Evaluating Glomalin and Its Role in the Sorption of Organic Contaminants
Project Number:	2004SD19B
Start Date:	3/1/2004
End Date:	2/28/2006
Funding Source:	104B
Congressional District:	First
Research Category:	None
Focus Category:	Groundwater, Water Quality, Solute Transport
Descriptors:	None
Principal Investigators:	Frank V. Schindler, James A. Rice

Publication

- Mercer, E.J., F.V. Schindler, and J. A. Rice. 2005. Solid-state 13C NMR Evaluation of Glomalin Extracted From Soil, 226th National Mtg., Am. Chem. Soc., Geochem. Div., Mar. 2005, San Diego, CA, abstracts.
- Mercer, E.J., F.V. Schindler, and J. A. Rice. 2004. Elemental and Structural Assessment of Glomalin. Sigma Xi Annual Meeting and Student Research Conference. Montréal, Quebec, Canada. Nov. 11-14, 2004.
- 3. Schindler, F.V., E. J. Mercer, and J. A. Rice. 2005. Evaluating the Elemental and Structural Character of Glomalin Extracted From Soil. In review

Annual Progress Report

State Water Resources Institute Program (SWRIP) March 2004 to February 2005

<u>PART I</u>.

Title: Evaluating Glomalin and Its Role in the Sorption of Organic Contaminants

Investigators: Dr. Frank V. Schindler, Dept. of Chemistry and Biochemistry Dr. James A. Rice, Dept. of Chemistry and Biochemistry

The following report discusses the results and progression of the research project titled "Evaluating Glomalin and Its Role in the Sorption of Organic Contaminants" during the funding period of March 2004 through February 2005. This project was designed and proposed as a three year project, but received only one year of funding. The information gathered to date provides valuable information regarding glomalin's chemical and structural character, which may have profound implications regarding glomalin's role in contaminant sorption and soil nutrient availability. The objective of this study was to analyze the elemental and structural character of glomalin extracted from whole soils through a series of chemical assays and solid-state NMR and infrared spectroscopic techniques.

Methodology:

<u>Soils</u>. The mineral soil samples used in this study were collected in eastern South Dakota, USA and are described by Malo (1994). They are referred to as the Poinsett silt loam (fine-silty, mixed, superactive, frigid Calcic Hapludolls), and the Hetland (fine, smectitic, frigid, Pachic Vertic Argiudolls). The Pahokee peat, an International Humic Substances Society (IHSS) reference material (Cat. No. BS103P), was used in this study and contained 45% C, 15% ash, 4.7% H, and 3.1% N (IHSS, 2005). All mineral soils were collected as random grabs from the top 0-15 cm of soil after the initial plant litter had been removed (Kohl, 1999). Mineral soil samples were air dried, crushed, and sieved to pass a 2mm mesh.

<u>Glomalin extraction and purification</u>. Glomalin was extracted using the total protein extraction procedure described by Wright et al. (1996). Eight mL of a 50mM sodium citrate solution was placed into a centrifuge tube containing one gram of whole soil. Samples were autoclaved for 60 minutes at 121°C followed by centrifugation at 3000 to 5000 xg for fifteen minutes to pellet the soil particles. The supernatant was decanted and stored at 4°C until purified. Repeated extractions were performed to effect complete glomalin extraction as evidenced by a transparent supernatant (Wright et al., 1996).





To assess the effect of purification method on the chemical and structural characteristics of glomalin, extracted glomalin samples were subjected to either the trichloroacetic acid (TCA) (i.e., Het-TCA, Poin-TCA or Peat-TCA) or hydrochloric acid (i.e., Het-HCI, Poin-HCI or Peat-HCI) precipitation procedures (Wright, 2004). The HCI method is recommended if the carbon (C) and nitrogen (N) concentrations are measured, since TCA may adhere to glomalin during the precipitation process and produce inaccurate C results (Wright et al., 1996). Glomalin extracts were added to centrifuge tubes in a 1:1 ratio with ice cold 20% TCA and incubated for 1 hr. Samples were centrifuged, the supernatant decanted, and the solid

material reconstituted with 1 mL of 100 mM sodium borate solution. This was transferred to hydrated dialysis tubing (3500 Daltons). The tubing was placed in 10 mM borate solution (pH =8) and dialyzed at 8 hr intervals under constant stirring. The dialysis solution was changed at least 3 times to ensure proper purification. The purified dialyzate was centrifuged at 10,000 rpm for 15 min to remove any extraneous particles. The supernatant was transferred to freeze drying flasks, immediately frozen in liquid N_2 , and lyophilized. Extracted glomalin samples were also purified and reconstituted similarly except 1N HCl and 0.1 M NaOH were used for precipitation and reconstitution, respectively (Wright, 2004). All reagents used in extraction and purification were purchased from Aldrich Chemical Co. Inc. at 99+% purity.

<u>Protein Assay.</u> The protein content of glomalin extracted from whole mineral and organic soils was quantified using the standard Bradford assay (Bradford, 1976). One hundred mg Coomassie Brilliant Blue G-250 (Sigma-Adrich Co.) was dissolved in 50 mL 95% ethanol and 100 mL of 85% phosphoric acid was added. The resulting solution was diluted to a final volume of 1 L, filtered thorough a Whatman #1 paper, and used as the color reagent for protein quantitation. Standard solutions of reagent grade Bovine Serum Albumin (Equitech-Bio, Inc., Kerriville, TX) were prepared containing 20 to 200 g protein. Color reagent was added to both standard and sample unknowns in a 50:1 (v:v) reagent to sample ratio, vortexed, and allowed to incubate for 10 min before absorbance measure at 595 nm. The protein in unknown samples was determined by fitting a least squares regression curve of the quantity of standard protein vs. absorbance. All standard and sample unknowns possessed the same solution matrix.

<u>Solid-state</u> ¹³C NMR. Glomalin was characterized by quantitative solid-state ¹³C NMR using the technique described by Mao et al. (2000). Glomalin samples were placed in a 4 mm o.d. zirconia rotor equipped with Kel-F endcaps and characterized on a Bruker AVANCE 300 (7.4T) widebore spectrometer. All spectra were acquired at 75 MHz using direct-polarization magic angel spinning (DPMAS) combined with a spin-lattice relaxation correction (T_1^C) and total sideband suppression (CP-TOSS) (Mao et al., 2000). T_1^C values used for DPMAS ranged from 3 to 6 sec. High-power pulse



lengths and power levels were optimized with respect to an external reference consisting of a mixture of L-leucine-1-¹³C, glycine-2-¹³C, and L-alanine-3-¹³C in a 1:1:1 ratio. During spectrum acquisition, rotor spin rate and the number of scans were held constant at 13 kHz and 20 000, respectively. The ¹³C NMR spectra were integrated according to the following chemical shift regions: 0-50 ppm = aliphatic carbon, 50-108 ppm = carbohydrate carbon, 108-160 ppm = aromatic carbon, and 160- 200 ppm = carboxyl carbon, and 200-220 ppm = carbohyl carbon (Mao et al., 2000; Wilson, 1987).

<u>Chemical Analyses.</u> Glomalin and BSA samples were sent to Huffman Laboratories, Inc. in Golden, Co. for C, H, N, O, Fe, Na, P, and ash content determinations. Iron, P, and Na were performed after mixed acid decomposition of the samples ending with complete oxidation of organic material by refluxing with perchloric acid. The diluted digestion solutions were analyzed by inductively coupled atomic emission spectroscopy (ICP-AES) following EPA method 200.7 protocol and using a Perkin-Elmer Optima 3000 analyzer. Carbon and H were determined using a custom built analyzer which uses coulometric detection. Ash content was determined by high temperature combustion of the sample until a constant weight was obtained. Moisture content of samples was determined by Karl Fisher titration. All elemental analyses presented in Table 1 are reported on a water-free basis. All non-ash elements reported on a water and ash-free basis.

Principal Findings and Significance:

Selected characteristics of whole soil, glomalin extracted material, and bovine serum albumin (BSA) protein standard are presented in Table 1. The Bradford assay ranged from 54 to 114 mg protein g⁻¹ of extracted material (Table 1), which corresponds to 3 to 68 mg of Bradford sensitive protein g⁻¹ of soil. This range is within that reported for mineral and organic soils (Lovelock et al., 2004; Wright, 2002). On average, glomalin accounted for 25% and 52% of the total C in the mineral soils (Hetland and Poinsett) and organic soil, respectively. This is consistent with the literature for a typical mineral soil (Wright, 2002). Little difference in the elemental composition of glomalin extracted from the Hetland and Poinsett soils was observed, however, marked increases in C and N and decreases in ash percentages were evident with the peat soil. The glomalin extracted from the mineral soils showed higher ash percentages compared to that extracted from peat because of the latter's higher total organic C content. Also, the high Na content among the whole soils was presumably the result of an inadequate purification process. That is, despite careful dialysis of the concentrated protein, Na residue persisted indicating a need to provide greater assurance of protein purity in future extractions. To date, no information regarding glomalin's ash content has been reported in the literature.

It has been reported that glomalin is comprised of N-linked oligosaccharides (Wright and Upadhyaya, 1998; Wright et al., 1998). The presence of N-linked oligosaccharides on arbuscular mycorrhizal (AM) fungus hyphal protein was used to support a proposal that glomalin is a glycoprotein (Wright et al. 1998). If glomalin is a glycoprotein, it would contain significant amounts of mannose units linked to N-acetylglucosamine groups (Kyte, 1995). The NMR spectra of glomalin show very little carbohydrate carbon (Fig. 1). Even BSA, which is not considered a glycoprotein, contains more carbohydrate than the glomalin extractions (Fig. 1 and Table 2). Given glycoproteins high mannose composition, one would have expected that glomalin's ¹³C NMR spectra show a significant carbohydrate fingerprint (Fig. 1). Literature suggests that glomalin contains approximately 60% carbohydrate (Wright and Upadhyaya, 1998) which is contrary to our findings (Table 2 and Fig. 1).

Another interesting observation is how similar glomalin is to humic substances despite suggestion that glomalin's structure differs from that of humic acid (Wright, 2002). The solid-state ¹³C NMR spectrum of the IHSS Pahokee Peat humic acid (PHA) fraction (IHSS, 2005) bears a carbon type distribution similar to that of the glomalin samples evaluated in this study. The PHA contains high aromatic (47%) and carboxyl (20%) carbon types and relatively low aliphatic and carbohydrate carbon (IHSS, 2005). Furthermore, the molar H:C and C:N ratios of the PHA are 0.81 and 17.8, respectively, which are similar to the glomalin ratios reported here (Table 1). Mao et al. (2000) also showed how certain humic acids contain very little aliphatic (12%) and carbohydrate (14%), but large amounts of aromatic and carboxyl carbon (i.e., 48% and 26%, respectively). Mao et al. (2000) discussed how this functional group arrangement could be expected with older humic acids given the long exposure to microorganism attack and the more rapid degradation of the easily decomposing compounds such as proteins, carbohydrates, and phenolic groups. Additional ¹³C NMR experiments need to be performed on the humic acid fractions of the Poinsett and Hetland soils to lend additional support to the humic acid and glomalin structural similarities.

Soil Type	Total Organic Carbon††	Mass of Extract¶	Protein Content#	Carbon	Hydrogen	Nitrogen	Oxygen- Merz	Ash	Iron	Phosphorus	Sodium	H:C	C:N
	%	mg g ⁻¹	mg g ⁻¹				%						
Poinsett ‡	3.5	41	64.3	45.3	5.90	3.97	44.8	57.19	2.35	0.0998	7.22	1.6	13.3
Poinsett §		60	53.8	41.4	6.31	3.37	48.9	65.96	1.68	0.0893	10.60	1.8	12.2
Hetland ‡	2.8	50	63.1	48.9	4.89	3.67	42.5	64.04	2.48	0.0726	9.11	1.2	15.6
Hetland §		58	45.1	44.3	5.69	3.60	46.4	74.73	1.57	0.0495	12.43	1.5	14.4
Pahokee Peat ‡	45.1	605	111.8	58.6	4.11	4.14	33.2	31.56	0.536	0.0330	9.05	0.85	14.5
Pahokee Peat §		488	113.7	56.5	4.25	4.04	35.2	17.51	0.488	0.0424	6.30	0.90	16.3
BSA	nd	nd	nd	53.9	7.08	15.5	23.5	1.34	<0.001	0.0148	0.596	1.6	48.6

Table 1. Characteristics of whole soil, glomalin extracted material, and bovine serum albumin (BSA) standard. †

All elemental analyses reported on a water-free basis. All non-ash elements (i.e., C, H, N, and O) also reported on an ash-free basis. †

; § †† 1.0 M HCl precipitation; 0.1 M NaOH reconstitution

20% TCA precipitation; 100 mM sodium borate reconstitution

Percent organic C of whole soil

milligrams of extracted material per gram of soil ¶ #

milligrams of Bradford sensitive protein per gram of extracted material

not determined nd

Glomalin Type	Aliphatic (0-50 ppm)	Carbohydrate (50-108 ppm)	Aromatic (108-160 ppm)	Carboxyl (160-200 ppm)	Carbonyl (200-220 ppm)
			%		
Poinsett †	9.2	4.7	44.3	30.2	1.6
Poinsett ‡	10.6	9.9	50.8	26.7	2.7
Hetland †	5.3	16.0	49.2	27.7	1.8
Hetland ‡	4.4	7.1	42.1	24.2	2.2
Pahokee Peat †	3.9	11.9	49.1	30.4	4.7
Pahokee Peat ‡	6.1	3.8	41.0	26.6	2.4
BSA	53.5	14.9	11.9	18.9	0.8

Table 2. Carbon type distribution of glomalin extracted from whole samples.

† ‡

1.0 M HCl precipitation; 0.1 M NaOH reconstitution 20% TCA precipitation; 100 mM sodium borate reconstitution



Figure 1. ¹³C DPMAS NMR spectra of glomalin extracted from Hetland and Poinsett soils, Pahokee peat and bovine serum albumin . HCl precipitation/NaOH reconstitution (A) and TCA precipitation/sodium borate reconstitution (B).

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PART II:

Information Transfer Program: The first year's results have been presented at a national and international meeting and a manuscript has been prepared and is currently in review. Publication of the manuscript in either the Environmental Science and Technology or Soil Science Society of America Journal is imminent. Furthermore, the results of this study, which seem to refute current understanding of glomalin's structural character, have prompted interest and interdepartmental discussions among other researchers at SDSU.

Student Support: This project made it possible for an undergraduate student from St. Olaf College in Northfield, MN, Ms. Erin Mercer, to participate in the National Science Foundation's (NSF) Research Experiences for Undergraduates (REU) program at SDSU. Ms. Mercer was able to present her work at the Sigma Xi Annual Meeting and Student Research Conference in Montréal, Quebec and at the National Meetings of the American Chemical Society in San Diego, California.

Invertebrate and Aquatic Plant Studies of Two Mesotrophic Lakes in South Dakota

Basic Information

Title:	Invertebrate and Aquatic Plant Studies of Two Mesotrophic Lakes in South Dakota
Project Number:	2004SD20B
Start Date:	3/1/2004
End Date:	2/28/2006
Funding Source:	104B
Congressional District:	First
Research Category:	Not Applicable
Focus Category:	Ecology, Acid Deposition, Water Quality
Descriptors:	
Principal Investigators:	David R. German

Publication

Annual Progress Report

State Water Resources Institute Program (SWRIP) March 2004 to February 2005

Title: Invertebrate and Aquatic Plant Studies of Two Mesotrophic Lakes in South Dakota

Principal Investigator: Mr. David R. German, South Dakota Water Resources Institute

Introduction

The purpose of this study was to describe the macro-invertebrate fauna, the aquatic macrophyte community, and current trophic state of two relatively rare lake habitats in South Dakota. The potential for exotic species such as zebra mussels, eurasian water milfoil and the rusty crayfish to negatively impact the native fauna in these lakes was a concern.

Introduced exotic species could have a severe impact on native flora and fauna in these lakes. In 1986, a ship released ballast water into Lake St. Clair, Michigan and introduced the zebra mussel. This organism can kill native clams and competes with larval fish and other aquatic organisms for food. The zebra mussel has spread throughout the Great Lakes and has been found in the Mississippi and Minnesota Rivers. If this exotic species is introduced into Enemy Swim and Pickerel Lakes it is expected to have a large impact on the ecological balance.

A second exotic species, Eurasian water milfoil, has already been introduced to Lake Sharp in South Dakota. Milfoil is a fast growing aquatic weed that crowds out native plants and forms dense mats in shallow water. This plant can reproduce from a single fragment and is easily carried from lake to lake on boats and trailers. Fishermen traveling from Minnesota to fish in South Dakota waters may eventually introduce zebra mussels and/or milfoil to these habitats. South Dakota fishermen traveling to the Missouri River or out-of-state lakes are also potential carriers of exotics back to South Dakota.

If invasion by exotic species does occur, data from this study would allow future managers set goals for reestablishing a more natural ecosystem and mitigate the impacts of the exotic species. Data presented in this summary report includes invertebrates and plants collected since 2002 and results of water sampling conducted in 2004.

Objectives

The objectives of this research were to:

1). To prepare a list of aquatic macro-invertebrates and their relative abundance for all major habitats in Enemy Swim and Pickerel Lakes.

2). To prepare a list of aquatic plants and their general distribution in both lakes.

3). To assess the current trophic state of the lakes by monitoring selected water quality parameters.

Lake Description

Enemy Swim is natural glacial lake located in northeast Day County about eight miles north of the town of Waubay, South Dakota. The lake covers approximately 2,146 acres and has a 22,310-acre watershed located mostly in Roberts County. The lake is not deep enough to form a thermally stratified system in most years (German, 1997). Most natural lakes in South Dakota are simple basins, but Enemy Swim has been described by Game, Fish and Parks Fisheries personnel as a "complex lake basin with highly variable substrate including rock, boulders, gravel, cobble, sand, etc." The varied habitat accounts for a diverse population of fish; twenty-one species have been reported in Enemy Swim Lake.

Pickerel Lake is also a natural glacial lake located in northeastern Day County about ten miles north of the town of Waubay, South Dakota. The lake covers approximately 955 acres to an average depth of 22 feet, and a maximum depth of 43 feet. The lake bottom is predominately rubble with scattered areas of sand and gravel. Silt and organic clay are found in the bays and deeper areas of the lake. Haworth (1972) reported that the north bay of the lake contains 24 feet of sediment, which has accumulated over the 12,000 years since the lake was formed. The lake is deep enough to thermally stratify during the summer months (Day Conservation District, 1991, German, 1996).

Pickerel Lake is the deepest natural lake in South Dakota and also has a highly variable substrate with many of the same characteristics as Enemy Swim. The main difference between the lakes is Enemy Swim has an extensive system of shallow bays whereas Pickerel Lake has fewer bays and much more deep water habitat.

Enemy Swim and Pickerel Lakes are mesotrophic to lower eutrophic which represents a relatively rare habitat in South Dakota. Most natural lakes are eutrophic to hypereutrophic and many have been identified as impaired because they are not meeting their designated beneficial uses. The State of South Dakota has assigned the following beneficial uses to both Enemy Swim and Pickerel Lakes:

- Warm water permanent fish life propagation
- Limited contact recreation

- Immersion recreation; and
- Wildlife propagation and stock watering

Methodology

Objective 1: Aquatic macro-invertebrates

Shoreline habitats sampled for macro-invertebrates included rocky/rubble, sand/gravel, and muddy vegetation and were sampled at several locations. Mid-lake samples were collected in several locations to describe deeper water habitats. Samples were collected by a variety of methods including the use of Eckman dredges and a Wildco Biological Dredge. Manual collection of organisms by D-frame dip net and picking organisms from rocks, plants, and submerged wood was also conducted. Snorkeling and scuba gear were used to collect clams in deeper waters. A photographic history of many organisms collected was also kept during 2002 and 2003 and was expanded in 2004. Hester Dendy samplers were placed in several shoreline locations to gather quantitative data on macroinvertebrate populations in 2004. (EPA, 1990 and APHA, 1985).

Students participating in the "Lakes Are Cool" program collected additional macroinvertebrate samples using a variety of methods including examination of submerged wood, rocks, vegetation, and detritus, which was examined in white pans or wash buckets. Participation by the students increased the number of macro-invertebrates collected, especially the more rare forms such as fishfly larvae and water scorpions.

Objective 2: Aquatic macrophytes

Plants were collected by wading in shallow water and by snorkeling. All sampling locations were recorded using a portable GPS unit. Aquatic plant identifications were verified by Dr. Gary Larson at South Dakota State University (SDSU). Several specimens were pressed and added to the SDSU Herbarium collection under the direction of Dr. Gary Larson.

Objective 3: Trophic State

Trophic state was assessed by using the same water quality monitoring methods used during the Lake Protection study in 1991-1995 (German, 1997). In-lake water quality samples were collected with a Van Dorn-type water sampler from three mid lake stations using a boat. A composite surface sample for the lake was formed by mixing equal amounts of water from each site. A composite near bottom sample was formed by mixing water collected near the bottom from each of the three sites in each lake. Parameters analyzed on lake samples included:

- 1. Total phosphorus
- 2. Total dissolved phosphorus
- 3. Organic nitrogen

- 4. Ammonia
- 5. Nitrate + nitrite
- 6. Suspended solids
- 7. pH
- 8. Air and water temperature
- 9. Dissolved oxygen
- 10. Secchi depth
- 11. Chlorophyll a (surface samples only)
- 12. Fecal coliform bacteria (surface samples only)

Water sampling was conducted at Enemy Swim Lake in mid June, July and August in 2004. Sampling was conducted at Pickerel Lake in mid May, June, July, August and September in 2004. Dennis Skadsen from the Day Conservation District, the Pickerel Lake Sanitary District, and the Enemy Swim Lake Sanitary District contributed to this effort.

Results

Objective 1: Aquatic macro-invertebrates

The invertebrate fauna in both lakes are more diverse than expected based on published studies of the invertebrate fauna in other South Dakota lakes. (Benson and Hudson 1975, Boehmer et. al. 1975, Donaldson 1979, Gengerke and Nickum 1972, German 1978, Hartung 1968, Hudson 1970, Schmulbach and Sandholm 1962, Smith 1971, Wolf and Goeden 1973). The presence of fishflies and stoneflies was particularly surprising because they had not been reported from this area of South Dakota prior to this study. Johnson (1997) first reported the presence of fishflies in South Dakota based on larvae collected in Lacreek refuge. The first adults reported in the state were collected during this project at both Enemy Swim and Pickerel Lakes in 2002. These specimens have been deposited in the Insect Research Collection at SDSU. Insects comprised the largest portion of the invertebrate fauna. The list of macro-invertebrates collected and identified so far at Enemy Swim and Pickerel Lakes is presented in Table 1. The list includes both adults and immature stages collected at both lakes. This list is a work in-progress. Additional taxa have been added since the last annual report. Additional work will be needed to complete the list, especially for the damselflies, beetles, dipteras, and caddisflies.

Insects

Order	Family	Genus/Species	Common Name
Ephemeroptera			
	Ephemeridae	<i>Hexagenia</i> sp.	
	Heptageniidae	Stenonema sp.	
	Caenidae	<i>Caenis</i> sp.	
Odonata			
	Aeshnidae		
		Anax junius	Common green darner
		Aeshna constricta	Lance-tipped darner
		Aeshna interrupta	Variable darner
	Corduliidae		
		Epitheca cynosura	Common basketail
	Limbellulidae		
		Libellula luctuosa	Widow skimmer
		Libellula Lydia	Common whitetail
		Libellula pulchella	Twelve-spotted skimmer
		Libellula quadrimaculata	Four-spotted skimmer
		Sympetrum costiferum	Saffron-winged meadowhawk
		Sympetrum internum	Cherry-faced meadowhawk
		Sympetrum rubincundulum	Ruby meadowhawk
		Sympetrum obtrusum	White-faced meadowhawk

Order	Family	Genus/Species	Common Name
Odonata (cont.)			
	Limbellulidae (cont.)	Sympetrum corruptum	Variegated meadowhawk
		Perithemis tenera	Eastern amberwing
		Pachydiplax longipennis	Blue dasher
		Erythemis simplicicollis	Eastern pondhawk
		Tramea lacerata	Black saddlebags
		Tramea onusta	Red saddlebags
		Leucorrhinia intacta	Dot-tailed whiteface
		Celithemis eponina	Halloween pennant
		Celithemis elisa	Calico pennant
	Coenagrionidae	Numerous species	Pond Damsels
		Enallagma antennatum	Rainbow Bluet
		Enallogma hageni	Hagen's Bluet
Trichoptera			
	Helicopsychidae	Helico borealis	Snail shell caddisfly
	Hydropsychidae		
	Hydroptilidae		Micro caddisfly
	Limnephilidae		Portable case makers
Megaloptera			
	Corydalidae	Chauliodes rastricornis	Fishfly
Hemiptera			
	Belostomatidae	Belostoma sp.	Small giant water bug
	Corixidae		Water boatman

Order	Family	Genus/Species	Common Name
Hemiptera (cont.)			
	Nepidae	Nepa apiculata	Water scorpion
	Notonectidae		Back swimmers
	Gerridae		Water strider
Coleoptera			
	Gyrinidae		Whirligig beetles
Diptera			
	Ceratopogonidae		Noseeums
	Chironomidae	Numerous species	Midges
	Sub Family	Tanypodinae:	
		Procladius sp	
	Sub Family	Chironomidae:	
		Chironomus plumosus	
		Chironomus attenuatus	
		Chironomus sp. (several)	
		Cryptochironomus sp.	
	Culicidae		Mosquitoes
	Chaoboridae	Chaoborus sp.	Phantom midge

Crustacea

Order	Family	Genus	Common Name
Amphipoda			
	Gammaridae		Scuds
Decapoda			
	Cambaridae	Orconectes virilis	Northern crayfish
		Orconectes immunis	Calico crayfish
		Unidentified Species	
		Unidentified Species	

Snails

Order	Family	Genus	Common Name
Lymnophila			
	Physidae		Tadpole snails
	Lymnaeidae		Pond snails

Clams

Order	Family	Genus	Common Name
Pelecypoda			
	Unionidae	Lampsilis	Fat mucket
		Anodonta grandis	Giant floater
Hirudinea (leech	ues)		
Order	Family	Genus	Common Name

Objective 2: Aquatic macrophytes

The list of macrophytes collected and identified so far at Enemy Swim and Pickerel Lakes is presented in Table 2. No new records of aquatic macrophytes for the state or for the area were recorded. There was no evidence of Eurasian water milfoil in either lake.

Table 2. Macrophytes collected at Enemy Swim and Pickerel Lakes

Common Name	Scientific Name
Water plantain	Alisma gramineum
Coontail	Ceratophyllum demersum
Needle spikesedge	Eleocharis acicularis
Spikerush	Eleocharis erythropoda
Mare's-tail	Hippuris vulgaris
Water milfoil	Myriophyllum sibiricum
Naid	Najas flexilis
pondweed	Potamogeton friesii
Variable pondweed	Potamogeton gramineus
Illinois pondweed	Potamogeton illinoensis
Floatingleaf pondweed	Potamogeton natans
Sago pondweed	Potamogeton pectinatus
Whitestem pondweed	Potamogeton praelongus
Claspingleaf pondweed	Potamogeton richardsonii
Flatstem pondweed	Potamogeton zosteriformis
Widgeon-grass	Ruppia cirrhosa
Arrowhead	Sagittaria latifolia
Hardstem bulrush	Schoenoplectus acutus
River bulrush	Schoenoplectus fluviatilis
Common bladderwort	Utricularia vulgaris
Water stargrass	Zosterella dubia

Objective 3: Trophic State

Water quality data collected for Enemy Swim Lake in 2004 is presented in Table 3. Water quality data collected for Pickerel Lake in 2004 is presented in Table 4.

Trophic state is a way of describing how productive or enriched a lake is compared to other lakes. Lakes range from nutrient poor (oligotrophic), to moderately rich (mesotrophic), to highly enriched (eutrophic), to excessively enriched (hypereutrophic). Pickerel Lake and Enemy Swim Lake exhibited characteristics of lakes that are described as mesotrophic to early eutrophic in 2004 (Tables 3 and 4).

Parameter	Unit	М	ay	Ju	June July		lly	August		September	
		5/19	2004	6/17/	2004	7/14/2004		8/12/2004		0/14/2004	
		3/18/	2004	0/1//	2004	//14/2004		8/13/2004		9/14/2004	
Air Temperature	°C	1	3	16				19			
Transparency	ft	13	8.4	8.3		8.8		6.6		7.1	
		Surface	Bottom	Surface	Bottom	Surface	Bottom	Surface	Bottom	Surface	Bottom
Water Temperature	°C	13.7	11.5	19	18.8	24	22	19.5	19	19.8	19.5
Dissolved Oxygen	mg/L	9.3	8.8	7.3	7.2	7.5	6.9	7.2	6.7	7.4	7.3
рН		8.52	8.28	8.86	8.84	8.71	8.38	8.85	8.78	8.62	8.63
Suspended Solids	mg/L	1.67	6.25	4.5	4.75	2	5.16	6.25	8.16	4.5	7
Total Kjeldahl N	mg/L	0.786	0.963	0.828	1.039	0.748	0.976	0.93	0.926	0.748	0.898
Organic N	mg/L	0.668	0.856	0.762	0.977	0.702	0.774	0.856	0.868	0.694	0.83
Nitrate (NO ₃)	mg/L	0.02	0.02	0.03	0.02	0.04	0.04	0.03	0.04	0.03	0.03
Ammonia (NH ₃)	mg/L	0.118	0.107	0.066	0.062	0.046	0.202	0.074	0.058	0.054	0.068
Total Phosphorus	mg/L	0.006	0.027	0.02	0.023	0.014	0.035	0.019	0.031	0.018	0.027
Total Dissolved P	mg/L	0.007	0.003	0.014	0.012	0.007	0.008	0.002	N.D.	0.01	0.008

Table 3. Water quality values from Enemy Swim in 2004.

Parameter	Unit	М	ay	Ju	ne	July		August		September	
		5/18/	2004	6/17/	/2004	7/14/	2004	8/13/	2004	9/14/	2004
Air Temperature	°C	1	8	1	5			20			
Transparency	ft	4	.2	5.5		13.5		8.3		4.3	
		Surface	Bottom	Surface	Bottom	Surface	Bottom	Surface	Bottom	Surface	Bottom
Water Temperature	°C	15.4	11.4	18.6	17.4	23	19	19.7	19.1	19.7	19.6
	/-	10	<i>.</i>			6.0	1.0				
Dissolved Oxygen	mg/L	10	6.3	6.6	1.1	6.3	1.2	5.9	5.9	7.2	7.1
pН		8.71	8.5	8.63	8.3	8.5	7.91	8.68	8.61	8.7	8.35
Suspended Solids	mg/L	5.75	45	4.75	25.8	0.67	4	4.25	8.33	10.2	10.7
Total Kieldahl N	mg/L	0.959	1 2 5 2	0.862	1.086	0.856	1.02	0.959	0.927	1.02	0.946
Organic N	mg/L	0.902	1.16	0.827	0.976	0.826	0 774	0.8	0.817	0.999	0.873
Nitrate (NO ₃)	mg/L	0.04	0.02	0.02	0.02	0.03	0.03	0.03	0.03	0.03	0.03
Ammonia (NH ₃)	mg/L	0.057	0.092	0.035	0.11	0.03	0.246	0.159	0.11	0.021	0.073
Total Phosphorus	mg/L	0.039	0.144	0.033	0.098	0.018	0.037	0.034	0.037	0.057	0.049
Total Dissolved P	mg/L	0.013	0.019	0.005	0.008	0.011	0.018	0.007	0.002	0.017	0.022

Table 4. Water quality values from Pickerel Lake in 2004.

Transparency

The transparency of lake water is important to the aesthetic value of a lake. In most lakes, water transparency is determined by variations in suspended sediment or algal populations. It is used as an indirect indicator of algal populations in lakes without significant suspended sediment. In reservoir systems, transparency may be a function of sediment load or turbidity. Most of the time water transparencies in Pickerel and Enemy Swim Lakes are a function of algal populations. Transparency in Enemy Swim Lake ranged from 13.4 feet in May 2004 to 6.6 feet in August 2004 (Table 3). Transparency in Pickerel Lake ranged from 13.5 feet in July 2004 (Table 4) to 4.2 feet in August 2004 (Table 4). Transparencies in this range are common in mesotrophic to eutrophic lakes.

Suspended Solids

Low suspended solids concentrations are desirable in lakes for aesthetic reasons and maintenance of a healthy fishery. Fish populations can be affected by high suspended solids in several ways. Fish can be killed directly or their growth, resistance to disease and reproduction success may be reduced. Migrations can also be affected (EPA, 1976). High suspended solids concentrations result in reduced aesthetic value of a lake which can limit recreational use. The state standard for maintaining a warm water permanent fishery is 90 mg/l. This standard was not exceeded on any of the sampling dates reported for either lake in 2004 (Tables 3 and 4).

Phosphorus

Phosphorus is required for the growth of all forms of algae, but relatively small quantities are needed. If other nutrients are available, one pound of phosphorus can produce 500 pounds of algae (Wetzel, 1983). It is often the nutrient that limits the growth of algal populations. It is therefore also the nutrient that must be controlled in order to maintain good water quality. Total phosphorus concentrations for Enemy Swim Lake surface samples ranged from 0.006 mg/l on 5/18/03 to 0.020 mg/l on 6/17/04 (Table 3). Total phosphorus concentrations for Pickerel Lake surface samples ranged from 0.018 mg/l on 7/14/04 to 0.057 mg/l on 9/14/04 (Table 4). Using phosphorus as a trophic state index, a concentration of .03 mg/l value represents the border between mesotrophic and eutrophic lakes. A concentration of 0.03 to 0.1 mg/l would be classified as eutrophic (Wetzel, 1983). Enemy Swim would be classified as mesotrophic and Pickerel Lake would be mesotrophic to eutrophic based on phosphorus concentrations.

Dissolved phosphorus is the most available form for use by algae and other plants. It is rapidly consumed by algae and seldom reaches high concentrations in surface waters unless other factors are limiting algal growth. Dissolved phosphorus enters lakes from runoff but it is also released from sediments into the water under anoxic conditions (oxygen levels near zero). In 2004 higher concentrations of dissolved phosphorus were observed in bottom waters compared to surface waters in Pickerel Lake in May, June, and July. Pickerel Lake was weakly stratified and oxygen concentrations were lower in deeper waters compared to surface waters in May, June, and July 2004 (Table 4). This probably contributed to the release of phosphorus from the sediments in Pickerel Lake. In Enemy Swim Lake concentrations of oxygen in surface

samples was essentially the same as bottom waters in 2004 (Table 3). Slightly higher concentrations of dissolved phosphorus were observed in bottom water samples at Enemy Swim when compared to surface water samples in 2004.

<u>Nitrogen</u>

Nitrogen is present in lakes in several forms, both inorganic and organic. The inorganic forms (ammonia, nitrite and nitrate) are important nutrients available for plant growth. Organic nitrogen represents nitrogen incorporated into living (or once living) material and can be used to define trophic state. Wetzel, (1983) reports that mesotrophic lakes worldwide generally range from 0.4 to 0.7 mg/l and eutrophic lakes have up to 1.2 mg/l of organic N. Organic N concentrations in Pickerel Lake surface water ranged from 0.80 mg/l on 8/13/04 (Table 4) to 0.99 mg/l on 9/14/04 (Table 4) indicating eutrophic conditions. The median concentration of organic nitrogen in Pickerel Lake from 1991 to 1995 was .62 mg/l which represents mesotrophic conditions (German, 1997). This indicates a possible increase in productivity in the lake and a move toward more eutrophic conditions based on organic nitrogen. Organic N concentrations in Enemy Swim surface samples ranged from 0.67 mg/l on 65/18/04 (Table 3) to 0.86 mg/l on 8/13/04 (Table 3). The median concentration of organic nitrogen in Picker 3). The median concentration of organic nitrogen in Picker 3). The median concentration of organic nitrogen in Enemy Swim surface samples ranged from 0.67 mg/l on 65/18/04 (Table 3) to 0.86 mg/l on 8/13/04 (Table 3). The median concentration of organic nitrogen in Picker 3).

Ammonia is generated as an end product of bacterial decomposition of dead plants and animals and is also a major excretory product of aquatic animals. Ammonia is directly available for plant growth and is the most easily used form of nitrogen. It can support the rapid development of algal blooms if other nutrients are present. Ammonia concentrations in Pickerel Lake surface samples ranged from 0.02 mg/l on 9/14/04 (Table 4) to 0.16 mg/l on 8/13/04 (Table 4). Ammonia concentrations in Pickerel lake surface samples ranged from below the detection limit to 0.15 mg/l with a median value of 0.01 mg/l in the period from 1991 to 1995 (German, 1997). Ammonia concentrations in Enemy Swim surface samples ranged from 0.05 mg/l on 7/14/04 to 0.12 mg/l on 5/18/04 (Table 3).

Dissolved Oxygen

Adequate dissolved oxygen is necessary to maintain a healthy lake. Lakes with good oxygen concentrations throughout the year are more likely to have a diverse population of aquatic organisms rather than one that is dominated by a few hardy species. Low oxygen concentrations are detrimental to populations of many organisms and usually reduces diversity and stability in a lake ecosystem.

Oxygen concentrations can also affect other chemical parameters in lakes. For example, when anoxic conditions form at the bottom of a lake, dissolved phosphorus, ammonia, and hydrogen sulfide and other undesirable substances are released from the lake sediments into the water column. These nutrients can contribute to algal growth when lakes turn over. Ammonia and hydrogen sulfide may also be toxic to aquatic organisms if they are present in sufficient concentrations.

Oxygen concentrations in Pickerel Lake and Enemy Swim Lake surface samples were consistently above the state standard of 5.0 mg/l in 2004. This was also true of the 1991-1995 period as well (German, 1997). Weak thermal stratification and depressed oxygen concentrations near the bottom were observed in Pickerel Lake in June and July 2004. From 1991 to 1995 oxygen concentrations less than 5 mg/l were observed near the lake bottom on 10 of 15 sampling dates (German, 1997).

Overall the health of Pickerel Lake and Enemy Swim is good although they may be drifting to a more eutrophic condition. Collecting additional data in the next few years will help determine if this is normal year to year variation or a true trend. A large amount of construction has occurred around the shoreline in recent years especially on Pickerel Lake and land in CRP has been put back into production, which can cause more nutrients to enter the lake. Installation of the sewer system on Pickerel Lake has probably helped reduce nutrients from septic tanks but other measures to control nutrients from construction, farming and lawn care should be considered.

Youth Education

The scope of the project includes the participation of several local agencies. Dennis Skadsen of the Day Conservation District initiated an educational program called "Lakes Are Cool" to educate youth in the watershed about the importance of keeping lakes clean. The project involves teachers and students from local schools that participated in the "Lakes Are Cool" program that was held in 2002, 2003, and 2004 as part of the Enemy Swim Lake Watershed Improvement Project. This EPA funded watershed project sponsored by the Day Conservation District allowed local students to participate in the collection and identification of aquatic macro-invertebrates.

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Coupled Carbon-Nitrogen Geochemistry under Reducing Conditions in a Prairie Pothole

Basic Information

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Coupled Carbon-Nitrogen Geochemistry under Reducing Conditions in a Prairie Pothole

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Introduction

The physical geography of the northern Great Plains region is dominated by small, often transient, wetlands called "potholes". This area is often called the "prairie pot-hole" region. In eastern South Dakota, the potholes are underlain by the Big Sioux Aquifer and are part of the Big Sioux hydrologic basin. The Big Sioux aquifer is a shallow groundwater system that supplies water to many municipalities and rural, domestic wells. The aquifer has large storage capacity and very rapid recharge characteristics (1) primarily through surface water percolation into the aquifer.

There is an extensive literature on the habitat in and surrounding the potholes (2, 3) but relatively little on the chemical and geochemical processes taking place in them. These bodies of water are relatively shallow, well-mixed by wind and as a result, oxic. In the winter, an ice cap forms and the potholes quickly become anoxic. Thus a pothole represents an ideal, closed-system chemical reactor to study the effect of the oxidation/reduction (redox) potential on the geochemistry of an aquatic environment. There is a significant literature that the microbial and abiotic processes taking place differ significantly based on the amount of oxygen available (eg, 4).

Problem and Objectives

The initial hypothesis that will be considered is that more nitrogen is incorporated into organic matter as heterocyclic nitrogen under anoxic conditions that under oxic conditions. The nature and amount of this form of organic nitrogen is an unanswered question, and examining it will represent a significant contribution that this work could make, particularly since we can perform the reactions in a natural, closed environmental chemical reactor. This study will utilize ¹⁵N solid-state NMR and gas chromatography and electrospray ionization mass spectrometry to study the differences in nitrogen incorporation into the organic under oxic or anoxic conditions. While these are not easy measurements, they can be done at natural abundances levels on natural organic matter. Nitrogen-15 and carbon-13 isotope ratio mass spectrometry will be used to study nitrogen incorporation into the organic matter using ¹⁵N-labeled and ¹³Clabeled model compounds. In this case, the ¹⁵N and ¹³C isotopes would be very effective, and environmentally safe, tracers to monitor the actual transformations and incorporations of nitrogen into the organic matter.

As originally proposed, this was to be a three-year project. Only the first year was funded. The objectives of the first year of the project are to:

- 1. determine the annual variation in the redox potential of the pothole.
- 2. create a nitrogen and carbon mass balance for the pothole.

Methodology

Water samples were collected every other week. An ice auger was used to open the pothole as needed. Samples were collected in 1L glass containers and sealed. The pH and redox potential (Eh) were measured at the time each sample was collected.

Upon return to the laboratory analyzed immediately for total dissolved organic nitrogen (DON) content, ammonia nitrogen, total organic carbon content (TOC). Separate aliquots were used to fractionate into the sample's TOC into hydrophobic and hydrophilic acids, bases and neutrals using the XAD-8/XAD-4 resin procedure (6). Samples were freeze-dried and weighed to construct a mass balance of the TOC among the various fractions.

Solid-state ¹³C DPMAS NMR spectra were acquired as described by Mao *et al.* (7).

Principal Findings

Figure 1 shows the temporal variation of the pothole water's redox potential (Eh). The Eh rapidly becomes strongly reducing (~-350 mV) (December 29, 2004) when the pothole freezes over. During the course of the following months the ice cover melted and then refroze which explains the increase in Eh and the second minima.

In conjunction with the decrease in Eh, the dissolved organic nitrogen content (DON) increases once the water becomes anoxic (Figure 2).

The water's dissolved organic carbon (DOC) content (Figure 3) tracks the changes in DON with iceover and transition from oxygen rich to oxygen-depleted conditions. This is not surprising since decomposition generally slows under anoxic conditions.

The ¹³C DPMAS spectra of the water's DOC fractions (Figure 4) show chemical characteristics typical of these materials (8). ¹⁵N CPMAS spectra of the whole DON and the hydrophobic and hydrophilic acid, base and neutral fractions is currently in progress.

Student Support

This project has provided support for field expenses and laboratory supplies for a female graduate student pursuing a MS (December 2004 –present) and a female undergraduate research assistant (summer 2004).

Notable Awards and Achievements

The Eh and DON analyses have shown the rapid transition of the water from oxic to anoxic condition after the pothole freezes. A spike in DON content of the water is observed immediately after the water becomes anoxic.

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Figure 1. Temporal variation in the pothole water's redox potential (Eh). The pothole was ice covered on December 29, 2004 and during March 2005.



Figure 2. Temporal variation in the pothole water's dissolved organic nitrogen content (DON). The pothole was ice covered on December 29, 2004 and during March 2005.



Figure 3. Temporal variation in the pothole water's dissolved organic carbon content (DOC). The pothole was ice covered on December 29, 2004 and during March 2005



Figure 4. Solid-state ¹³C DMPAS spectra of the waters hydrophobic and hydrophilic acid fractions. The hydrophilic neutral fraction has not been characterized yet.

Establishing a Relationship Between Soil Test P and Runoff P for a South Dakota Soil Using Simulated Rainfall

Basic Information

Title:	Establishing a Relationship Between Soil Test P and Runoff P for a South Dakota Soil Using Simulated Rainfall
Project Number:	2002SD4B
Start Date:	3/1/2003
End Date:	2/28/2005
Funding Source:	104B
Congressional District:	First
Research Category:	None
Focus Category:	Agriculture, Nutrients, Water Quality
Descriptors:	None
Principal Investigators:	Frank V. Schindler, David R. German, Ronald H. Gelderman

Publication

- Guidry, A.R., F.V. Schindler, D. R. German, R. H. Gelderman, and J.R. Gerwing. 2004. Influence of Soil Test Phosphorus on Phosphorus Runoff Losses from South Dakota Soils. 34th Annual North Central Extension-Industry Soil Fertility Conference. Volume 20 pp 27-34. Des Moines, Iowa. Nov. 17-18, 2004.
- Guidry, A.R., D. R. German, R. H. Gelderman, J.R. Gerwing, and F.V. Schindler. 2004. Evaluating phosphorus loss from Midwestern soils Using Simulated Rainfall. ASA, CSSA, SSSA Annual Meetings. Oct. 31-Nov.4, 2004. Seattle, Washington, abstract No. 3277.
- 3. German, D.R., A.R. Guidry, R. H. Gelderman, F.V. Schindler, and J.R. Gerwing, 2004. Soil and runoff P relationships: Implications for lake and watershed management. ASA, CSSA, SSSA Annual Meetings. Oct. 31-Nov.4, 2004. Seattle, Washington, abstract No. 4732.
- 4. Guidry, A.R., F.V. Schindler, D. R. German, R. H. Gelderman, and J.R. Gerwing. 2005. Estimating Phosphorus Loss From Benchmark Eastern South Dakota Soils. 8th Annual Student Research Poster Session funded by the South Dakota Board of Regents, South Dakota NSF-EPSCoR Program, and the South Dakota Academy of Science. February 15, 2005. State Capitol Rotunda, Pierre, South Dakota, abstract No. 89.
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Annual Progress Report

State Water Resources Institute Program (SWRIP) March 2004 to February 2005

<u>PART I</u>.

Title: Establishing the relationship between soil test phosphorus and runoff phosphorus for South Dakota soils

Investigators: Dr. Ronald H. Gelderman, Plant Science Department

- Dr. Frank V. Schindler, Dept. of Chemistry and Biochemistry
- Mr. David R. German, Water Resources Institute

The following report discusses the results and progression of the research project titled "Establishing the relationship between soil test phosphorus and runoff phosphorus for South Dakota soils" during the funding period of March 2004 through February 2005. This project is part of an ongoing P study to evaluate the relationships that exist between surface runoff and soil P. The information gathered from this project will provide the South Dakota Dept. of Environment and Natural Resources sound scientific data in which to base their regulations of manure and fertilizer P application to agricultural land. This reporting period is the result of a no-cost extension granted through February 2005. The extension was granted to assist in the financial support of our graduate student. The specific objectives of this project are as follows:

Objective 1: Establish correlations between STP and runoff P for South Dakota soils by conducting *in situ* rainfall simulation in the field.

Objective 2: Evaluate P sorption saturation of South Dakota soil and STP relationship to runoff P by conducting controlled, laboratory rainfall simulation.

Objective 3: Use the research results to develop educational brochures, field day demonstration events, and offer manure management education to extension educators and area animal producers

Methodology:

Field Studies: The protocol for the National Research Project for simulated rainfall-surface runoff studies was used in this study (1). Ten conventionally tilled cropland areas were identified for the



Poinsett and Barnes soil series. These areas possessed similar slope and topography and were chosen based on their range in soil test phosphorus (STP) (i.e., low to high agronomic STP). The Poinsett and Barnes sites were identified in the upper Big Sioux Watershed near Watertown, SD. Rainfall simulation was conducted on each site for three consecutive days: one at field moist conditions, and two at field capacity. Rainfall was applied at an intensity of 2.5 in hr⁻¹. Runoff collection began after 2.5 min of continuous runoff, and was collected in toto for 30 min. Runoff was weighed to determine runoff volume, and a composite sample was taken for analysis. Surface

runoff water was analyzed for Total Dissolved P (organic and inorganic P species minus sediment associated P), and Total P (total dissolved plus sediment associated P) by the South Dakota

Analytical Services Laboratory. Composite soil samples were collected after raining and analyzed for STP and other select chemical parameters by the South Dakota Soil and Plant Testing Laboratory. The relationship between total dissolved P in surface runoff and STP was determined.

Laboratory Studies: Bulk 0-2 inch soil samples were collected from the ten field sites following field simulation. Soils were dried at low temperatures, sieved, and packed, in triplicate, into soil runoff boxes according to the National Research Project protocol (1). Rainfall application, runoff collection, and sample analyses for the indoor simulation were the same as described for the field studies. Representative soils samples were collected during runoff box preparation. Soil samples were crushed and passed through a 2 mm sieve. Soil P sorption saturation was determined as water extractable soil P content (mg kg⁻¹) divided by P_{MAX} (mg kg⁻¹) and multiplied by 100 (2). The P_{MAX} is the maximum amount of P that could be adsorbed by the soil and is defined as

$$P_{MAX} = (PSI + 51.9)/0.5 \tag{1}$$

where PSI is a single-point P sorption index described by (3). The PSI is calculated as

$$PSI = X(logP_F)^{-1}$$
(2)

where X is P sorbed (mg kg⁻¹) = $[(P_I)(V) - (P_F)(V)]$ (kg of soil)⁻¹, P_I is initial P concentration in sorption solution (mg L⁻¹), V is the volume of P sorption solution (L), and P_F is the final P concentration in solution (mg L⁻¹).

Principal Findings and Significance:

Objective 1: Figure 1 shows the STP and runoff P relationships developed for the Poinsett soil at the 0-2 and 0-6 soil depth using in-field simulated rainfall. The reported R^2 values at each soil depth for the Poinsett soil indicate that STP does a fair job of estimating the P concentration in runoff. Unlike the other soils studied, however, e.g., Moody, Vienna, and Kranzburg soils, the Poinsett soil exhibited a lower relative R^2 at either soil depth (Fig.1). A stepwise regression analysis indicates a significant increase in the amount of variation explained in surface runoff P concentrations when Olsen P, clay, and clay x Olsen P are added to the regression model (Table 1).

Based on the field runoff results, the Poinsett soil did not exhibit a STP threshold. However, the linearity that existed between STP and runoff P for the Poinsett soil suggests that continued manure or fertilizer P applications will eventually lead to deleterious surface water P enrichments. Livestock and crop producers will not be able to apply infinite amounts of manure or fertilizer P to soil without concern for water quality.

No field runoff evaluations were performed on the Barnes soil during this reporting period. All Barnes investigations will be performed in April through June 2005 and will be included in the final report.

It must be noted, that the relationships developed in these studies say nothing about total or dissolved P loss from the field site, but rather give only an indication of the P concentration in runoff as a function of STP. STP alone can not predict total P loss because it is a single soil parameter and does not account for climatic, topographic or agronomic influences on P loss to sensitive water bodies. These runoff relationships, when evaluated in conjunction with climate, topography and various agronomic strategies will aid state water quality experts in determining the critical level of P in surface runoff considered problematic for water resource eutrophication. Livestock and crop producers will benefit from this information in terms of being able to develop more comprehensive nutrient management plans that safeguard South Dakota's water quality.

The next phase of our ongoing P runoff research will evaluate P loss on a watershed basis and relate P loss on a microplot scale to that of a larger, watershed scale. These studies are scheduled to being in the summer of 2005 and are funded, in part, by the South Dakota Dept. of Environment and Natural Resources and the USGS State Water Resources Institute Program. *Objective 2*: Relationships between P-sorption saturation and Olsen-P and total dissolved P concentrations of indoor surface runoff for the Poinsett soil are shown in Figs. 2 and 3, respectively. As indicated, very strong linear relationships exist suggesting both Olsen-P and P saturation percentage are good predictors of total dissolved P levels in surface runoff. Note the indoor relationships are much stronger than that of the outdoor (Fig. 1). The outdoor relationships are developed under the more variable field conditions, whereas the indoor relationships are subject to less surface disruptions, e.g., earthworm activity, compared to the field soils. Controlled variability enhances the relationship explained by the predictor variables, i.e, Olsen-P and P-sorption saturation.

These data suggest that the Olsen-P, which is a routine and simple P extraction method, may prove to be a very useful environmental predictor of P loss potential for South Dakota. This information is critical to developing simple, but effective P management strategies for South Dakota.

Similar to objective one, above, no indoor P runoff and P saturation determinations were performed on the Barnes soil during this reporting period. All indoor and laboratory investigations related to the Barnes soil will be performed in April through June 2005 and will be included in the final report.

Objective 3: The progress and/or products of objective 3 are discussed in the "Information Transfer Program" section of PART II, below.

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Figure 1. The relationship between total dissolved P (TDP) in field runoff and STP (Olsen-P) of the Poinsett soil at the 0-2 and 0-6 inch depths.

Table 1. S	Stepwise re	egression	analysis of	f select	South	Dakota	Soils	using t	he p	redictor	variables
Olsen-P,	Clay Conte	ent, and O	len-P x Cla	ay conte	ent to e	stimate	total of	dissolve	ed P	concent	rations in
field surfa	ce runoff.										

Soil Type	Variables	R^2	P>F
Vienna	a. Olsen	0.93	<.0001
	b. Olsen, Clay	0.93	<.0001
	c. Olsen, Clay,	0.93	0.0007
	Olsclay		
Kranzburg	a. Olsen	0.80	0.0012
	b. Olsen, Clay	0.80	0.0078
	c. Olsen, Clay,	0.85	0.0169
	Olsclay		
Poinsett	a. Olsen	0.75	0.001
	b. Olsen, Clay	0.79	0.0048
	c. Olsen, Clay,	0.91	0.017
	Olsclay		
Moody	a. Olsen	0.93	<.0001
	b. Olsen, Clay	0.93	<.0001
	c. Olsen, Clay,	0.93	0.006
	Olsclay		



Figure 2. The relationship between total dissolved P (TDP) in indoor runoff and P sorption saturation percentage of the Poinsett soil.



Figure 3. The relationship between total dissolved P (TDP) in indoor runoff and Olsen-P of the Poinsett soil.

PART II:

Information Transfer Program: Mr. Jim Gerwing, South Dakota Soil Extension Specialist, presented the P runoff information generated from this project at his annual manure training and meetings/workshops. Meetings conducted included soil testing workshops for ag consultants and fertilizer dealers, manure application training workshops for people applying for state CAFO permits, and Certified Crop Advisor CEU workshops.

A field day demonstration of rainfall simulation, in cooperation with the SDSU Plant Science Department, College of Agriculture & Biological Sciences, and SDSU Agricultural Experiment Station & Cooperative Extension Service, was also conducted at the Northeast Research Farm Summer Tour at Watertown, SD on July 1, 2004. Two demonstrations were given that day to a total of 30 area producers, extension personnel, and various other stakeholders.





Rainfall simulation demonstrations were conducted at the Dakota Fest on August 17-19, 2004. Brochures and various P runoff handouts were made available to the public. An example of the handout can be seen in Appendix 1 of this report.

Approximately one hundred P runoff brochures were created with updated information and distributed to area producers via the cooperative extension service. Copies of the brochures were given to the directors of the South Dakota Corn Utilization and the South Dakota Pork Producers

Councils to distribute at their discretion. Approximately 100 P runoff handouts were created and distributed to producers, agronomists, and extension personnel during our field demonstration at the Northeast Research Farm Summer Tour at Watertown, SD on July 1, 2004.

P-runoff brochures are also made available to area producers and agronomists at the SDSU Soil and Plant Testing and the SDSU Water Resources Institute laboratories.

Student Support: This project helps support one graduate student enrolled in the Doctor of Philosophy program in the Atmospheric, Environmental, and Water Resources field of study. This program is in cooperation with South Dakota State University and South Dakota School of Mines. This project will be a significant part of the graduate student's dissertation.

Phosphorus Runoff Research in South Dakota



Dakota Fest (August 17-19, 2004)

Research Need:

- ✓ Declining water quality has been linked to poor manure management
- ✓ When meeting N needs of the crop with manure, P is often over-applied for crop needs
- ✓ Average soil test P (STP) levels of manured soils in South Dakota have increased
- ✓ Soil and Runoff P relationships need to be developed for South Dakota soils to ensure the development of sound P management strategies

Objectives:

- ✓ Develop correlation between runoff P and soil test P on select soils of South Dakota using rainfall simulation
- ✓ Evaluate P sorption saturation relationships to runoff P
- ✓ Relate field runoff to indoor runoff

Methods:

- ✓ Identify Field Sites (Vienna, Poinsett, Kranzburg, Barnes, and Moody)
- ✓ Sites range from low to very high STP
- ✓ Use National P protocol (SERA-17)
- ✓ Use Rainfall Simulation

Results:



Figure 1. Relationship between Olsen Soil Test P (STP) and total dissolved P in runoff for the Vienna and Moody soil series at 0-2 inch soil depth.



Financial Support Provided by:

- South Dakota Department of Environment and Natural Resources
- SD Corn Utilization Council
- SD Pork Producers Council
- National Institutes for Water Resources-US Geological Survey
- South Dakota Agricultural Experiment Station



South Dakota State University

Department of Chemistry and Biochemistry South Dakota Cooperative Extension Service Water Resources Institute Department of Plant Science



Information Transfer Program

Information dissemination is an important activity that the South Dakota Water Resources Institute (SD WRI) undertakes. We carry out our information dissemination in several ways: newsletter and website audiences, professional interaction with school-age children through water festivals and other programs as well as participating in state, federal and local committees and organizations.

SD WRI also encourages faculty, staff, and students to participate in science related activities. These activities range from giving talks, presentations and demonstrations to K-12 students as well as area producers and judging at science fairs.

Several other local and state agencies conduct cooperative research with SD WRI or contribute funding for research. Feedback to these agencies is often given in the form of presentations at state meetings, local zoning boards, and informational meetings for non-point source and research projects.

SD WRI staff also routinely responded to questions unrelated to laboratory analysis from the general public, other state agencies, livestock producers, and County Extension Agents. These inquiries include water quality and quantity, stream monitoring, surface water/ground water interactions, livestock poisoning by algae, lake protection and management, fish kills, soil-water compatibility, and irrigation drainage.

Information Transfer

Basic Information

Title:	Information Transfer
Project Number:	2002SD8B
Start Date:	3/1/2003
End Date:	3/28/2004
Funding Source:	104B
Congressional District:	First
Research Category:	Not Applicable
Focus Category:	None, None, None
Descriptors:	
Principal Investigators:	Van Kelley, David R. German

Publication

Information Transfer Program

South Dakota Water Resources Institute

Public Outreach

The South Dakota Water Resources Institute *Water News* newsletter is in its first year of publication. This is the first time that SD WRI has utilized a newsletter format to disseminate information about activities in which the Institute participates, funds, and promotes. The newsletter is published quarterly via e-mail, as well as a link on the SD WRI homepage (<u>http://wri.sdstate.edu</u>) in PDF format to view past and present issues. Water-related research including updates on present projects, notification of requests for proposals, state-wide water conditions, as well as information on youth activities are highlights in each issue.

Public outreach is an important part of the South Dakota Water Resources Institute's Information Transfer Program. This outreach takes many forms. One of the most recent at SD WRI is providing information over the Internet. SD WRI's web site (http://wri.sdstate.edu) has been redesigned to improve user access to the updated links which include publications to help diagnose and treat many water quality problems. The site allows the public to keep in touch with the activities of the Institute, gather information on specific water quality problems, learn about recent research results and links with other water resource related information available on the Web. The "Research Projects" section of the SD WRI Web is updated with past and present research projects, highlighting the Institute's commitment to improving water quality.

SD WRI made the region's drought situation a priority in its outreach/information transfer efforts by posting information for farmers and ranchers on this subject on the Institute's web page (<u>http://wri.sdstate.edu/drought.htm</u>). The SDSU Agricultural Communications Department also developed a press release and special web page dealing specifically with the drought. This web page referred producers who had questions about their water quality to the SD WRI web page.

An extensive library of information has been developed and continues to be updated on-line. Information regarding analytical services available at the Oscar E. Olson Biochemistry Labs Water Quality Laboratory and information that may be used to address drinking water problems is available on-line.

Another important component of the Institute's Information Transfer Program is the Water Quality Laboratory (WQL). The lab was consolidated with the Oscar E. Olson Biochemistry Labs in 2004. The WQL continues to provide important testing services to water users across the state. Water Resources Institute staff continues to provide interpretation of analysis and recommendations for use of water samples submitted for analysis. Assistance to individual water users in identifying and solving water quality problems is a priority of the Institute's Information Transfer Program. Interpretation of analysis and recommendations for suitability of use is produced for water samples submitted for livestock suitability, irrigation, lawn and garden, household, farmstead, heat pump, rural runoff, and land application of waste.

The severe drought in western South Dakota which continued in 2004 has demonstrated the importance of the services offered by the Water Quality Laboratory. The inherent quality of

surface waters in western South Dakota is commonly low, leading to chronic livestock production problems. However, drought has intensified this problem for livestock producers in these semi-arid rangelands. Many dugouts and ponds degraded to the point of causing cases of livestock illness and, in some instances, deaths. Lab services provided by the WQL and interpretation of results by WRI staff is important to livestock producers as they try to manage risks associated with water shortages and poor water quality. Although water quality problems in western South Dakota are common, some isolated cases of livestock illness and deaths due to poor surface water quality have occurred in eastern South Dakota as well.

SD WRI staff also routinely responded to questions unrelated to laboratory analysis from the general public, other state agencies, livestock producers, and County Extension Agents. These inquiries include water quality and quantity, stream monitoring, surface water/ground water interactions, livestock poisoning by algae, lake protection and management, fish kills, soil-water compatibility, and irrigation drainage. WRI continues to provide soil and water compatibility recommendations for irrigation permits to the SD Division of Water Rights.

Agency Interaction

The SD WRI Information Transfer program includes interaction with local, state, and federal agencies/entities in the discussion of water-related problems in South Dakota and the development of the processes necessary to solve these problems. One of the most productive agency interactions is with the Non-Point Source (NPS) Task Force. The NPS Task Force coordinates, recommends, and funds research and information projects in this high priority area. Participation on the NPS Task Force allows SD WRI input on non-point source projects funded through the state and has provided support for research in several key areas such as phosphorus in soil and lake research. Many of the information transfer efforts of the Institute are cooperative efforts with the other state-wide and regional entities that serve on the Task Force.

Another example of this interaction to solve water quality problems is a program started by the Cooperative Extension Service (CES) to help livestock producers identify unsuitable water sources. The CES provides many of its Extension Educators with hand-held conductivity meters for use in the field. If samples are shown to be marginal by field testing, they are sent to the Water Quality Lab for further analysis. Often, high sulfates limit the use of waters that have elevated conductivity.

Another important interaction is with the South Dakota Department of Environmental and Natural Resources (DENR). Completion of Total Maximum Daily Load (TMDL) studies on South Dakota lakes has been a priority for DENR over the past several years. SD WRI is providing technical assistance to local sponsors working with DENR to complete the TMDL water quality assessments on several publicly owned lakes that do not have an established lakeside community.

Several other local and state agencies conduct cooperative research with SD WRI or contribute funding for research. Feedback to these agencies is often given in the form of presentations at state meetings, local zoning boards, and informational meetings for non-point source and research projects.

Youth Education

Non-point source pollution contributes to the loss of beneficial uses in many impaired water bodies in South Dakota. An important part of reducing non-point pollution is modifying the behavior of people living in watersheds through education. Programs designed to educate youth about how their activities affect water is important because attitudes regarding pollution and the human activities that cause it are formed early in life. For these reasons, Youth Education is an important component of SD WRI's Information Transfer Program.

Water Festivals provide an opportunity for fourth grade students to learn about water. Since they began in 1992 Water Festivals have been held in seven sites including Spearfish, Rapid City, Pierre, Huron, Vermillion, Brookings and Sioux Falls. Since their inception, Water Festivals in South Dakota have impacted approximately 82,000 Fourth Grade state wide. The Big Sioux Water Festival, held locally in Brookings, has recorded attendance of over 15,600 kids, 1,950 adults and 3,250 workers since 1993. SD WRI staff members continued to support and participate in Water Festivals throughout the state in FY2004. SD WRI also supported water quality education in local schools including classroom presentations and assisting local educators with field trips. Institute staff also conducted sessions on aquatic invertebrates and their use as water quality indicators for the "Lakes are Cool" field trip in conjunction with the Enemy Swim Lake 319 project in Day County and at the Sportfishing Day held in Aberdeen annually.

Publications

Distribution of research findings to the public, policy makers and sponsors of non-point source pollution control projects is another important component of the SD WRI Information Transfer program. This is needed so that the lessons learned through research and implementation projects are not lost as the next generation of projects develops. SD WRI is committed to making this material readily available to persons within South Dakota as well as in other states. A library is maintained at SD WRI to make these materials readily available. Abstracts of research projects funded by the institute have been placed on the WRI web site along with photos and summaries showing progress on these projects will be published on the site as they become available.

Student Support

Student Support										
Category	Section 104 Base Grant	Section 104 RCGP Award	NIWR-USGS Internship	Supplemental Awards	Total					
Undergraduate	6	0	0	0	6					
Masters	0	0	0	0	0					
Ph.D.	1	0	0	0	1					
Post-Doc.	0	0	0	0	0					
Total	7	0	0	0	7					

Notable Awards and Achievements

Publications from Prior Projects