

Clean Water Act Section 319(h) Nonpoint Source Pollution Control Program Projects

Field Validation of the Texas Phosphorus Index in the Poultry Areas of Texas

Quality Assurance Project Plan
Texas State Soil and Water Conservation Board

prepared by
Soil and Crop Sciences
Texas Cooperative Extension

Effective Period: October 2004 to September 2007

Questions concerning this quality assurance project plan should be directed to:

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Section A1 Approval Sheet

Quality Assurance Project Plan for project, Field Validation of the Texas Phosphorus Index

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Title: TCE Field Operations Supervisor and QA Officer

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Name: Mark McFarland and Tony Provin

Title: Co-Principle Investigators

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Title: Soil and Crop Sciences, Interim Dept. Head

Signature: _____ Date: _____

Name: Edward Smith

Title: Interim Director, Texas Cooperative Extension

Signature: _____ Date: _____

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List of Acronyms and Abbreviations

| | |
|--------------------|---|
| AFO | animal feeding operation |
| BMP | best management practice |
| CAFO | concentrated animal feeding operation |
| CEA | County Extension Agent |
| CAR | corrective action report |
| COC | chain of custody |
| DQO | data quality objectives |
| EC | electrical conductivity |
| EPA | Environmental Protection Agency |
| GIS | geographic information system |
| GPS | global positioning system |
| MDL | method detection limit |
| NIST | National Institute for Standards and Technology |
| NO ₃ -N | nitrate-nitrogen |
| NPS | nonpoint source |
| NRCS | Natural Resources Conservation Service |
| QA | quality assurance |
| QAPP | quality assurance project plan |
| P | phosphorus |
| PI | Phosphorus Index |
| RPD | relative percent deviation |
| SOP | standard operating procedure |
| SO ₄ -S | sulfate-sulfur |
| S | sulfur |
| TCE | Texas Cooperative Extension |
| TCE SWFTL | Texas Cooperative Extension Soil, Water and Forage Testing Laboratory |
| TCEQ | Texas Commission on Environmental Quality |
| TMDL | total maximum daily load |
| TSSWCB | Texas State Soil and Water Conservation Board |
| USEPA | United States Environmental Protection Agency |
| WAF | waste application field |
| WQMP | water quality management plan |

Section A3: Distribution List

Organizations, and individuals within, which will receive copies of the approved QAPP and any subsequent revisions include:

- United States Environmental Protection Agency, Region VI
State Tribal Programs Section
US EPA Region 6
1445 Ross Ave. (6WQ-AT)
Dallas, Texas 75202

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Title: USEPA Chief, State/Tribal Programs Section
Name: Ellen Caldwell
Title: USEPA Texas Nonpoint Source Project Manager

- Texas State Soil and Water Conservation Board
P.O. Box 658
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Name: T.J. Helton
Title: TSSWCB Project Manager

Name: Donna Long
Title: TSSWCB Quality Assurance Officer

- Soil and Crop Sciences, Texas Cooperative Extension
Texas A&M University
2474 TAMU
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Name: Sam Feagley
Title: TCE Project Manager/Quality Assurance Officer - Laboratory

Name Mark Atwell
Title: TCE Field Operations Supervisor/Quality Assurance Officer - Field

Name Tony Provin
Title: TCE Laboratory Director/Co-PI

Name Mark McFarland
Title: Soil Fertility Specialist/Co-PI

Section A4: Project/Task Organization

The following is a list of individuals and organizations participating in the project with their specific roles and responsibilities:

USEPA – United States Environmental Protection Agency (USEPA), Region VI, Dallas.
Provides project overview at the Federal level.

Donna Miller, USEPA Chief, State/Tribal Programs Section

Responsible for Region VI assistance programs.

Ellen Caldwell, USEPA Texas Nonpoint Source Project Manager

Responsible for overall performance and direction of the project at the Federal level.
Approves the final products and deliverables.

TSSWCB - Texas State Soil and Water Conservation Board (TSSWCB), Temple, Texas.
Project Lead.

T.J. Helton, TSSWCB Project Manager

Responsible for project management for the TSSWCB and coordination of project activities with TCE. Responsible for facilitating technical review as part of the project. Will assist in the organization, serve on, and be an integral part of the decision-making activities of the quarterly project meetings.

Donna Long, TSSWCB Quality Assurance Officer

Responsible for determining that the Project Plan meets the requirements for planning, quality control, quality assessment, and reporting under the Section 319 program.
Conducts audits of field and laboratory systems and procedures.

Soil and Crop Sciences/TCE – Soil and Crop Sciences, Texas Cooperative Extension, Texas A&M University, 2474 TAMU, College Station, TX 77843-2474. Responsible for soil and runoff collection and analyses, data analyses, and reporting tasks for the project including development of data quality objectives (DQOs) and a quality assurance project plan (QAPP). TCE will be responsible for coordination, development, and delivery of quarterly reports and the final project report.

Sam Feagley, State Soil Environmental Specialist, TCE Project Manager

Responsible for coordinating project activities conducted by TCE including site selection, sample collection, laboratory and data analyses portions of project. Will interact with the NRCS and CEAs for site selection and soil series confirmation. Direct Ph.D. student involved in the project. Graduate student is responsible for field operations management, soil and runoff water sample collection, soil extractions and analyses done in the field and in the research laboratory discussed throughout section B. Responsible for providing TSSWCB with timely runoff water quality and soil data

reports and providing the final project report to the TSSWCB. Will be responsible for research laboratory quality assurance/quality control (QA/QC).

Mark McFarland, State Soil Fertility Specialist

Responsible for providing field help for conducting rainfall simulations and runoff and soil sample collection. Cooperates with project manager for data interpretations, information distribution, manuscript development and quarterly and final report development.

Tony Provin, State Soil Chemist, Director TCE Soil, Water and Forage Testing

Laboratory

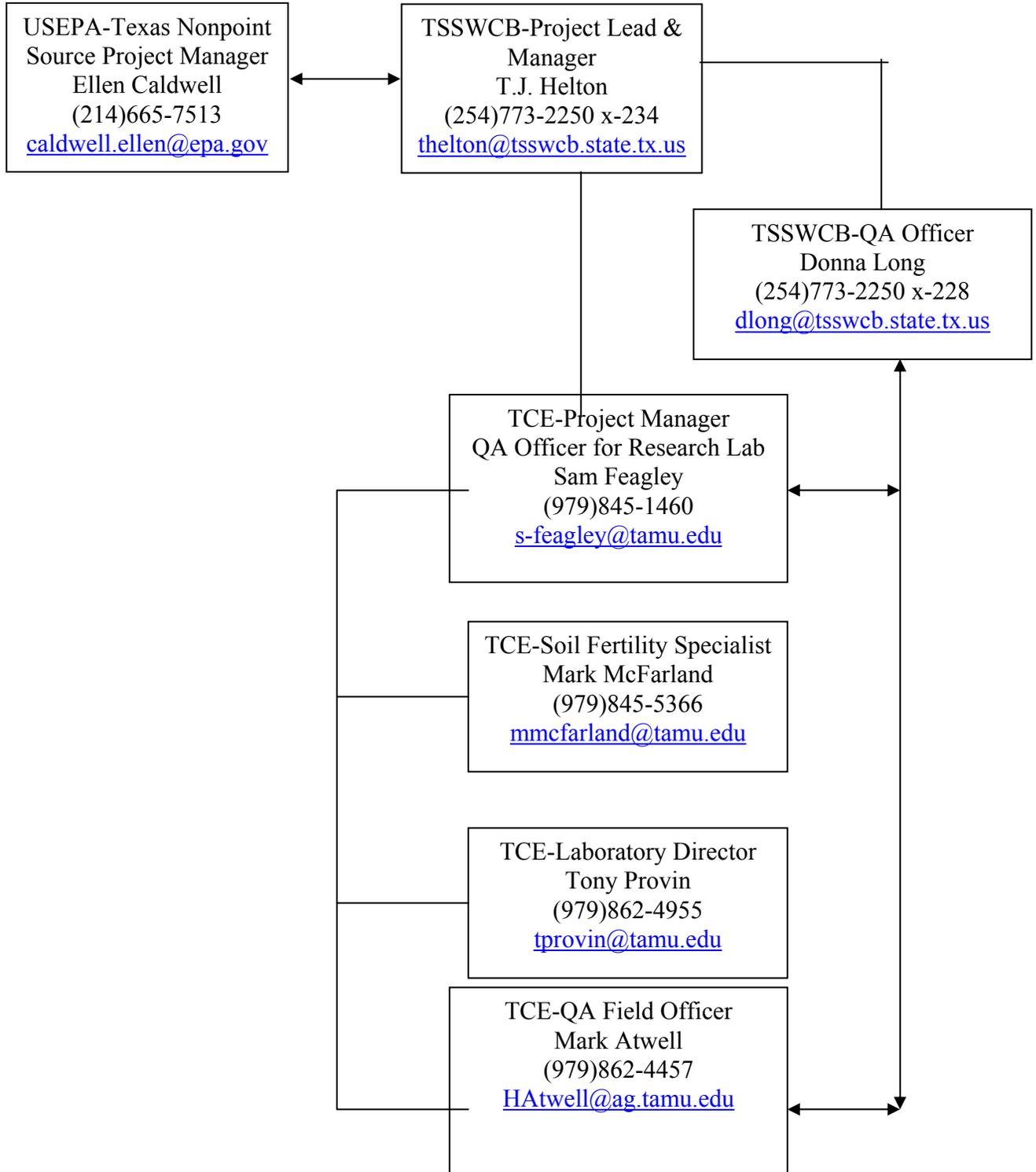
Responsible for directing TCE Soil, Water and Forage Testing Laboratory personnel involved in generating analytical data for this project. Responsible for ensuring that laboratory personnel involved in generating analytical data have adequate training in all standard operating procedures (SOPs) specific to the analyses or task performed and/or supervised. Responsible for oversight of all laboratory operations and ensuring that all QA/QC requirements in the lab are met. Responsible for documentation related to laboratory analyses. Enforces corrective action, as required. Develops and facilitates laboratory system audits. Cooperates with project manager for data interpretations, information distribution, manuscript development and quarterly and final report development.

Mark Atwell, Extension Assistant, TCE Quality Assurance Officer and Field Operations Manager

Responsible for determining that the Quality Assurance Project Plan (QAPP) meets the requirements for planning, quality control, quality assessment, and reporting for activities conducted by TCE and that data collected meet the data quality objectives of the project. Conducts audits of field and laboratory systems and procedures. Will be responsible for rainfall simulations.

Figure A4-1. Project Organization Chart

Arrow lines indicate communication only



Section A5: Problem Definition/Background

Phosphorus (P) is an essential element in plant and animal nutrition. However, it also has been identified as an element that may serve a controlling function in the occurrence of eutrophication in surface waters. Eutrophication has been identified as one of the major causes of impaired water quality in the United States (USEPA, 1996). It restricts water use for fisheries, recreation, industry, and drinking due to the increased growth of undesirable algae, aquatic weeds and resulting oxygen shortages caused by their death and decomposition (Sharpley et al., 2000).

Although watershed-scale studies are important to evaluate gross potential nutrient losses, research has clearly shown that field-scale evaluations will be most critical for effective targeting of limited resources. Significant effort has been directed toward development of predictive tools which can be used to estimate potential nonpoint source losses of P. One example is a simple P index developed by the U.S. Department of Agriculture, Agricultural Research Service, as a field-level screening tool to rank the vulnerability of fields as sources of P loss in runoff water (Lemunyon and Gilber, 1993).

The Phosphorus Index (PI) is designed to provide a basic assessment of both source and transport factors (collectively referred to as site factors) controlling P loss in surface runoff. Source factors include soil test P level, and inorganic and organic fertilizer phosphorus application rates and methods of application. Transport factors include proximity of the nearest field edge to a named stream or lake, runoff class and erosion potential. In Texas, the P index is a simple 8 x 5 matrix that combines site factors with a series of condition classes which identify Very Low-Low, Medium, High and Very High levels of runoff potential. Site factors and condition classes are assigned weighted values based on relative importance. Utilizing field specific data, condition classes are assigned for each site factor and enable calculation of a numeric point value. Total index points for an individual site are then compared to a standard index to determine overall P runoff potential for the site.

Gburek et al. (1996) found that when the original PI was applied to a larger watershed in Pennsylvania, its field rankings did not accurately identify all areas with substantive impacts on stream water quality. Sharpley et al. (2000) reported that since the overall flow systems of upland watersheds are largely fixed in space, limited opportunity exists to control or manipulate the hydrology of these systems. Thus, the most realistic and likely most effective means for modification of potential P losses will be through management of the source terms of the PI.

One key area of concern deals with the soil test P level source factor and its relationship to potential P loss. Research in Texas has shown that soil test P level can be highly dependent on several site factors including soil type, field history, P source, and soil test extractant. A first step in refining effective site classification strategies such as the PI is to evaluate the efficiency of the key soil test parameter, and its relationship to other source and transport variables.

Rainfall simulation has been used as a tool for predicting the effects of site specific characteristics on potential P loss. It is much easier and cost effective than watershed scale studies. Most importantly, it offers an opportunity to verify the accuracy of less intensive methods, such as the PI, by examining the impacts of specific source and transport parameters on measured and predicted outcomes.

In theory, the PI provides a reasonably rapid approach for planners and land managers to identify sites with the greatest potential to contribute to nonpoint source pollution. In addition, it enables comparison of selected alternative management practices which can be used to reduce P losses. However, very limited research has been conducted to provide field validation of the effectiveness of the PI for predicting actual site vulnerability. Weighting factors for both source and transport factors, and vulnerability classifications largely have been intuitively defined. In addition, other soil and site factors may play important roles in controlling the potential for P loss under specific environmental conditions.

Field studies for this project will be conducted on sites within the poultry areas of Texas from areas near Mt. Pleasant to Nacogdoches to College Station to Gonzales. Based on the results of this project and others that are currently being done or completed for the major soil series of Texas where CAFOs are prevalent, the establishment of scientifically based economic and environmental P thresholds should be established.

Section A6: Project/Task Description

Study sites in the poultry producing areas of Texas, mainly an area from Sulfur Springs to Mt. Pleasant to Nacogdoches, Sam Rayburn Reservoir watershed (Fig. A6-1), Lake O' the Pines watershed (Fig. A6-2), and areas around Brazos and Gonzales Counties (see list of potential water body segments in Table A6-1) will be selected based on predetermined characteristics designed to facilitate the evaluation of specific input or related variables of the PI. Emphasis will be placed on selection of soil series which represents the dominant series in the region and state. A total of 40 sites representing the dominant soil series used as litter application fields will be evaluated during the three year project (13 to 14 sites per year).

Soil parameters to be used in site selection are:

- a) PI risk assessment: L, M, and VH.
- b) Soil test P: L/M/H, >200 ppm.
- c) pH: non-calcareous (pH < 7.5) soils and calcareous (pH = 7.5 or greater) soils within each of the PI/soil test P parameters.
- d) Mineralogy, slope, leaching index, etc. will be documented for the PI.

For each field site, the PI will be determined based on a thorough site evaluation conducted by Texas Cooperative Extension (TCE) and/or USDA-Natural Resources Conservation Service (NRCS) personnel. Each site will be subjected to a soil characterization to determine soil series by USDA-NRCS and/or TCE staff that is a certified nutrient management specialist in Texas. In addition, soil samples will be collected from each site at depth increments of 0 to 2, 2 to 6, and 0 to 6 inches for laboratory analysis of pH, electrical conductivity (EC), and extractable P, Ca, Mg, Na, K, SO₄-S (S), B, and NO₃-N. This evaluation will include the Mehlich III method for extractable P, Ca, Mg, Na, K, and S; hot water soluble B; 2:1 water to soil pH and EC (salinity) (Provin, 2003); and soil solution soluble P (SSSP) (Jacoby and Feagley, 2003). The Texas A&M extract will not be used in this study because the TCE Soil, Water and Forage Testing Laboratory (TCE SWFTL) converted to Mehlich III January 20, 2004. The SSSP will be based on a 0.1M KCl extraction. Analyzes of all elemental components will be done in the TCE SWFTL, except for colorimetric P, which will be done in the project manager's research lab (referred to as research lab throughout the rest of the QAPP). All sample preparations, extractions, digestions, etc. will be done in the research lab.

Rainfall simulations will be conducted to estimate runoff P levels from field sites. Specific locations within each site will be selected to best represent the characteristics and properties upon which the PI characterization was based. These will include the soil series and related runoff and erosion potential classifications, slope, vegetative cover, proximity to nearest waterbody, organic and inorganic nutrient application rates and timing of application.

The rainfall simulations will be conducted using a Tlaloc 3000 rainfall simulator built by Joern's Inc. The simulator is based on the design of Miller (1987), and is an aluminum frame suspending a single low pressure, square pattern nozzle approximately 3 m above the soil surface. The simulator is capable of variable application rates up to 7.62 cm (3 in.) per hr. Based on this nozzle size and operating pressure, the actual application rate will be 7.5 cm per

hr. This rate is being used across the nation for the P Benchmark Soils Project on which Sam Feagley is a cooperator. The rate is equivalent to the 10yr/1hr storm event for Stephenville, Texas. Simulations will be conducted on 1.5 m x 2 m plots. All rainfall simulation procedures will be conducted in accordance with the Sera-17 National P Project guidelines for rainfall simulations.

One rainfall simulation on each of four plots will be conducted at each of the 40 locations, providing four replications for statistical comparison. Runoff samples (50 mL) will be collected during each simulation at 7 intervals (5, 10, 15, 20, 25 and 30 minutes, and two composites (one composite will be analyzed as the seven interval samples (1L) and one composite sample will be used for nitrate-N (250 mL non-acidified)) after runoff is initiated. Runoff weight will be recorded at one minute intervals after runoff is initiated. We are reporting weight instead of volume in the field due to the presence of sediment in samples and we are weighing the runoff. Water samples will be analyzed for pH and EC in the field, and NO₃-N (non-acidified composite sample), Ca, Mg, Na, K, P, S and B (acidified interval and composite sample) by the TCE SWFTL. Selected water samples will be analyzed for dissolved P, suspended P, and total P. The dissolved P will be analyzed using a filtered sample by the TCE SWFTL as a water sample. When sufficient sediment is collected during the rainfall simulation, a portion will be filtered, and the residue will be extracted with Mehlich III extracting solution in the research lab and analyzed by ICP in the TCE SWFTL. The total P will come from a portion of the composite sample that is digested according to Pote and Daniel (2000) in the research lab and analyzed by ICP in the TCE SWFTL.

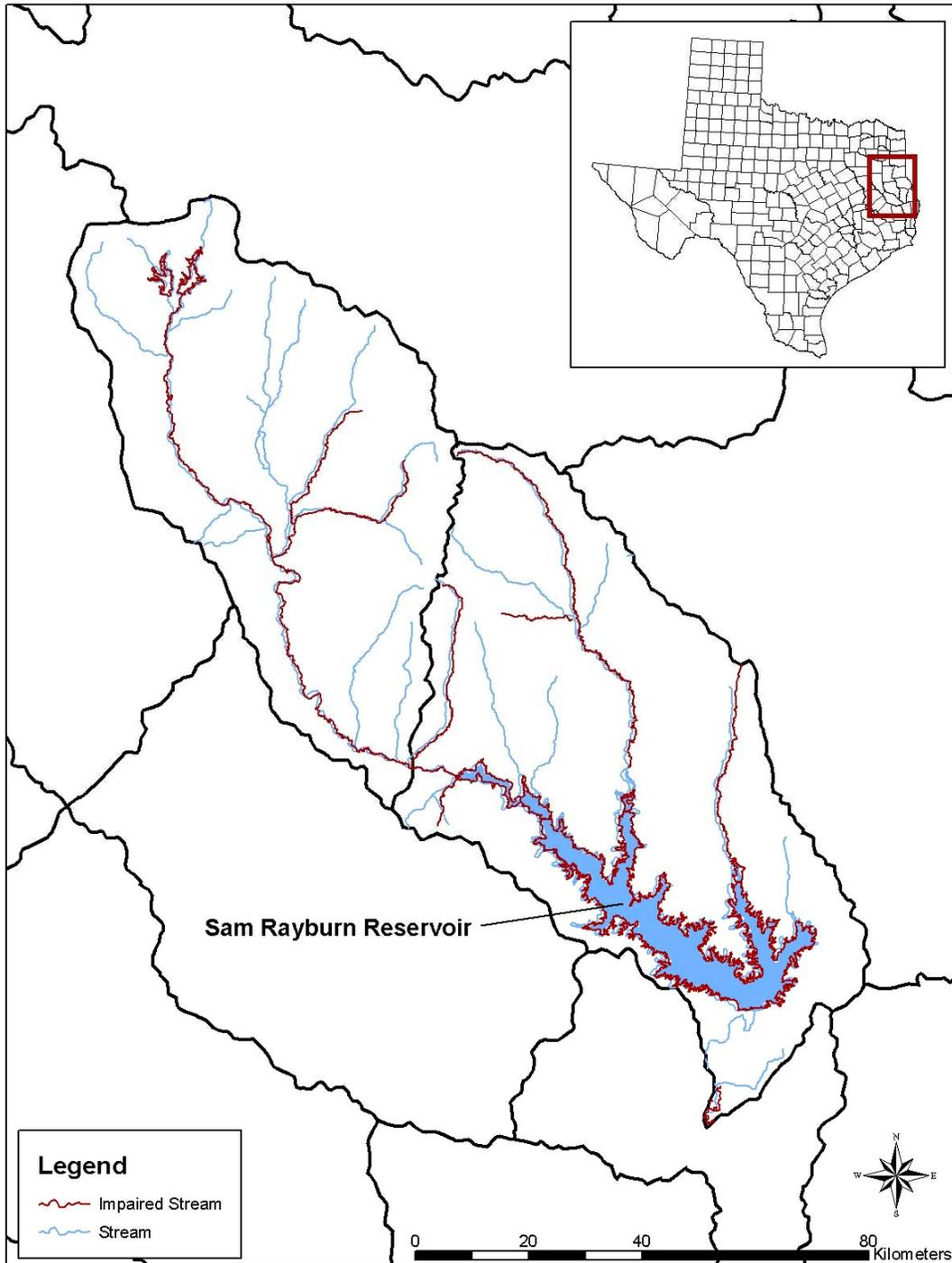


Figure A6-1. Sam Rayburn Reservoir watershed map showing impaired water body segments.

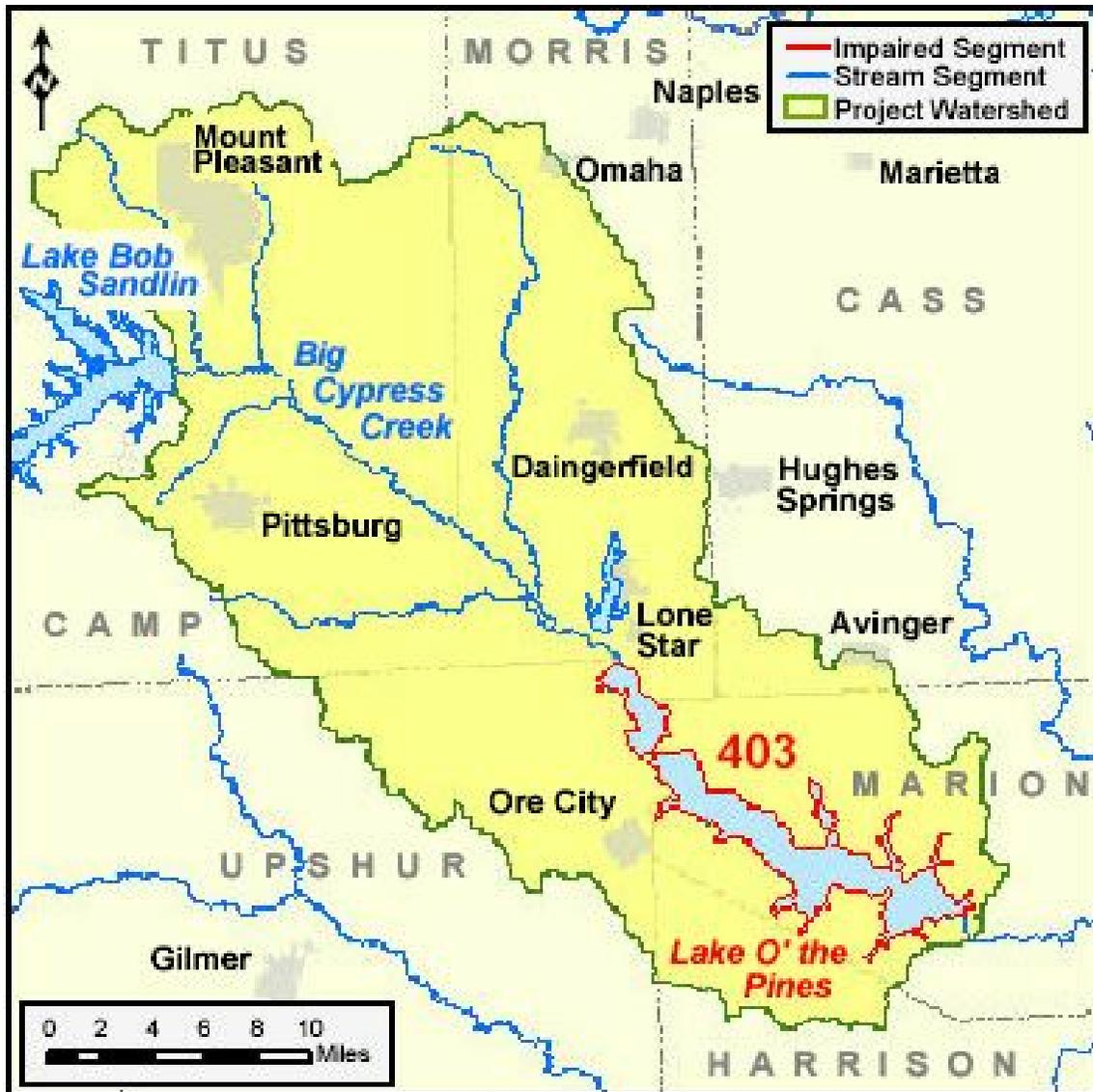


Figure A6-2. Lake O' the Pines watershed map showing impaired water body segments.

Three complementary components to the project will be client soil samples, participant litter analyses, and evaluation of three soil sample collection techniques. The project will more intensively evaluate correlations between soil test P in the target region by analyzing selected incoming client soil samples at the TCE SWFTL. Approximately 150 samples will be selected for analysis per year. These samples also will be analyzed using the Mehlich III method for extractable P, K, Ca, Mg, Na, and S; nitrate-N; and 2:1/water:soil pH and EC (salinity). One litter analysis (total N, P, K, Ca, Mg, Na, K, Fe, Mn, Cu, Zn, % moisture, pH and EC (Provin, 2003)) will be provided to each participating producer if they will allow us to collect the sample. Due to biosecurity, these samples may not be available. The three soil sampling techniques suggested by TCEQ (TCEQ, 2003) will be evaluated from a selected 20 (two fields) to 40 (one field) acre field where poultry litter has been applied for several years and has Mehlich III extractable P concentrations close to 200 ppm.

The Mehlich III and SSSP extracts will be analyzed by ICP methods for all soil samples. Selected 0-6 inch samples will be analyzed colorimetrically in the research lab for P. This will provide the needed insight into the influence of soluble organic P that will be required in order to establish more rigid laboratory methodology and protocols. All other soil test parameters will be determined using the established standard operating procedures (SOPs) of the SWFTL (Provin, 2003).

Data will be analyzed utilizing standard statistical methods including regression, analysis of variance, and mean separation.

This proposal addresses poultry agricultural activities in or near the stream segments as proposed in the DRAFT 2004 303d list that are listed in Table A6-1.

Table A6-1. Water body segments where poultry agriculture is in or near.

| Water Body Segment ID | Water Body Segment Name | Parameter |
|------------------------------|--|------------------------------|
| 0306 | Upper South Sulphur River | high pH, depressed DO |
| 0402 | Big Cypress Creek Below Lake O' the Pines | depressed DO, low pH, Hg, Pb |
| 0403 | Lake O' the Pines | depressed DO |
| 0404 | Big Cypress Creek Below Lake Bob Sandlin | bacteria |
| 0404B | Tankersley Creek | bacteria |
| 0505 | Sabine River Above Toledo Bend Reservoir | bacteria |
| 0505B | Grace Creek | bacteria, depressed DO |
| 0506 | Sabine River below Lake Tawakoni | bacteria |
| 0512B | Elm Creek | bacteria |
| 0604A | Cedar Creek | bacteria |
| 0604B | Hurricane Creek | bacteria |
| 0606A | Prairie Creek | bacteria |
| 0610 | Sam Rayburn Reservoir | Hg, depressed DO |
| 0610A | Ayish Bayou | bacteria |
| 0611 | Angelina River Above Sam Rayburn Reservoir | bacteria |
| 0611A | East Fork Angelina River | bacteria, Pb |
| 0611B | La Nana Bayou | bacteria |
| 0611C | Mud Creek | bacteria |
| 0612 | Attoyac Bayou | bacteria |
| 0615 | Angelina River/Sam Rayburn Reservoir | Hg, depressed DO |
| 1242 | Brazos River above Navasota River | bacteria |
| 1242K | Mud Creek | bacteria |
| 1242L | Pin Oak Creek | bacteria |
| 1242M | Spring Creek | bacteria |
| 1242P | Big Creek | bacteria |
| 1247A | Willis Creek | bacteria |
| 1803B | Sandies Creek | Bacteria, depressed DO |
| 1803C | Peach Creek | bacteria |

Table A6-2. Project Plan Milestones

| TASK | PROJECT MILESTONES | AGENCY | START | END |
|------|--|------------|-------|-------|
| 1.1 | Develop QAPP | TCE | Oct04 | Feb05 |
| 1.2 | Work with CEA, TSSWCB, and NRCS to select potential sites for rainfall simulations based upon selected soil chemical and physical characteristics and PI rating. | TCE | Oct04 | May06 |
| 1.3 | Collect and analyze soil samples from potential sites. | TCE | Jun05 | May06 |
| 1.4 | Based upon field observations in 1.2 and analyses in 1.3, select field sites for rainfall simulations. | TCE | Jan05 | Jun06 |
| 1.5 | Submit quarterly progress reports of project activities. | TCE & TWRI | Jan05 | Sep07 |
| 2.1 | Establish rainfall simulation sites. | TCE | Mar05 | Jul07 |
| 2.2 | Conduct rainfall simulations and collect soil and runoff. | TCE | Mar05 | Jul07 |
| 2.3 | Analyze soil and runoff samples. | TCE | Mar05 | Sep07 |
| 3.1 | Analyze soil samples for Mehlich III extractable P. | TCE | Mar05 | Aug07 |
| 3.2 | Analyze soil samples for solution soluble P and selected runoff samples for total and suspended P. | TCE | Mar05 | Aug07 |
| 3.3 | Analyze all soil samples for routine plus B. | TCE | Mar05 | Aug07 |
| 3.4 | Analyze extracted soil P using ICP and colorimetric (selected samples) techniques. | TCE | Mar05 | Aug07 |
| 4.1 | Compare initial PI risk assessment to runoff P. | TCE | Mar05 | Aug07 |
| 4.2 | Compare initial PI risk assessment to different extractable P concentrations. | TCE | Mar05 | Aug07 |
| 5.1 | Conduct initial attempt to modify the PI to predict actual P in runoff based upon P index points. | TCE | Apr05 | Aug07 |
| 5.2 | Conduct multi-county educational outreach to educate landowners and managers about runoff P and uses of PI. | TCE | Oct04 | Sep07 |
| 5.3 | Recommend potential changes of PI to NRCS and incorporate into Nutrient Management Certification Short Course and CEU opportunities. | TCE | Oct05 | Sep07 |
| 5.4 | Provide updates and training for TCEQ, TSSWCB, NRCS and other groups on PI use and proposed modifications. | TCE | Apr07 | Sep07 |
| 5.5 | Submit final report. | TCE & TWRI | Oct04 | Dec07 |

Section A7: Data Quality Objectives for Measurement Data

The Objectives of This Project are as Follows:

- 1) Determine the effects of selected soil properties on measured and predicted P runoff.
- 2) Compare and correlate soil test and soil solution extractable P levels to runoff P.
- 3) Validate and/or modify the Texas Phosphorus Index as a predictive tool for classification of field sites relative to P loss potential.
- 4) Evaluate the TCEQ soil sampling guidance for soil test P reproducibility.

Project plan milestones are listed in Table A6-2. Study sites in the poultry producing areas of Texas in and near East Texas Sam Rayburn Reservoir (Fig. A6-1) and Lake O' the Pines (Fig. A6-2) watersheds and in Central Texas will be selected based on predetermined characteristics designed to facilitate the evaluation of specific input or related variables of the PI. Emphasis will be placed on selection of soil series which represent the dominant series in the region and state. A total of 40 sites representing the dominant soil series used as poultry litter application fields will be evaluated in three years of the study (13 to 14 sites per year).

Soil parameters to be used in site selection are:

- a) PI risk assessment: L, M, and VH.
- b) Soil test P: L/M/H, >200 ppm.
- c) pH: non-calcareous (pH < 7.5) soils and calcareous (pH = 7.5 or greater) soils within each of the PI/soil test P parameters.
- d) Mineralogy, slope, leaching index, etc. will be documented for the PI.

For each field site, the PI will be determined based on a thorough site evaluation conducted by a Certified Nutrient Management Specialist working for TCE and/or NRCS. Each site will be subjected to a soil characterization by NRCS and/or TCE staff sufficient to determine soil series.

Rainfall simulations will be conducted to measure potential runoff P levels from field sites. Specific locations within each field will be selected to best represent the characteristics and properties upon which the PI characterization was based. These will include the soil series and related runoff and erosion potential classifications, slope, vegetative cover, proximity to nearest waterbody, organic and inorganic nutrient application rates and timing of application.

The rainfall simulations will be conducted using a Tlaloc 3000 rainfall simulator built by Joern's Inc. The simulator is based on the design of Miller (1987), and is an aluminum frame suspending a single low pressure, square pattern nozzle approximately 3 m above the soil surface. The simulator is capable of variable application rates up to 7.62 cm (3 in.) per hr. Based on this nozzle size and operating pressure, the actual application rate will be 7.5 cm per hr. This rate is being used across the nation for the P Benchmark Soils Project on which Sam Feagley is a cooperator. The rate is equivalent to the 10yr/1hr storm event for Stephenville, TX. Simulations will be conducted on 1.5 m x 2 m plots. All rainfall simulation procedures

will be conducted in accordance with the Sera-17 National P Project guidelines for rainfall simulations.

One rainfall simulation will be conducted on each of 4 plots at each of the 40 locations, providing four replications for statistical comparison. Runoff samples (50 mL) will be collected during each simulation at 6 intervals (5, 10, 15, 20, 25, and 30 minutes) after runoff is initiated and a composite (1000 mL for water and sediment (selected) analyses and 50 mL for NO₃-N). Runoff weight will be recorded every minute after runoff is initiated, and total runoff weight will also be recorded. All of the water samples except the NO₃-N sample will have pH and EC analyzed and recorded in the field and acidified to pH 2 with nitric acid following these analyses. Water samples will be analyzed for NO₃-N (the 50 mL composite sample), Ca, Mg, Na, K, P, S, and B by the TCE Soil, Water and Forage Testing Laboratory (Provin, 2003). All samples will be analyzed for dissolved P and selected composite samples for suspended and total P will be extracted or digested in the research lab and analyzed by ICP in the TCE SWFTL. Soil samples will be collected from each plot after the simulation is completed at 0-2, 2-6, and 0-6 inch increments. These samples will be analyzed using the Mehlich III method for extractable P, K, Ca, Mg, Na, and S. Boron will be done by the hot water method, NO₃-N by Cd reduction, and 2:1/water:soil will be used for pH and EC (salinity) (Provin, 2003), and SSSP using a 0.1M KCl extraction (Jacoby and Feagley, 2003).

A 20 (two fields) to 40 (one field) acre area will be selected to evaluate the three soil sampling techniques that are approved by TCEQ (TCEQ, 2003). The three techniques are professional judgment (recommended by TCE Soil and Crop Sciences), simple random sampling, and systematic random sampling. Initially, 0-6 inch samples will be collected on a 0.25 A grid with four cluster samples at the same depths collected every ten 0.25 A samples randomly selected across the field. Each of these samples will be analyzed for P using Mehlich III by ICP. The cluster samples will be used to determine the variability within the field. A P soil map will be constructed for the field from this information. At the time these samples are collected, the field will be evaluated and sampling sites where the professional judgment samples would be taken will be noted. Sufficient quantities of subsamples will be collected so that subsamples from each subsampling site can be removed at equal weights and combined according to each of the three sampling protocols for the composite. These samples, each subsample collected and each composite made from the subsamples to represent the three sampling protocols, will be analyzed for P using Mehlich III. All subsampling locations will be located and recorded using GPS. The three sampling techniques will be evaluated based upon reproducibility and accuracy compared to the intensive sampling. The number of samples for this part of the project will be approximately 200.

Complementary components of the project will include additional soil and litter samples. One composite litter sample from one representative house from each cooperating producer will be collected, if the producer will allow us to collect a litter sample. Biosecurity is extremely important in poultry, thus if the producer does not allow us entrance into a house, we will not be able to collect a sample. The litter sample is not a critical part of this project since the concentration of P in the litter is not a part of the PI. When litter samples are

collected, they will be analyzed for total N, P, K, Ca, Mg, Na, B, Fe, Mn, Zn and Cu, % moisture, pH and EC (Provin, 2003). Additional soil samples will be collected to more intensively evaluate correlations between soil test P in the target region by analyzing selected incoming client soil samples at the TCE Soil, Water and Forage Testing Laboratory. Approximately 150 samples will be selected for analysis per year. These samples also will be analyzed for the same analyses as the rest of the soil samples listed above.

The Mehlich III and SSSP extracts will be analyzed by colorimetric (selected samples) (research lab) and ICP (all samples) (TCE SWFTL) methods. Instrumentation will be calibrated prior to use. An internal standard soil sample (every 30 samples) (ICP only), P calibration standard (colorimetric only), and a multi-element standard (every 44 samples) (ICP only) will be analyzed. If the results for the multi-element standards are more than 10% different from the known concentrations, the instrument will be recalibrated and the previous 44 samples reanalyzed. The standards will be made using NIST or other appropriate ICP, atomic adsorption or colorimetric standards. All other soil test parameters will be determined using the established standard operating procedures (SOPs) of the TCE SWFTL (Provin, 2003).

Data will be analyzed utilizing standard statistical methods including regression, analysis of variance, and mean separation (SPSS, 2001).

Soil and water samples will be analyzed by ICP and colorimetric methods within the estimated accuracy and precision limits of measured parameters to insure data quality (Table A7-1).

Table A7-1. Estimated Accuracy and Precision Limits of Measured Parameters

NA = Not applicable; mg/L = milligrams per liter; mL = milliliters; mg/kg = milligrams per kilogram; dS/m = decisiemens per meter;

| Parameter | Precision Limits ¹ (RPD) | Accuracy Limits | SWFTL ² Code | Method Reporting Limit ³ |
|------------------------------|--|--------------------|----------------------------|--|
| Laboratory Parameters | | | | |
| Soil | | | | |
| pH | NA | ±0.2 | 0015 | 0.2 pH units |
| Electrical Conductivity | NA | ± 2% of range | 0015 | 0.05 dS/m |
| Nitrate-Nitrogen | 20% | 80-120% | 0014 | 1.0 mg/kg |
| Phosphorus (ICP) | 20% | 80-120% | 0079 | 1.0 mg/kg |
| Phosphorus (colorimetric) | 20% | 80-120% | 0079 | 0.1 mg/kg |
| Potassium | 20% | 80-120% | 0079 | 5.0 mg/kg |
| Calcium | 20% | 80-120% | 0079 | 10 mg/kg |
| Magnesium | 20% | 80-120% | 0079 | 5.0 mg/kg |
| Sodium | 20% | 80-120% | 0079 | 10.0 mg/kg |
| Sulfate-Sulfur | 20% | 80-120% | 0079 | 5.0 mg/kg |
| Boron | 20% | 80-120% | 0022 | 0.1 mg/kg |
| Runoff | | | | |
| pH | NA | ± 0.2 units | 0041 | 0.2 pH units |
| Electrical Conductivity | NA | ± 2% of range | 0040 | 0.05dS/m |
| Nitrate-Nitrogen | 20% | 80-120% | 0038 | 0.1 mg/L |
| Phosphorus | 20% | 80-120% | 0037 | 0.2 mg/L |
| Potassium | 20% | 80-120% | 0037 | 5.0 mg/L |
| Calcium | 20% | 80-120% | 0037 | 10 mg/L |
| Magnesium | 20% | 80-120% | 0037 | 5.0 mg/L |
| Sodium | 20% | 80-120% | 0037 | 5.0 mg/L |
| Sulfate-Sulfur | 20% | 80-120% | 0037 | 5.0 mg/L |
| Total Suspended Solids | 20% | NA | 0057 | 8 mg/L |
| Litter | | | | |
| Nitrogen | 20% | 80-120% | 0073 | 200.0 mg/kg |
| Phosphorus | 20% | 80-120% | 0074 | 200.0 mg/kg |
| Potassium | 20% | 80-120% | 0074 | 200.0 mg/kg |
| Calcium | 20% | 80-120% | 0074 | 200.0 mg/kg |
| Magnesium | 20% | 80-120% | 0074 | 200.0 mg/kg |
| Sodium | 20% | 80-120% | 0074 | 200.0 mg/kg |
| Boron | 20% | 80-120% | 0074 | 10.0 mg/kg |
| Zinc | 20% | 80-120% | 0074 | 3.0 mg/kg |
| Iron | 20% | 80-120% | 0074 | 3.0 mg/kg |
| Copper | 20% | 80-120% | 0074 | 3.0 mg/kg |
| Manganese | 20% | 80-120% | 0074 | 3.0 mg/kg |
| Moisture | NA | ± 2% | 0080 | 1 % |
| pH | NA | ± 0.2 units | 0071 | 0.2 pH units |
| Electrical Conductivity | NA | ± 2% of range | 0072 | 0.05dS/m |

¹ RPD = relative percent deviation

² SWFTL = Soil, Water and Forage Testing Laboratory, SOP code

³ Estimated MRL for TCE laboratory parameters as of February 22, 2005. MRLs for laboratory parameters are reevaluated about once every six months.

Although 100 percent of collected data should be available, accidents, insufficient sample volume, or other problems must be expected. A goal of 90 percent data completeness will be required for data usage. Should less than 90 percent data completeness occur, the Project Manager will initiate corrective action. Data completeness will be calculated as a percent value and evaluated with the following formula:

$$\% \text{ completeness} = \frac{SV}{ST} \times 100$$

where: SV = number of samples with a valid analytical report
ST = total number of samples collected

The project manager will determine precision of all analyses by analyzing a duplicate sample once per batch or once per 30 samples, whichever is the greater frequency. Relative percent deviation (RPD) of duplicate analyses (X_1 and X_2) will be calculated with the formula with the precision limits indicated in Table A7-1:

$$\text{Relative Percent Deviation} = \frac{(X_1 - X_2)}{(X_1 + X_2)/2} \times 100\%$$

The accuracy of the analytical process will not be monitored. This is because, the proper procedure for spiking is to add a known to the sample. Due to the reactions of each of the parameters with soil being determined, accuracy can not be determined. These reactions may include precipitation, anion exchange, and cation exchange. Instead, reproducibility will be used. Reproducibility will be determined by two methods. The first method will be to use a soil standard every 30 samples. Approximately 300 samples are analyzed per day in the TCE SWFTL, thus 10 standard soil samples would be analyzed each day samples are analyzed. The average and standard deviation will be acceptable at the 10% level. A second method will be used in the research lab for colorimetric analyses due to lower numbers of samples being analyzed at one time by randomly selected samples and analyzed 5 replicates. The average and standard deviation acceptable level will be 30%. The difference is due to the first standard soil not having litter applied and the collected samples containing litter. The litter introduces more error.

Database checks for validity will be performed on an on-going basis by the QA officers. Data will be reviewed for abnormalities or any unusual results, e.g., a multi-element standard (referred to in the TCE SWFTL as the CV standard) will be analyzed every 44 samples. If the standard deviation is greater than 10%, the samples back to the previous CV standard will be reanalyzed. Any unusual results will be traced for error sources. In the event no error is found, the data will be assumed normal and appropriate for decision determinations. If an error is found and cannot be resolved, the raw samples will be prepared again and reanalyzed. If there is not sufficient raw sample for preparation, the data will be discarded based upon the decisions of the QA officers.

The Project Manager will coordinate with the Laboratory Director, Soil Fertility Specialist, and Research Staff to ensure that proper protocols are utilized.

Section A8: Special Training Requirements/Certification

All personnel involved in sampling, sample analyses, and statistical analyses have received the appropriate education and training required to adequately perform their duties. Individuals responsible for the PI will have to be certified as a TX Nutrient Management Specialist.

Section A9: Documentation and Records

Hard copies of all field data sheets, general maintenance (GM) records, chain of custody forms (COCs), laboratory data printout sheets, field data entry sheets, and corrective action reports (CARs) will be archived by the TCE project manager for at least five years. In addition, the TCE project manager will archive electronic forms of all project data for at least five years. A CAR form is presented in Appendix A, a copy of a COC and field data sheets are presented in Appendix B, and copies of GM are presented in Appendix C.

The TCE SWFTL director will produce an annual quality assurance/quality control report, which will be kept on file at TCE with copies made available upon request. Any items or areas identified as potential problems and any variations or supplements to QAPP procedures noted in the laboratory quality assurance/quality control report will be made known to pertinent project personnel and included in an update or amendment to the QAPP.

Quarterly progress reports will note activities conducted in connection with the soil and water analyses, items or areas identified as potential problems, and any variations or supplements to the QAPP. CARs will be utilized when necessary (Appendix A). CARs will be maintained in an accessible location for reference at TCE. CARs that result in any changes or variations from the QAPP will be made known to pertinent project personnel and documented in an update or amendment to the QAPP.

Section B1: Sampling Process Design (Experimental Design)

This project is designed to evaluate the PI. The evaluation is being done through the use of rainfall simulation. Each site will be evaluated based upon the current PI, rainfall simulations will be conducted, and the load of P collected during the simulation in the runoff will be compared to the PI rating and soil test P results. The soil and water constituents that will be measured are shown in Table B1-1. The litter analyses are listed as non-critical because we will not have access to litter samples that have already been applied to fields and because of biosecurity, we may not be given access to poultry houses. When available, we will collect subsamples of litter and the listed analyses will be critical on these samples. However, they are not critical to the completion of this project. The evaluation of the three soil sampling methods detailed in Regulatory Guidance 408 (TCEQ, 2003), will be critical to the project. These samples will be analyzed using Mehlich III as soil samples listed in Table B1-1.

Table B1-1. Soil, Water and Litter Constituents

| Parameter | Status | Reporting Units |
|--------------------------|--------------|-----------------|
| Soil Parameters | | |
| pH | Critical | pH units |
| Electrical Conductivity | Critical | dS/m |
| Nitrate-Nitrogen | Critical | mg/kg |
| Phosphorus | Critical | mg/kg |
| Potassium | Critical | mg/kg |
| Calcium | Critical | mg/kg |
| Magnesium | Critical | mg/kg |
| Sodium | Critical | mg/kg |
| Sulfate-Sulfur | Critical | mg/kg |
| Boron | Critical | mg/kg |
| Runoff Parameters | | |
| pH | Critical | pH units |
| Electrical Conductivity | Critical | dS/m |
| Nitrate-Nitrogen | Critical | mg/L |
| Phosphorus | Critical | mg/L |
| Potassium | Critical | mg/L |
| Calcium | Critical | mg/L |
| Magnesium | Critical | mg/L |
| Sodium | Critical | mg/L |
| Sulfate-Sulfur | Critical | mg/L |
| Total Suspended Solids | Critical | mg/L |
| Litter | | |
| Nitrogen | Non-critical | mg/kg |
| Phosphorus | Non-critical | mg/kg |
| Potassium | Non-critical | mg/kg |
| Calcium | Non-critical | mg/kg |
| Magnesium | Non-critical | mg/kg |
| Sodium | Non-critical | mg/kg |
| Boron | Non-critical | mg/kg |
| Zinc | Non-critical | mg/kg |
| Iron | Non-critical | mg/kg |
| Copper | Non-critical | mg/kg |
| Manganese | Non-critical | mg/kg |
| Moisture | Non-critical | % |
| pH | Non-critical | pH units |
| EC | Non-critical | dS/m |

The sampling program associated with the rainfall simulation is designed to characterize water quality and quantity of simulated runoff and correlate the water quality to extractable and SSSP. At least two sites will be selected that currently are equipped with edge of field runoff collection equipment. These sites will be used to correlate the rainfall simulation runoff water quality to normal rainfall runoff water quality. These sites will most likely be located in the Lake O' the Pines Watershed. The correlation of the rainfall simulation runoff to edge of field runoff is essential to the completion of this project.

The rainfall simulations will be conducted using a Tlaloc 3000 rainfall simulator built by Joern's Inc. The simulator is based on the design of Miller (1987), and is an aluminum frame suspending a single low pressure, square pattern nozzle approximately 3 m above the soil surface. Based on this nozzle size and operating pressure, the actual application rate will be 7.5 cm per hr. This rate is being used across the nation for the P Benchmark Soils Project on which Sam Feagley is a cooperator. The rate is equivalent to the 1hr/10yr storm event for Stephenville, TX. Simulations will be conducted on 1.5 m x 2 m plots. All rainfall simulation procedures will be conducted in accordance with the Sera-17 National P Project guidelines for rainfall simulations.

One rainfall simulation will be conducted on each of 4 plots at each of the 40 locations, providing four replications for statistical comparison. Runoff samples (50 mL) will be collected during each simulation at 6 intervals (5, 10, 15, 20, 25, and 30 minutes) after runoff is initiated and a composite (1000 mL for water and sediment (selected samples) analyses and 50 mL for NO₃-N). Runoff weight will be recorded every minute after runoff is initiated, and total runoff weight will also be recorded. All of the water samples except the NO₃-N sample will have pH and EC analyzed and recorded in the field and acidified to pH 2 with nitric acid following these analyses. Water samples will be analyzed for NO₃-N (the 50 mL composite sample, filtered), Ca, Mg, Na, K, P, S, and B by the TCE SWFTL (Provin, 2003). All samples will be analyzed for dissolved P and selected composite samples for total and suspended P. Soil samples will be collected from each plot after the simulation is completed at 0-2, 2-6, and 0-6 inch increments. These samples will be analyzed using the Mehlich III method for extractable P, K, Ca, Mg, Na, and S. Boron will be done by the hot water method, NO₃-N by Cd reduction, and 2:1/water:soil will be used for pH and EC (salinity) (Provin, 2003), and SSSP (Jacoby and Feagley, 2003).

For each field site, the PI will be determined based on a thorough site evaluation conducted by TCE and/or USDA-NRCS personnel. Each site will be subjected to a soil characterization by NRCS and/or TCE staff sufficient to determine soil series.

Complementary components of the project will include additional soil and litter samples. One composite litter sample from one representative house from each cooperating producer will be collected, if the producer will allow us to collect a litter sample. Due to biosecurity issues, if the producer does not allow us entrance into a house, we will not be able to collect the sample. The litter sample is not a critical part of this project since the concentration of P in the litter is not a part of the PI. When litter samples are collected, they will be analyzed for total N, P, K, Ca, Mg, Na, B, Fe, Mn, Zn and Cu, % moisture, pH and EC (Provin, 2003). Additional soil samples will be collected to more intensively evaluate correlations between soil test P in the target region by analyzing selected incoming client soil samples at the TCE SWFTL. Approximately 150 samples will be selected for analysis per year. These samples also will be analyzed for the same analyses as the rest of the soil samples listed above.

Data will be analyzed utilizing standard statistical methods including regression, analysis of variance, and mean separation (SPSS, 2001).

Section B2: Sampling Method Requirements

Runoff water and sediment sample collection will be done according to National P Benchmark Soils Project from portable 1.5 x 2.0m frames. One rainfall simulation will be conducted on each of 4 plots at each of the 40 locations, providing four replications for statistical comparison. Runoff samples (50 mL) will be collected during each simulation at 6 intervals (5, 10, 15, 20, 25, and 30 minutes) after runoff is initiated and a composite (1000 mL for water and sediment (selected) analyses and 50 mL for NO₃-N). Runoff weight will be recorded every minute after runoff is initiated, and total runoff weight will also be recorded. All of the water samples except the NO₃-N sample will have pH and EC analyzed and recorded in the field and acidified to pH 2 with nitric acid following these analyses. Water samples will be stored in an ice chest at approximately 4°C and transported to the research lab as soon as possible. Upon arrival to the research lab, samples will be filtered. Samples collected for suspended soluble P analyses will be filtered first. The sediment will be air dried, ground and extractable nutrient analyses run. The filtrate will be analyzed as a water sample. Both samples will be stored in a refrigerator until analyses are completed in the TCE SWFTL. After the runoff event is completed, the sediment remaining in the collection portion of the frame will be recovered so that the total sediment load can be calculated. This sample will also be used from selected sites as part of the suspended P concentrations. These selected samples will be extracted with Mehlich III and digested for total P (Provin, 2003).

Approximately 15 soil subsamples from each of the four plots will be collected after the simulation to depths of 0-2, 2-6, and 0-6 inches. Samples will be combined per plot in soil sample bags provided by the TCE SWFTL and stored in the vehicle at ambient temperature. At the end of the day, soil samples will be transferred to the motel room or research lab. The samples will be air dried, pulverized to pass through a 2 mesh sieve and weighed for the various extractions. At least two blind duplicate samples will be sent to the laboratory for analyses from every 25 to 30 samples for QA/QC. A 0-6 inch P concentration will be estimated from the 0-2 and 2-6 inch samples and compared to the 0-6 inch sample. Soil samples will be collected according to Regulatory Guidance 408 (TCEQ, 2003) and on a 0.25 A grid at 0-2, 2-6 and 0-6 inch depths and processed as discussed above.

Poultry litter samples will be collected from one representative poultry house for each participant if they will allow us to collect the sample. The sample will be collected using a grid sampling technique developed and evaluated by Dr. Casey Ritz, University of Georgia, and modified by Dr. John Carey, Texas A&M. The rafters in the poultry house will be counted, divided by 7, and three evenly spaced samples will be collected going in the first direction across the house from rafters 1 to 7 and two in the return direction from rafters 7 to 14, etc. This should net 15 to 20 subsamples per poultry house. Each subsample will be collected from a whole dug through the litter to the original floor of the house, making sure none of the original floor is sampled. Approximately a one inch slice of the litter will be collected all the way down to the original floor and then the middle one inch of this slice will be placed into a clean plastic bucket. After all subsamples are collected, the litter will be thoroughly mixed and a sample for analyses collected from the mixture.

Section B3: Sample Handling and Custody Requirements

The sample number, date, sample type, changes in possession and other pertinent data will be recorded in indelible ink on the COC. The sample collector will sign the COC and transport it with the sample to the laboratory, where the laboratory staff member who receives the sample will sign. A copy of a blank COC form used on this project is included as Appendix B.

Table B3-1 delineates sample container, preservation and holding time information for parameters of interest in this project.

Each container will be marked with an identification number. A member of the team will document in a field notebook or COC form the sample number, date, sample type, initials of person collecting the samples, and comments (if needed). A sample number will be assigned to the sample in the field and data for each sample container will then be entered on a COC. The COC form will accompany all sets of sample containers. After samples are received at the research lab, they will be inventoried against the accompanying COC. Any discrepancies will be noted at that time and the COC will be signed for acceptance of custody. Sample numbers will then be recorded into a research laboratory sample log, filtered or pretreated as necessary, and placed in a refrigerated cooler in the research lab dedicated to sample storage, where required.

The project manager has the responsibility to ensure that all holding times are met. This is documented on COC for sample dates and times and on analytical data printouts for analyses dates and times. Any problems will be documented with a CAR.

Table B3-1. Sample Procedures and Handling Methods

| Parameter | SWFTL | Container | Preservation | Temperature | Holding Time |
|--------------------------|-------|--------------|-------------------------------|-------------|--------------|
| Soil Parameters | | | | | |
| pH | 0015 | Sample Bag | Air Drying | 25°C | NA |
| Electrical Conductivity | 0015 | Sample Bag | Air Drying | 25°C | NA |
| Nitrate-Nitrogen | 0014 | Sample Bag | Air Drying | 25°C | NA |
| Phosphorus | 0079 | Sample Bag | Air Drying | 25°C | NA |
| Potassium | 0079 | Sample Bag | Air Drying | 25°C | NA |
| Calcium | 0079 | Sample Bag | Air Drying | 25°C | NA |
| Magnesium | 0079 | Sample Bag | Air Drying | 25°C | NA |
| Sodium | 0079 | Sample Bag | Air Drying | 25°C | NA |
| Sulfate-Sulfur | 0079 | Sample Bag | Air Drying | 25°C | NA |
| Boron | 0022 | Sample Bag | Air Drying | 25°C | NA |
| Runoff Parameters | | | | | |
| pH | 0041 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Electrical Conductivity | 0040 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Nitrate-Nitrogen | 0038 | HDPE | None | 4°C | 28 days |
| Phosphorus | 0037 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Potassium | 0037 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Calcium | 0037 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Magnesium | 0037 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Sodium | 0037 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Sulfate-Sulfur | 0037 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Total Suspended Solids | 0057 | HDPE | Acid. HNO ₃ , pH 2 | 4°C | 28 days |
| Litter | | | | | |
| Nitrogen | 0073 | Zip-lock bag | Air Drying | 25°C | NA |
| Phosphorus | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Potassium | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Calcium | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Magnesium | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Sodium | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Boron | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Zinc | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Iron | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Copper | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Manganese | 0074 | Zip-lock bag | Air Drying | 25°C | NA |
| Moisture | 0080 | Zip-lock bag | Air Drying | 25°C | NA |
| pH | 0071 | Zip-lock bag | Air Drying | 25°C | NA |
| Electrical Conductivity | 0072 | Zip-lock bag | Air Drying | 25°C | NA |

SWFTL = Soil, Water and Forage Testing Laboratory Standard Operating Procedures (SOPs)

HDPE = High Density Polyethylene bottles

HNO₃ = concentrated nitric acid

°C = degrees centigrade

NA = not applicable, indefinite holding time after air drying

Section B4: Analytical Methods Requirements

The EC and pH of runoff water from simulated rainfall events will be measured in the field. The remainder of the parameters listed in Table B2-1 will be analyzed by TCE in the SWFTL and research lab as specified in preceding sections, College Station, Texas. A listing of analytical methods and equipment is provided in Table B4-1. SOPs have been established for almost all of the procedures undertaken by TCE SWFTL staff that concerns soil, water and litter analyses, and copies of the SOPs are available upon request.

In the event of a failure in the analytical system, the Project Manager will be notified. The Laboratory Manager, Quality Assurance Officer, and Project Manager will then determine if the existing sample integrity is intact, if re-sampling should and/or can be done, or if the data should be omitted.

Table B4-1. Laboratory Analytical Methods

| Parameter | SWFTL | Equipment Used |
|--------------------------|--------------|--------------------------------------|
| Soil Parameters | | |
| pH | 0015 | pH meter |
| Electrical Conductivity | 0015 | Conductivity meter |
| Nitrate-Nitrogen | 0014 | Nitrate analyzer (Cd reduction) |
| Phosphorus | 0079 | ICP, Colorimetric (selected samples) |
| Potassium | 0079 | ICP |
| Calcium | 0079 | ICP |
| Magnesium | 0079 | ICP |
| Sodium | 0079 | ICP |
| Sulfate-Sulfur | 0079 | ICP |
| Boron | 0022 | ICP |
| Runoff Parameters | | |
| pH | 0041 | pH meter |
| Electrical Conductivity | 0040 | Conductivity meter |
| Nitrate-Nitrogen | 0038 | Nitrate analyzer (Cd reduction) |
| Phosphorus | 0037 | ICP |
| Potassium | 0037 | ICP |
| Calcium | 0037 | ICP |
| Magnesium | 0037 | ICP |
| Sodium | 0037 | ICP |
| Sulfate-Sulfur | 0037 | ICP |
| Total Suspended Solids | 0057 | Metler Balance |
| Litter | | |
| Nitrogen | 0073 | Nitrate analyzer (Cd reduction) |
| Phosphorus | 0074 | ICP |
| Potassium | 0074 | ICP |
| Calcium | 0074 | ICP |
| Magnesium | 0074 | ICP |
| Sodium | 0074 | ICP |
| Boron | 0074 | ICP |
| Zinc | 0074 | ICP |
| Iron | 0074 | ICP |
| Copper | 0074 | ICP |
| Manganese | 0074 | ICP |
| Moisture | 0080 | Metler Balance |
| pH | 0071 | pH meter |
| Electrical Conductivity | 0072 | Conductivity meter |

SWFTL = Soil, Water and Forage Testing Laboratory Standard Operating Procedures (SOPs)

Section B5: Quality Control Requirements

The TCE SWFTL and research lab will determine the precision of their analyses. Annual laboratory audits, sampling site audits, and quality assurance of field sampling methods will be conducted by TCE QA officers.

Table B5-1 outlines the required analytical quality control for the parameters of interest. There will be no spiked sample analyses. The reason no spikes can be used is due to the different adsorptive capacities of different soil types for most of the elements being measured in this study. Therefore, adding elements to soils or runoff containing soil particles would always yield varying returns due to the chemical properties of soils.

The use of approved sampling and analytical methods will ensure that measured data accurately represent field conditions. Table A7-1 in Section A7 “Data Quality Objectives” lists the required accuracy limits for the parameters of interest. The completeness of the data will be affected by the reliability of the equipment, frequency of field and laboratory errors or accidents, and unexpected events; however, the general goal requires 90 percent data completion.

In the database, missing values will be left as blanks. Graphical screening of the data will be used to highlight questionable data points. Questionable data will be traced through the COC forms, CARs, and, as necessary, through research laboratory notebooks and field data sheets to ensure that data are properly entered. Changes will be made only if an error is found in transcription into database. Values determined to be below the laboratory method detection limit will be noted as such in the comment column of the database and used in statistical analyses as one-half the method detection limit (MDL), as recommended by Gilliom and Helsel (1968) and Ward et al. (1988). Values that are greater than the upper method detection limit will be diluted or re-extracted at a lower soil to extractant ratio and reanalyzed.

It is the responsibility of the project manager to verify that the data are representative. The chemistry data’s precision, accuracy, and comparability generated in the TCE SWFTL will be the responsibility of the laboratory director. The project manager has the responsibility of determining that the 90 percent completeness criteria is met, or will justify acceptance of a lesser percentage. All incidents at TCE requiring corrective action will be documented through use of CARs (Appendix A).

Table B5-1. Required Quality Control Analyses

| Parameter | Blank | Standard | Duplicate |
|--------------------------|--------------|-----------------|------------------|
| Soil Parameters | | | |
| pH | NA | A | B |
| Electrical Conductivity | NA | A | B |
| Nitrate-Nitrogen | A | A | B |
| Phosphorus | A | A | B |
| Potassium | A | A | B |
| Calcium | A | A | B |
| Magnesium | A | A | B |
| Sodium | A | A | B |
| Sulfate-Sulfur | A | A | B |
| Boron | A | A | B |
| Runoff Parameters | | | |
| pH | NA | A | B |
| Electrical Conductivity | NA | A | B |
| Nitrate-Nitrogen | A | A | B |
| Phosphorus | A | A | B |
| Potassium | A | A | B |
| Calcium | A | A | B |
| Magnesium | A | A | B |
| Sodium | A | A | B |
| Sulfate-Sulfur | A | A | B |
| Total Suspended Solids | NA | NA | B |
| Litter | | | |
| Nitrogen | A | A | B |
| Phosphorus | A | A | B |
| Potassium | A | A | B |
| Calcium | A | A | B |
| Magnesium | A | A | B |
| Sodium | A | A | B |
| Boron | A | A | B |
| Zinc | A | A | B |
| Iron | A | A | B |
| Copper | A | A | B |
| Manganese | A | A | B |
| Moisture | NA | NA | B |
| pH | NA | A | B |
| EC | NA | A | B |

A - Where specified, blanks and standards shall be performed each day that samples are analyzed.
B - Where specified, duplicate analyses shall be performed every 30 samples each day that samples are analyzed. At least one duplicate sample will be run each day of analyses.
NA indicates not applicable

Section B6: Equipment Testing, Inspection, & Maintenance Requirements

Manufacturers' recommendations for scheduling testing, inspection, and maintenance of each piece of equipment will be followed or exceeded. Maintenance and inspection logs will be kept on each piece of TEC chemistry laboratory equipment; general maintenance checklists will be filled out for rainfall simulation equipment at least once per week of equipment usage.

A general maintenance (GM) sheet will be filled out for each GM inspection (Appendix C). The GM sheet contains a check list for all equipment and routine maintenance activities. Any equipment, which needs attention, will be serviced during the inspection, when possible, with all additional activities described in the comment section. Any maintenance or other required activities that can not be completed during the GM inspection will be reported to the QA officer and the project manager then arranges for resolution. The QA officer checks the GM sheets and schedules additional maintenance to ensure that any problems or potential problems are resolved as soon as possible. Some back-up equipment will be maintained by TCE so that failing equipment can be replaced if possible.

To minimize downtime of all measurement systems, all field measurement and sampling equipment, in addition to all laboratory equipment, must be maintained in a working condition. Also, backup equipment or common spare parts will be made available if any piece of equipment fails during use so that repairs or replacement can be made quickly, allowing measurement tasks to be resumed. All staff who use chemicals, reagents, equipment whose parts require periodic replacement and other consumable supplies receive instruction concerning the remaining quantity (unique for each supply) which should prompt a request to order additional supplies.

Section B7: Instrument Calibration and Frequency

All instruments or devices used in obtaining data will be used according to appropriate laboratory or field practices. Written copies of TCE's SOPs are available for review upon request.

Standards used for instrument or method calibrations shall be of known purity and be National Institute for Standards and Testing (NIST) traceable whenever possible. When NIST traceability is not available, standards shall be of American Chemical Society (ACS) or reagent grade quality, or of the best attainable grade. All certified standards will be maintained traceable with certificates on file in the laboratory. Dilutions from all standards will be recorded in the standards log book and given unique identification numbers. The date, analyst initials, stock sources with lot number and manufacturer, and how dilutions will also be recorded in the standards log book.

Normally calibrations are performed with a minimum of four standards of increasing concentrations and a reagent blank. Standards shall not exceed the linear range of the instrument or method. Calibration shall be verified immediately after a set of standards is analyzed and continuously throughout an analytical run, every 44 samples, and at the end of an analysis to verify that the instrument or method has not drifted or changed since calibration more than 10%. The initial calibration verification and continuing calibration verification will be matched to the generated standard curve and screened for acceptability. If the values are not acceptable, the samples within the 44 samples not passing, will be re-analyzed. Laboratory equipment and devices needing calibration and recalibration are numerous and varied. All equipment will have verifiable calibration documentation maintained and available for inspection in the laboratory. Laboratory standards will be checked to verify that the concentrations are those which are prescribed for the analytical method.

All instruments or devices used in obtaining data will be calibrated prior to use. Each instrument has a specialized procedure for calibration and a specific type of standard used to verify calibration. All calibration procedures will meet the requirements specified in the TCE SWFTL SOP's common to soil testing laboratories. The frequency of calibration recommended by the equipment manufacturer, as well as any instructions specified by applicable analytical methods, will be followed. All information concerning calibration will be recorded by the person performing the calibration and will be accessible for verification during either a laboratory or field audit.

All calibration procedures used in the field or laboratory will meet or exceed the calibration frequencies published in the test methods used for this project. Additional calibration procedures may be conducted if laboratory personnel determine additional calibration is warranted as beneficial to this project. Instruments and laboratory equipment used in the analyses of these samples are listed in Table B4-1 in Section B-4 "Analytical Methods Requirements." All instruments that require calibration prior to use will be calibrated before each day's analyses. Calibration is normally performed with a 4

point standard curve. The analytical balance for TSS requires no calibration other than class "S" weights to check the balance. The electronic balances used in the field to measure runoff water volume will be calibrated with these same weights.

Section B8: Inspection/Acceptance Requirements for Supplies and Consumables

All supplies and consumables received by the TCE laboratory are inspected upon receipt for damage, missing parts, expiration date, and storage and handling requirements. Labels on reagents, chemicals, and standards are examined to ensure they are of appropriate quality, the packing slip is initialed by staff member and marked with receipt date. Volumetric glassware is inspected to ensure class "A" classification, where required.

Research lab glassware and high density polyethylene containers used for chemical analyses and to obtain water samples are cleaned in soapy water, rinsed in tap water and 1N HCl, and then rinsed at least three times in deionized water with conductivity of less than 2 microsiemens per centimeter. No phosphate-based detergents are used in the cleaning process. The hydrochloric acid is used only once and is rinsed down the drain after neutralization or dilution with the tap water. Glassware is never rinsed with compounds of the constituent being analyzed.

Section B9: Data Acquisition Requirements (Non-direct Measurements)

Soil, water and litter quality determinations will be based upon data collected during the time frame of this project. These data, in conjunction with the PI evaluation of each site, will be used to validate or make recommended amendments to the PI to better predict the potential for P runoff. We will work closely with TCE County Extension Agents, NRCS, and TSSWCB to select appropriate sites and classify soils on a series basis.

Section B10: Data Management

Field Collection and Management of Routine Samples

Once sites are selected, rainfall simulations will be scheduled with individual land owners. The plot frames will be installed and the plot area pre-wet the day before the actual simulations. Water will be obtained from the closest municipality from a water hydrant if at all possible to decrease the amount of time required to fill the 1,100 gallon tank. This water is not treated for the pre-wetting and is passed through the water treatment columns for rainfall simulations. Rainfall simulators will be calibrated daily for flowrate of 7.5 cm/hr. One rainfall simulation will be conducted on each of four plots at each site. Runoff water will be collected during each of the simulations and soil samples following the simulation. These samples will be collected in specified containers (Table B3.1), stored according to protocol (Table B3.1), and analyzed according to specified parameters (Table B4.1). It will take approximately two days at each site. A field data sheet and COC form will be completed at each site as shown in Appendix B.

The pH and EC will be measured on the water samples in the field prior to acidification and placing the samples in the ice chest. Each soil, water, and if available, litter sample will be given a unique sample number and the sample container labeled by two different methods to assure sample identification. Sample ID numbers are recorded on the COC forms. Samples will be transported to the laboratory as soon as the field sampling crew returns. This may be as soon as one day and as long as one week. Samples will be stored according to protocol during transportation. When samples enter the TCE SWFTL, a unique lab number will be assigned. This number will be carried through the lab and reunited with the field number when the report is generated.

Sample containers being processed are typically placed in order of sample number, so the order of the sample containers matches the order of the field data and the COC sample ID numbers, reducing transcription errors. Sample number, comments, and other pertinent data are copied from the field data sheets to the COC. The COC and accompanying sample containers are submitted to the lab, with relinquishing and receiving personnel both signing and dating the COC.

Chain of Custody Forms

A chain of custody (COC) form is used to record sample identification parameters and to document the submission of samples from the field crew to the Soil, Water and Forage Testing Laboratory staff. Each COC has space to record data for numerous samples. A copy of the COC is found in Appendix B. All entries onto the COC forms will be completed in ink, with any changes made by crossing out the original entry, which should still be legible, and initialing and dating the new entry. COCs are kept in three-ring binders in the TCE office for at least five years.

Laboratory Analyses and Data Collection

A number code is selected for each sample on either the Soil Information Form, Water Information Form, or Biosolids Information Form (Appendix B). The number code, which is marked on the information form by the field staff, tells the laboratory staff which analytes to measure. All lab analyses are stored in computers and pulled together after analyses to form one report. Copies of all sample reports will be kept in a folder in the project manager's office.

Soil, Water and Litter Quality Data Entry

The composite data report in Excel will be transmitted to the project manager, the QA officer and the undergraduate student. This will allow data to be statistically analyzed without having to input data into a spread sheet and reduce the potential for error. The undergraduate student will be responsible for verifying that data in the Excel data entry table match the data in the research laboratory notebooks. After verification has been completed on all data for a group of samples, the laboratory manager will notify the graduate student that a group of data is ready for review. The QA officer will check for abnormalities or problems by examining all sample data, that is, COC, field, and laboratory data for a sample. Site names, appropriateness of data values, completeness of data, sample container numbers, comments and all other data will be reviewed within the Excel data table. Any questions or abnormalities will be investigated, relying largely on field data and general maintenance sheets, field technicians, laboratory QA/QC sheets, and laboratory personnel. As appropriate, corrections will be made to the Excel data table with appropriate documentation maintained.

Systems Design

TCE uses laptop personal computers and desktop personal computers. The computers run Windows operating system, Microsoft® Excel, and a SAS database management system. Currently, the Soil, Water and Forage Testing Laboratory collects data using a variety of automated instrumentation.

Backup and Disaster Recovery

Once the data is generated in the TCE SWFTL and research lab, results will be stored on three different computers and a hard copy. If all three of these computers fail, samples will be re-entered from the hard copy. Upon arrival in the office the field data will be transferred to the graduate student's computer and transmitted to the program manager's and QA officer's computers and on a backup portable hard drive. All data transferred from the lab will be saved on these same three computers, disks, and the hard copies.

Archives and Data Retention

Original data recorded on paper files are stored for at least five years by the project manager. Data in electronic format are stored on hard drives in computers and on either floppy, zip or CD disks.

Information Dissemination

Pertinent TCE data will be sent to TSSWCB. Data will be evaluated and discussed with TSSWCB and NRCS to make decisions on how the PI will be revised if deemed necessary. Based upon the soil sampling evaluation, the variability of each of the three techniques will be given relative to the comprehensive 0.25 A grid sampling. The final version of the PI and soil sampling evaluation will be disseminated to all Texas Certified Nutrient Management Specialist, TCEQ CAFO personnel, NRCS, CAFO industry personnel, Region VI EPA and placed on the nutrient management web site, <http://nmp.tamu.edu>. The information will also be disseminated through multi-county meetings in the poultry areas of Texas, Soil and Crop Sciences publications, and appropriate scientific journals.

Section C1: Assessments and Response Actions

The commitment to use standard equipment and standard methods for soil, water and litter samples and when producing field or laboratory measurements requires periodic verification that the equipment and methods are being employed properly. This verification will be provided through an annual field and laboratory performance audit performed by TCE QA officer. Individual field personnel will be observed during the actual field investigation to verify that equipment and procedures are properly applied. Any problems that are discovered in the monitoring procedures that would affect the quality of data collected at the demonstration sites will be addressed by the project participants and followed up with a CAR. Follow-up observations will occur within three months when discrepancies are noted. Also, TSSWCB and EPA may conduct a performance audit for this project.

Depending on the analysis, certain methodologies require that standards and reagent blanks be analyzed to verify that no instrument or chemical problem will affect the quality of the data. The specific requirements are presented in Section B5 of the QAPP.

To minimize downtime of all measurement systems, all field equipment and all laboratory equipment must be maintained in a working condition. Also, backup equipment or common spare parts will be available if any piece of equipment fails during use so that repairs or replacement can be made quickly and the sample tasks resumed.

Section C2: Reports to Management

Quarterly progress reports will note activities conducted in connection with the PI evaluation program, items or areas identified as potential problems, and any variations or supplements to the QAPP. CAR forms will be utilized when necessary (Appendix A). CARs will be maintained in an accessible location for reference at TCE. CARs that result in any changes or variations from the QAPP will be made known to pertinent project personnel and documented in an update or amendment to the QAPP.

The field measurement and sampling for the project will be done according to the QAPP. However, if the procedures and guidelines established in this QAPP are not successful, corrective action is required to ensure that conditions adverse to quality data are identified promptly and corrected as soon as possible. Corrective actions include identification of root causes of problems and successful correction of identified problem. CARs will be filled out to document the problems and the remedial action taken. Copies of CARs are included with TCE's annual Quality Assurance reports. The quality control reports will contain a quality assurance section to address TCE's accuracy, precision and completeness of the measurement data. They will also discuss any problems encountered and solutions made. These QA reports are the responsibility of the project manager, QA officer and the laboratory manager.

Section D1: Data Review, Validation and Verification

All data obtained from field and laboratory measurements will be reviewed and verified for integrity and continuity, reasonableness, and conformance to project requirements, and then validated against the data quality objects outlined in Section A7, “Data Quality Objectives for Measurement Data” by the project manager. Only those data that are supported by appropriate quality control data and meet the DQOs defined for this project will be considered acceptable for use.

The procedures for verification and validation of data are described in Section D2, below. The Field Supervisor is responsible for ensuring that field data are properly reviewed, verified, and submitted in the required format for the project database. Likewise, the Laboratory Manager is responsible for ensuring that laboratory data are reviewed, verified, and submitted in the EXCEL format for the project database. The project manager is responsible for validating that all data collected meet the data quality objectives of the project.

Section D2: Validation and Verification Methods

Quality control aspects of databases include the following:

- Sample data are identified with a unique, sequential sample number, which is documented on COC forms, field information forms (soil, water and biosolids) and all research laboratory logbooks. A standard operating procedure has been written for sample custody and control to ensure proper identification and analyses of all samples.
- TCE laboratory logbooks are reviewed by the Laboratory Manager for passage of quality control parameters before the data are entered into EXCEL database. This constitutes an ongoing internal audit.
- Data are entered into the TCE soil, water and litter database only after quality control parameters have been verified. Data are reviewed to ensure a complete record for each sample.
- Entries into the Excel data entry table are verified against COCs, field data sheets and research laboratory notebooks prior to transfer into the Excel quality databases. This constitutes an on-going internal audit.
- Databases are scanned for outliers by graphical presentation of the data by the graduate student and anomalies are investigated for errors in data entry and/or transfer. Correctly entered values that appear to be outliers are statistically analyzed for outliers.
- All extreme outliers will be verified by review of the field data sheets or research laboratory notebooks to make sure these points are not transcription errors. If an error is found, the project manager will be notified with the appropriate documentation of the change that is needed in the Excel and SAS databases.
- CARs are completed for each soil, water and litter quality analysis discrepancy or for missing information that cannot be immediately resolved. These reports serve as checks for subsequent queries concerning the database.
- Unusual circumstances associated with sampling sites or collection of samples are noted in the Comments section of the field notebooks.
- Entries in soil, water and litter quality databases are verified by the person who enters the data.
- Print-outs of electronically generated data are archived for subsequent verification of data by the graduate student and project manager. All materials are archived for five years.
- Mistakes in COCs and logbooks are crossed out with a single line, corrected, initialed and dated by the person correcting it. This ensures proper lines of communications concerning queries of data validity.
- Quality assurance field duplicate samples are marked as such in the comments section of the COC, along with the sample number duplicated. Duplicate parameter data should not be used in data analysis because it doubles the influence of the duplicated sample on the data set. To avoid this, duplicate data are split into a separate SAS database from the

general sample quality database when data are transferred from the Excel soil, water and litter quality database.

- The COC is filled out by the field person bringing in the samples to eliminate misunderstandings in data transfer from the field data sheet to the COC.
- Samples are transferred to the laboratory immediately to avoid violating holding times.
- Sample numbers are assigned at the time that they are used to prevent more than one sample from having the same sample number.

Microsoft Excel will be used for general spreadsheet computation and laboratory control charting of quality control parameters. The TCE SWFTL will employ various data handling software on IBM compatible personal computer stations for data on many of the analyzed parameters. Specific software and/or hardware handle data for the different instruments. The TCE SWFTL laboratory manager is responsible for review of calculations made by these programs. Soil, water and litter quality statistical analyses are performed with SAS programs by the graduate student.

Section D3: Reconciliation with Data Quality Objectives

The laboratory manager shall be responsible for reviewing raw data produced by the TCE laboratory. The laboratory manager shall check calculations to verify that data are entered into the database correctly and be responsible for internal lab error corrections. CARs will be initiated in cases where invalid or incorrect data have been detected.

Data completeness in this project will be relative to accidents in handling, shipping and laboratory analysis for completeness of the sampling program. It will be the goal of this project to achieve 90 percent completeness; however, statistical analysis will be the final indicator of data validity.

Representativeness and comparability of data, while unique to each individual collection site, is the responsibility of the project manager. By following the guidelines described in this QAPP, and through careful sampling design, the data collected in this project will be representative of the actual field conditions and comparable to similar applications. Representativeness and comparability of laboratory analyses will be the responsibility of the laboratory manager.

The project manager will review the final data to ensure that it meets the requirements as described in this QAPP.

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Appendix A
Corrective Action Report Form

Corrective Action Report
CAR #: _____

Date: _____ Area/Location: _____

Reported by: _____ Activity: _____

State the nature of the problem, nonconformance or out-of-control situation:

Possible causes:

Recommended Corrective Actions:

CAR routed to: _____

Received by: _____

Corrective Actions taken:

Has problem been corrected?: YES NO

Immediate Supervisor: _____

Program Manager: _____

Quality Assurance Officer: _____

Appendix B

Chain-of-Custody Form

Laboratory Data Entry Sheet

Field Data Entry Sheet

Field Sample Sheet and Chain of Custody

| | | | | | |
|--|-------|------|----------------|-------|--|
| Samples for Dr. Feagley / Todd Carpenter | | | | | |
| Samples Collected By: | | | | Date: | |
| Samples Processed By: | | | | Date: | |
| Samples Submitted By: | | | | Date: | |
| Received By: | | | | Date: | |
| Returned By: | | | | Date: | |
| Received: | | | | Date: | |
| Sample Type: | Water | | | | |
| Lab ID | Site | Plot | Test Requested | | |
| | | 1 | | | |
| | | 2 | | | |
| | | 3 | | | |
| | | 4 | | | |
| | | 1 | | | |
| | | 2 | | | |
| | | 3 | | | |
| | | 4 | | | |
| | | 1 | | | |
| | | 2 | | | |
| | | 3 | | | |
| | | 4 | | | |
| | | 1 | | | |
| | | 2 | | | |
| | | 3 | | | |
| | | 4 | | | |
| | | 1 | | | |
| | | 2 | | | |
| | | 3 | | | |
| | | 4 | | | |
| | | 1 | | | |
| | | 2 | | | |
| | | 3 | | | |

| | | | | | |
|--|--|---|--|--|--|
| | | 4 | | | |
|--|--|---|--|--|--|



SOIL SAMPLE INFORMATION FORM

D-494

TEXAS AGRICULTURAL EXTENSION SERVICE
 THE TEXAS A&M UNIVERSITY SYSTEM

Soil, Water and Forage Testing Laboratory

Please submit this completed form and payment with samples. Mark each soil sample bag with your sample identification and ensure that it corresponds with the sample identification written on this form. See sampling procedures and mailing instructions on the back of this form. **(PLEASE DO NOT SEND CASH)**

SUBMITTED BY: Results will be mailed to this address **ONLY**

Name _____ County where sampled _____
 Address _____ Phone _____
 City _____ State _____ Zip _____

FOR: (Optional-will not receive copy)

Name _____
 Address _____
 City _____ State _____ Zip _____

Payment (DO NOT SEND CASH).
 Check
 Money Order
 Government Account

Amount Paid \$ _____
 Make Checks Payable to: Soil Testing Laboratory

| Laboratory # (For Lab Use) | CROP/PLANT INFORMATION (Required for Recommendations) | | | | Requested Analyses (See Options Listed Below) | | How is Forage Used? | | | | | | |
|-------------------------------|---|---------------------|-----------------------------|----------------------------------|--|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| | Your Sample I.D. | Acreage Represented | Previous Lime or Fertilizer | What Are You Growing-Yield Goal? | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |

Describe any specific problems you have observed or want to correct:

- Choose one analysis group per sample
- 1. Routine Analysis (R) (pH, NO₃⁻, P, K, Ca, Mg, Na, S, and Conductivity) **\$10 per sample**
 - 2. R + Micronutrients (Micro) (Zn, Fe, Cu, Mn) **\$15 per sample**
 - 3. R + Micro + Boron (B) **\$20 per sample**
 - 4. R + Detailed Salinity (Sal) **\$25 per sample**
 - 5. R + Micro + Sal **\$30 per sample**
 - 6. R + Micro + Detailed Lime Requirement (Lime) **\$20 per sample**

- 7. R + Micro + B + Lime + Organic Matter + Sal **\$50 per sample**
 - 8. R + Texture Analysis **\$20 per sample**
 - 9. R + Organic Matter **\$20 per sample**
- Note: Organic Matter, Detailed Salinity and Texture may require longer processing time.
- *Minimum requirement for establishment

Procedure for Taking Soil Samples

Taking the Soil Sample (Refer to Figure 1)

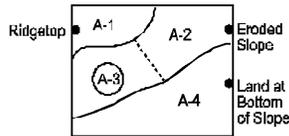


Fig. 1

- Take one composite sample for every 10 to 40 acres. A separate sample should be taken for:
 - ⇒ Areas with different soil types
 - ⇒ Areas with different land uses or fertilizer uses
 - ⇒ Areas with different terrain
- Approximately 1 pint of the composite soil sample is required for routine analyses.
- Additional sample is required for texture or detailed salinity (submit 2 sample bags marked identically).
- Avoid sampling areas such as small gullies, slight field depressions, terrace waterways, or unusual areas.
- When sampling fertilized fields, avoid sampling directly in fertilized band.

Taking a Composite Sample (Refer to Figures 2 and 3)

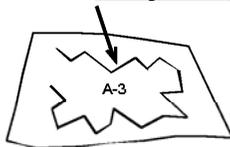


Fig. 2



Fig. 3

- Take a sample from 10 to 15 different areas.
- Use a spade, soil auger or soil sampling tube.
- Clear litter from the surface (do not remove decomposed black material).
- When using a soil auger or sampling tool, make the core or boring 6 inches deep into the soil (3 to 4 inches deep for permanent sod).
- When using a spade:
 - ⇒ Dig a V-shaped hole and take a 1-inch slice from the smooth side of the hole.
 - ⇒ Take a 1 x 1 inch core from the center of the shovel slice.
- Repeat in 10 to 15 different places. Put in a clean plastic bucket or other non-metallic container, thoroughly mix and remove a pint (or more if additional tests are desired) as a composite sample representing the whole field or area.
- To improve the nitrate-nitrogen analysis, samples may be **air-dried** before sending to the laboratory. **Do not use heat** to dry samples. Completely fill soil sample bag or other suitable pint container. Do not use old vegetable cans, tobacco cans, match boxes, glass containers, etc. to submit samples. If more than one sample bag is used, label bags as 1 of 2, 2 of 2,

Shipping the Sample and Payment (Refer to Figure 4)

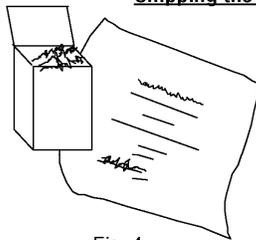


Fig. 4

- Complete the information form on the front page (information required for recommendations).
- Please include payment with the sample. Send check or money order made out to Soil Testing Laboratory. **DO NOT SEND CASH.** Please note that the **price is per sample**.
- Be sure to keep a record for yourself of the area represented by each sample.
- Be sure that sample numbers on sample bags correspond with sample numbers on the front page.
- Send samples and payment to:

**Soil, Water and Forage Testing Laboratory
 2474 TAMU
 College Station, TX 77843-2474**

**For further information please contact:
 Your local County Extension Service Office
 or**

**Soil, Water and Forage Testing Laboratory
 2474 TAMU
 345 Heep Center
 College Station, TX 77843-2474
 Phone: (979) 845-4816**



WATER SAMPLE INFORMATION FORM

D-617A

TEXAS AGRICULTURAL EXTENSION SERVICE

THE TEXAS A&M UNIVERSITY SYSTEM

Soil, Water and Forage Testing Laboratory

Please submit this completed form and payment with samples. Mark each sample bottle with your sample identification and ensure that it corresponds with the sample identification written on this form. See sampling and mailing instructions on the back of this form.

(PLEASE DO NOT SEND CASH)

SUBMITTED BY:

Results will be mailed to this address **ONLY**

Name _____ County where sampled _____
Address _____ Phone _____
City _____ State _____ Zip _____

FOR:

(Optional-will not receive copy)

Name _____
Address _____
City _____ State _____ Zip _____

Payment (DO NOT SEND CASH).

- Check
- Money Order
- Government Account

Amount Paid \$ _____

Make Checks Payable to: Soil Testing Laboratory

| SAMPLE INFORMATION | | | (Required for Evaluation/Recommendations) | | | | Requested Analyses |
|-------------------------------|------------------|--|---|--|--|----------------------------|--------------------|
| Laboratory # (For Lab Use) | Your Sample I.D. | Source of Water: | Water Use: | | | (See options listed below) | |
| | | <input type="checkbox"/> Public <input type="checkbox"/> Well <input type="checkbox"/> Wastewater treatment <input type="checkbox"/> Private <input type="checkbox"/> Pond <input type="checkbox"/> Other <input type="checkbox"/> Lake <input type="checkbox"/> Stream <input type="checkbox"/> Processing plant <input type="checkbox"/> Animal feedlot | <input type="checkbox"/> Aquaculture <input type="checkbox"/> Commercial <input type="checkbox"/> Domestic <input type="checkbox"/> Greenhouse <input type="checkbox"/> Hydroponics <input type="checkbox"/> Irrigation-forages <input type="checkbox"/> Irrigation-ornamentals | <input type="checkbox"/> Irrigation-turf <input type="checkbox"/> Irrigation-vegetables <input type="checkbox"/> Livestock <input type="checkbox"/> Recreation <input type="checkbox"/> Wastewater <input type="checkbox"/> Other | <input type="checkbox"/> 1 <input type="checkbox"/> 2 <input type="checkbox"/> 3 <input type="checkbox"/> 4 <input type="checkbox"/> 5 <input type="checkbox"/> 6 <input type="checkbox"/> 7 | | |
| | | <input type="checkbox"/> Public <input type="checkbox"/> Well <input type="checkbox"/> Wastewater treatment <input type="checkbox"/> Private <input type="checkbox"/> Pond <input type="checkbox"/> Other <input type="checkbox"/> Lake <input type="checkbox"/> Stream <input type="checkbox"/> Processing plant <input type="checkbox"/> Animal feedlot | <input type="checkbox"/> Aquaculture <input type="checkbox"/> Commercial <input type="checkbox"/> Domestic <input type="checkbox"/> Greenhouse <input type="checkbox"/> Hydroponics <input type="checkbox"/> Irrigation-forages <input type="checkbox"/> Irrigation-ornamentals | <input type="checkbox"/> Irrigation-turf <input type="checkbox"/> Irrigation-vegetables <input type="checkbox"/> Livestock <input type="checkbox"/> Recreation <input type="checkbox"/> Wastewater <input type="checkbox"/> Other | <input type="checkbox"/> 1 <input type="checkbox"/> 2 <input type="checkbox"/> 3 <input type="checkbox"/> 4 <input type="checkbox"/> 5 <input type="checkbox"/> 6 <input type="checkbox"/> 7 | | |
| | | <input type="checkbox"/> Public <input type="checkbox"/> Well <input type="checkbox"/> Wastewater treatment <input type="checkbox"/> Private <input type="checkbox"/> Pond <input type="checkbox"/> Other <input type="checkbox"/> Lake <input type="checkbox"/> Stream <input type="checkbox"/> Processing plant <input type="checkbox"/> Animal feedlot | <input type="checkbox"/> Aquaculture <input type="checkbox"/> Commercial <input type="checkbox"/> Domestic <input type="checkbox"/> Greenhouse <input type="checkbox"/> Hydroponics <input type="checkbox"/> Irrigation-forages <input type="checkbox"/> Irrigation-ornamentals | <input type="checkbox"/> Irrigation-turf <input type="checkbox"/> Irrigation-vegetables <input type="checkbox"/> Livestock <input type="checkbox"/> Recreation <input type="checkbox"/> Wastewater <input type="checkbox"/> Other | <input type="checkbox"/> 1 <input type="checkbox"/> 2 <input type="checkbox"/> 3 <input type="checkbox"/> 4 <input type="checkbox"/> 5 <input type="checkbox"/> 6 <input type="checkbox"/> 7 | | |

Describe any specific problems you have observed or want to correct:

| | |
|--|------------------------|
| 1. Routine Analysis (R) (Conductivity, pH, Na, Ca, Mg, K, CO ₃ ²⁻ , HCO ₃ ⁻ , SO ₄ ²⁻ , Cl, B, Nitrate-N, Hardness, and SAR) | \$20 per sample |
| 2. R + Metals In addition to Routine Analysis includes: (Zn, Fe, Cu, Mn, and P) | \$30 per sample |
| 3. R + Metals + Heavy Metals (Heavy) + Fluoride In addition to Routine and Metal analyses includes: (As, Ba, Ni, Cd, Pb, Cr, and Fluoride) | \$50 per sample |

| | |
|--|------------------------|
| 4. R + Titrate for Drip Irrigation | \$25 per sample |
| 5. R + Metals + Titrate for Drip Irrigation | \$35 per sample |
| 6. R + Metals + Heavy + Fluoride + Titrate for Drip Irrigation | \$55 per sample |
| 7. Animal Waste Water (fertility analysis) (Total N, P, K, Ca, Mg, Na, Zn, Mn, Fe, and Cu) | \$20 per sample |

How To Take A Water Sample

Water analyses can only be accurate if the sample is taken correctly. When collecting a water sample, please follow these simple guidelines:

CONTAINERS

Samples should be collected in a new clean, plastic bottle with a screw cap. A new eight-ounce plastic, disposable baby bottle is highly recommended. Please note that the lab does not test for bacteria, pesticides, or petrochemicals. Clearly identify each contain with a simple sample I.D. match those use on the front side of this form. When mailing, place bottles in a box and pack with a loose, soft packing material to prevent crushing. Avoid glass containers, as boron concentrations may change and glass has higher potential for breakage.

AQUACULTURE

Provide as much information as possible about the condition of the pond. If fresh water is running into the pond, collect the sample in the area of the pond least affected by the fresh water. When samples are taken from salt-water ponds where fresh water may have been added, gather water from both the top and bottom of the pond. The lab cannot test for dissolved oxygen, free carbon dioxide, or hydrogen sulfide, even though these criteria all affect fish mortality. These substances must be tested for on-site, and kits for conducting these tests are commercially available.

WELL WATER

Let the pump operate ten minutes to an hour before taking the sample. Take the sample from water at the pump.

ASSESSING PROBLEM WATERS

Two separate water samples may be required to address water related problems due to plumbing and/or fixtures. One sample should be collected at the point of entry (well or water service) and another at point of use (faucet, pool and etc.). This sampling method will help pinpoint problematic plumbing.

LIVESTOCK

Collect samples from the specific area of the trough or pond where the water was consumed. Place these samples in a clean plastic container. In the event of sick or dead livestock, samples should be sent to the Texas Veterinary Medical Diagnostic Laboratory (409) 845-3414.

ANIMAL WASTE WATER

This analysis involves digestion of the wastewater and is primarily designed to address potential fertilizer value of the material. Samples submitted for this analysis should have at least 30 percent headspace volume in the sample bottle.

Please enclose the information form and payment for each sample inside the box with the samples.

Extension Soil, Water and Forage

Testing Laboratory

Texas A&M University
2474 TAMU
College Station, Texas 77843-2474
(979) 845-4816

**** NOTICE:** Water samples will be tested for the salts commonly found in water. Interpretations will be given only for suitability for irrigation and consumption by livestock but not for human consumption. Our laboratory does NOT analyze for or organic compounds such as pesticides or petrochemicals.

Educational programs conducted by the Texas Agricultural Extension Service serve people of all ages regardless of socioeconomic level, race, color, sex, religion, handicap, or origin.

Issued in furtherance of Cooperative Extension Work in Agricultural and Home Economics, Acts of Congress of May 8, 1914, as amended, and June 30, 1914, in cooperation with the United States Department of Agriculture. The Director, Texas Extension Service, The Texas A&M University System.



BIOSOLID SAMPLE INFORMATION FORM
MANURE, LITTER, AND EFFLUENTS
TEXAS AGRICULTURAL EXTENSION SERVICE
THE TEXAS A&M UNIVERSITY SYSTEM
 Soil, Water and Forage Testing Laboratory

Please submit this completed form with samples. Mark each sample bag with your sample identification and ensure that it corresponds with the sample identification written on this form. See sampling procedures and mailing instructions on the back of this form.

SUBMITTED BY:

Name _____ County where sampled _____
 Address _____ Phone _____
 City _____ State _____ Zip _____

FOR: (Will not receive copy)

Name _____
 Address _____
 City _____ State TX Zip _____

Payment (DO NOT SEND CASH).

- Check
- Money Order
- Government Account

Amount Paid \$ _____

| SAMPLE I.D. | | SAMPLE INFORMATION (Required) | | | (See options listed below) |
|-------------------------------|------------------|-------------------------------|---|--|---|
| Laboratory # (For Lab Use) | Your Sample I.D. | Quantity Represented | Biosolid to be applied to: <small>(cultivated crop land, non cultivated crop land, or other)</small> | Sample Type: <small>Please check box</small> | Requested Analyses |
| | | | | <input type="checkbox"/> solid manure <input type="checkbox"/> litter <input type="checkbox"/> liquid-effluent <input type="checkbox"/> other _____ | <input type="checkbox"/> 1 manure/litter <input type="checkbox"/> 2 effluent and other |
| | | | | <input type="checkbox"/> solid manure <input type="checkbox"/> litter <input type="checkbox"/> liquid-effluent <input type="checkbox"/> other _____ | <input type="checkbox"/> 1 manure/litter <input type="checkbox"/> 2 effluent and other |
| | | | | <input type="checkbox"/> solid manure <input type="checkbox"/> litter <input type="checkbox"/> liquid-effluent <input type="checkbox"/> other _____ | <input type="checkbox"/> 1 manure/litter <input type="checkbox"/> 2 effluent and other |
| | | | | <input type="checkbox"/> solid manure <input type="checkbox"/> litter <input type="checkbox"/> liquid-effluent <input type="checkbox"/> other _____ | <input type="checkbox"/> 1 manure/litter <input type="checkbox"/> 2 effluent and other |

Please select one analysis group per sample:

Analysis for Biosolids

1. Nitrogen + Minerals + % Moisture **\$15 per sample**
(N, P, K, Ca, Mg, Na, Zn, Fe, Cu, Mn and % moisture)
 (for solid manures only)

2. Nitrogen + Minerals **\$15 per sample**
(N, P, K, Ca, Mg, Na, Zn, Fe, Cu, and Mn.
 (for effluents and other misc. samples)

Please follow all sampling and shipping instructions on back of of this form.

-----Available Services-----

Analyses conducted on biosolids (i.e., litter, composts or manure samples) are intended to provide accurate data for determining application rates and nutrient loading. *All samples are analyzed with the understanding that the results are not in any way associated with environmental control regulations.*

| |
|--------------------------|
| Sample Collection |
|--------------------------|

Manure and litter samples

- Collect at least 5 and preferably 10 subsamples from piles. Be sure to sample throughout the pile, not just the outside surface.
- Mix subsamples thoroughly in clean plastic bucket.
- Transfer sample to suitable container (see below).
- Label sample container using a permanent marker.
- Separate samples should be taken for each type or age of manure and litter.

Effluent samples

- Collect at least 5, and preferably 10 subsamples from the lagoon.
- Sample the lagoon using a plastic cup (8 ounce) secured to an aluminum rod (6 to 10 feet long).
- Samples collected with depth will better represent effluent.
- Collect subsamples and mix in clean plastic bucket.
- Transfer sample to suitable container (see below).
- Label sample container using a permanent marker.
- Separate samples should be taken for each lagoon.

Sample containers

- Biosolids, manure and litter samples should be collected in sealable plastic bags.
- Liquid samples (i.e., lagoon or effluent samples) should be collected in plastic bottles (16 ounce) with at least 50% headspace. Failure to provide adequate headspace may result in rupture of container.
- Do not use cola bottles or other containers containing phosphorus or nutrients to be analyzed.

Shipping Samples

- Complete this information form.
- Enclose completed information form and payment in package.
- Verify payment check is made out to **Soil Testing Laboratory**.
- **DO NOT SEND CASH.**
- Address the letter and package to the following address:

**Extension Soil, Water and Forage Testing Laboratory
Texas A&M University
Soil and Crop Sciences
College Station, TX 77843-2474
(979) 845-4816**

For further information please contact your local County Extension Agent

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Appendix C
General Maintenance Sheet

General Maintenance

Soil Sampling:

- Tools: Sharp shooters, shovels, augers, soil sampling tubes, plastic bucket—cleaned after each site sampled.
- Maintain soil sample bag supply.
- Maintain soil sample labeling supplies.

Water Sampling:

- Clean plastic bottles.
- Maintain water sample labeling supplies.
- Maintain pH and EC field meter.

Rainfall Simulator:

- Check all valves.
- Maintain gasoline supply.
- Check water treatment columns.
- Check conductivity meter on water treatment columns.
- Check and maintain trailer.
- Maintain generator.
- Maintain balances.
- Clean water containers after each rainfall simulation.
- Maintain plot frames.