

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

***Demonstration and Transfer of Selected New Technologies for  
Animal Waste Pollution Control***  
**TSSWCB Project FY03-10**

**Quality Assurance Project Plan**

**Texas State Soil and Water Conservation Board**

prepared by

Texas Water Resources Institute  
Texas A&M University - Biological and Agricultural Engineering Department  
Texas A&M University – Department of Soil and Crop Sciences

Effective Period: November 2003 to April 2008  
5<sup>th</sup> Revision

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

Dr. Saqib Mukhtar; Associate Professor, Animal Waste Management  
Texas A&M University  
Biological and Agricultural Engineering Department  
207-A Scoates Hall  
2117 TAMU  
College Station, TX 77843-2117  
[mukhtar@tamu.edu](mailto:mukhtar@tamu.edu)  
(979) 458-1019

## **Section A1 Approval Sheet**

*Demonstration and Transfer of Selected New Technologies for Animal Waste Pollution Control*

### **United States Environmental Protection Agency (USEPA), Region VI**

Name: Donna Miller

Title: USEPA Chief State/Tribal Programs Section

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Name: Ellen Caldwell

Title: USEPA Texas Nonpoint Source Project Manager

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

### **Texas State Soil and Water Conservation Board (TSSWCB)**

Name: Thomas J. Helton

Title: TSSWCB Project Leader

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Name: Donna Long

Title: TSSWCB Quality Assurance Officer

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

### **Texas Water Resources Institute-Texas A&M University (TWRI)**

Name: C. Allan Jones, Ph.D.

Title: TWRI Director; Project Coordinator

Signature: \_\_\_\_\_ Date: \_\_\_\_\_

**Section A1 Approval Sheet**

*Demonstration and Transfer of Selected New Technologies for Animal Waste Pollution Control*

**United States Environmental Protection Agency (USEPA), Region VI**

Name: Donna Miller

Title: USEPA Chief State/Tribal Programs Section

Signature: Donna R. Miller Date: 7-17-07

Name: Ellen Caldwell

Title: USEPA Texas Nonpoint Source Project Manager

Signature: Ellen Caldwell Date: 7-17-07

**Texas State Soil and Water Conservation Board (TSSWCB)**

Name: Thomas J. Helton

Title: TSSWCB Project Leader

Signature: Tom Helton Date: 06/12/07

Name: Donna Long

Title: TSSWCB Quality Assurance Officer

Signature: Donna K. Long Date: 06/12/07

**Texas Water Resources Institute-Texas A&M University (TWRI)**

Name: C. Allan Jones, Ph.D.

Title: TWRI Director; Project Coordinator

Signature: Charles Allan Jones Date: 08/09/07



1870

1870

1870

1870

1870

1870

1870

1870

1870

1870

Name: Kevin Wagner  
Title: TWRI Quality Assurance Officer

Signature: Kevin Wagner Date: 5-9-07

**Texas A&M University-Biological and Agricultural Engineering Department (TAMU)**

Name: Saqib Mukhtar, Ph.D.  
Title: Associate Professor, Animal Waste Management; Project Leader

Signature: Saqib Mukhtar Date: 5-18-07

Name: Clyde Munster, Ph.D., P.E.  
Title: Professor, Environmental Soil and Water Engineering

Signature: Clyde Munster Date: 5-17-07

**Texas A&M University-Department of Soil and Crop Sciences (TAMU)**

Name: Donald Vietor, Ph.D.  
Title: Professor, Agronomy

Signature: Donald M. Vietor Date: 5-17-2007

**Texas Institute for Applied Environmental Research (TIAER)**

Name: Nancy Easterling  
Title: TIAER Quality Assurance Officer

Signature: Nancy Easterling Date: 31 May 07

Name: Mark Murphy  
Title: TIAER Laboratory Manager

Signature: Mark Murphy Date: 5/29/07

6

7

100

100

100

100

100

100

100

100

100

100

100

100

100

**TCE Soil, Water and Forages Testing Laboratory (SWFTL)**

Name: Tony Provin, Ph.D.

Title: TCE-SWFTL Laboratory Director

Signature: \_\_\_\_\_

Date: \_\_\_\_\_

5/7/07

Name: John Pitt

Title: TCE-SWFTL Laboratory Manager & Quality Assurance Officer

Signature: \_\_\_\_\_

Date: \_\_\_\_\_

5/17/07

0

d

!

**Section A2: Table of Contents**

<b>Section: Title</b>	<b>Page</b>
A1 Approval Sheet .....	2
A2 Table of Contents .....	5
List of Acronyms .....	7
A3 Distribution List .....	9
A4 Project/Task Organization .....	11
A5 Problem Definition/Background .....	16
A6 Project/Task Description .....	18
A7 Data Quality Objectives for Measurement Data .....	24
A8 Special Training Requirements/Certification.....	31
A9 Documentation and Records.....	32
B1 Sampling Process Design (Experimental Design).....	33
B2 Sampling Methods Requirements.....	36
B3 Sample Handling and Custody Requirements.....	37
B4 Analytical Methods Requirements .....	42
B5 Quality Control Requirements.....	46
B6 Equipment Testing, Inspection, & Maintenance Requirements.....	53
B7 Instrument Calibration and Frequency .....	54
B8 Inspection/Acceptance Requirements for Supplies and Consumables.....	55
B9 Data Acquisition Requirements (Non-direct Measurements) .....	56
B10 Data Management.....	57
C1 Assessments and Response Actions .....	59
C2 Reports to Management.....	61
D1 Data Review, Validation and Verification .....	62
D2 Validation and Verification Methods.....	63
D3 Reconciliation with Data Quality Objectives.....	64
References .....	65
Appendix A Corrective Action Report Forms .....	67
Appendix B Chain of Custody Form .....	68
Appendix C Field Data Reporting Form.....	70
Appendix D Data Review Checklist .....	71
Appendix E SWFTL Methodology References.....	73
Appendix F Dr. Vietor & Schnell Methodology References .....	76

**List of Tables**

Table A6-1	Project Plan Milestones.....	23
Table A7-1	TIAER Measurement Performance Specifications .....	25
Table A7-2	Estimated SWFTL Accuracy and Precision Limits .....	26
Table A7-3	Estimated Accuracy and Precision Limits for Dr. Viotor’s Lab .....	28
Table B1-1	Experimental Variables .....	35
Table B3-1	TIAER Sample Procedures and Handling Methods .....	38
Table B3-2	SWFTL Sample Procedures and Handling Methods .....	39
Table B3-3	Drs. Viotor and Munster Sample Procedures & Handling Methods .....	40
Table B4-1	TIAER Laboratory Analytical Methods.....	42
Table B4-2	SWFTL Laboratory Analytical Methods .....	43
Table B4-3	Dr. Viotor’s Laboratory Analytical Methods .....	44
Table C1-1	Assessment and Response Actions.....	59

**List of Figures**

Figure A4-1	Project Organization Chart.....	15
Figure A6-1	Bosque River and Leon River Watersheds.....	21

## List of Acronyms and Abbreviations

ACS	American Chemical Society
AFO	animal feeding operation
ASTM	American Society for Testing and Materials
AWRL	Ambient Water Reporting Limit
BMP	best management practices
BOD	biochemical oxygen demand
BRA	Brazos River Authority
CAFO	concentrated animal feeding operation
CAR	corrective action report
CDT	capacitive deionization technology
COC	chain of custody
CWA	Clean Water Act
DQO	data quality objectives
EPA	Environmental Protection Agency
GM	general maintenance
HCl	hydrochloric acid
HDPE	high density polyethylene
HNO <sub>3</sub>	concentrated nitric acid
H <sub>2</sub> SO <sub>4</sub>	concentrated sulfuric acid
ID	identification
LCS	laboratory control standards
LCSD	laboratory control standard duplicate
MDL	method detection limit
MS	matrix spike
NIST	National Institute for Standards and Technology
NO <sub>2</sub> +NO <sub>3</sub> -N	nitrite+nitrate-nitrogen
NPS	nonpoint source
NRCS	Natural Resources Conservation Service
QA	quality assurance
QAM	quality assurance manual
QAO	quality assurance officer
QAPP	quality assurance project plan
QC	quality control
QMP	quality management plan
P	phosphorus
PAM	polyacrylamides
PD	percent deviation
pH	potential hydrogen
PM	project manager
PO <sub>4</sub> -P	orthophosphate phosphorus
RL	reporting limit
RPD	relative percent deviation
SAS	statistical analysis software
SOP	standard operating procedures
SRP	soluble reactive phosphorus
SWFTL	Soil, Water and Forages Testing Laboratory
SWQM	surface water quality monitoring
TAES	Texas Agricultural Experiment Station
TAMU	Texas A&M University
TCE	Texas Cooperative Extension

TCEQ	Texas Commission on Environmental Quality
TFB	Texas Farm Bureau
TIAER	Texas Institute for Applied Environmental Research
TKN	total kjeldahl nitrogen
TMDL	total maximum daily load
TP	total phosphorus
TSS	total suspended solids
TS	total solids
TSSWCB	Texas State Soil and Water Conservation Board
TWRI	Texas Water Resources Institute
USDA	United States Department of Agriculture
USDA-NRCS	United States Department of Agriculture Natural Resources Conservation Commission
USEPA	United States Environmental Protection Agency
VS	volatile solids
WAF	waste application field

**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**

6WQ-AT  
1445 Ross Avenue  
Suite 1200  
Dallas, Texas 75202-2733

Name: Donna Miller  
Title: USEPA Chief; Assistance Programs Branch

Name: Ellen Caldwell  
Title: USEPA Texas Nonpoint Source Project Manager

**Texas State Soil and Water Conservation Board**

P.O. Box 658  
Temple, TX 76503

Name: Donna Long  
Title: TSSWCB Quality Assurance Officer (QAO)

Name: Thomas J. Helton  
Title: TSSWCB Project Leader

**Texas Water Resources Institute**

1500 Research Parkway, Suite 240  
2118 TAMU  
College Station, TX 77843-2118

Name: C. Allan Jones, Ph.D.  
Title: TWRI Director; Project Coordinator

Name: Kevin Wagner  
Title: TWRI QAO

**Texas A&M University - Biological and Agricultural Engineering Department**

207-A Scoates Hall  
2117 TAMU  
College Station, TX 77843-2117

Name: Saqib Mukhtar, Ph.D., P.E.  
Title: Associate Professor, Animal Waste Management; Project Leader

**Texas A&M University - Biological and Agricultural Engineering Department**

127 Hobgood  
2117 TAMU  
College Station, TX 77843-2117

Name: Clyde Munster, Ph.D., P.E.  
Title: Professor, Environmental Soil and Water Engineering

**Texas A&M University – Department of Soil and Crop Sciences**

431B Heep Center  
370 Olsen Blvd.  
2474 TAMU  
College Station, TX 77843-2474

Name: Donald Vietor, Ph.D.  
Title: Professor, Crop Physiology

**Texas Institute for Applied Environmental Research**

Box T-0410  
Tarleton State University  
Stephenville, TX 76402

Name: Nancy Easterling  
Title: TIAER QAO

Name: Mark Murphy  
Title: TIAER Laboratory Manager

**TCE Soil, Water and Forages Testing Laboratory**

2474 TAMU  
College Station, TX 77843-2474

Name: Tony Provin, PhD.  
Title: TCE-SWFTL Laboratory Director

Name: John Pitt  
Title: TCE-SWFTL Laboratory Manager & QAO

#### **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, Region VI, Dallas. Provides project overview at the Federal level.

Ellen Caldwell, USEPA Texas Nonpoint Source Project Manager

Responsible for overall performance and direction of the project at the Federal level. Ensures that the project assists in achieving the goals of the federal Clean Water Act (CWA). Reviews and approves the quality assurance project plan (QAPP), project progress, and deliverables.

**TSSWCB** – Texas State Soil and Water Conservation Board, Temple, Texas. Provides project overview at the State level.

Thomas J. Helton, TSSWCB Project Leader

Responsible for ensuring that the project delivers data of known quality, quantity, and type on schedule to achieve project objectives. Tracks and reviews deliverables to ensure that tasks in the work plan are completed as specified.

Donna Long, TSSWCB QAO

Reviews and approves QAPP and any amendments or revisions and ensures distribution of approved/revised QAPPs to TSSWCB and USEPA participants. Responsible for verifying that the QAPP is followed by project participants. Determines that the project meets the requirements for planning, quality assessment (QA), quality control (QC), and reporting under the CWA Section 319 program. Monitors implementation of corrective actions. Coordinates or conducts audits of field and laboratory systems and procedures.

**TWRI** – Texas Water Resources Institute, College Station, Texas. Project Facilitator. Provides the primary point of contact between the TSSWCB and the project contractors. Tracks and reviews deliverables to ensure that tasks in the work plan are completed as specified. Responsible for coordination, review, and delivery of quarterly reports and the final project report.

Dr. C. Allan Jones, TWRI Director; Project Coordinator

Responsible for ensuring that tasks and other requirements in the contract are executed on time and as defined by the grant work plan; assessing the quality of work by participants; submitting accurate and timely deliverables and costs to the TSSWCB Project Lead; and coordinating attendance at conference calls, meetings, and related project activities.

Kevin Wagner, TWRI QAO

Responsible for determining that the QAPP meets the requirements for planning, quality control, quality assessment, and reporting for activities conducted by TWRI. Responsible for maintaining records of QAPP distribution, including appendices and amendments. Coordinates the research and review of technical QA material and data related to water quality monitoring system design and analytical techniques.

**TAMU-Biological and Agricultural Engineering Department** – Texas A&M University, College Station, Texas. Project Leader. Responsible for collection of water samples. Responsible for the data analysis and interpretation in water samples. TAMU - Bio. and Ag. Eng. Dept. will contribute to the development of quarterly reports and the final project report.

Dr. Saqib Mukhtar, Associate Professor, Animal Waste Management; Project Leader

Responsible for coordinating and supervising field sampling activities. Responsible for ensuring that field personnel have adequate training and a thorough knowledge of standard operating procedures (SOPs) specific to the analysis or task performed and/or supervised. Responsible for ensuring that tasks and other requirements in the contract are executed on time and in accordance with the QA/QC requirements in the system as defined by the contract work plan and in the QAPP. Responsible for verifying that the data produced are of known and acceptable quality. Responsible for ensuring adequate training and supervision of all activities involved in generating analytical data for this project. Responsible for the facilitation of audits and the implementation, documentation, verification, and reporting of corrective actions. Responsible for submitting accurate and timely data analyses and other materials for progress and final reports to TWRI.

Dr. Clyde Munster, Professor, Environmental Soil and Water Engineering

Responsible for conducting column lysimeter experiments. This will include construction of the column lysimeters, filling them with soil, planting the turfgrass and applying the geotube biosolids. Responsible for conducting the leaching experiments to determine the impacts of biosolid application on the quality of water collected as surface runoff or water moving through the soil profile.

**TAMU-Department of Soil and Crop Science** – Texas A&M University, College Station, Texas. Responsible for collection of biosolid samples. Responsible for the data analysis and interpretation for biosolid and leachate samples. TAMU – Soil and Crop Sciences Dept. will contribute to the development of quarterly reports and the final project report.

Donald Vietor, Professor, Agronomy

Responsible for collecting samples of geotube biosolids and analyzing its physical and chemical properties. Responsible for conducting analysis of  $\text{NO}_3\text{-N}$ ,  $\text{NH}_4\text{-N}$ , dissolved P, and soluble reactive P in water extracts of geotube residues, soil, and leachate from

lysimeters. In addition, will conduct statistical analysis of results of soil and leachate analysis from the column lysimeter experiments.

**TIAER** – Texas Institute for Applied Environmental Research, Tarleton State University, Stephenville, Texas. Laboratory Support. Responsible for data analysis and reporting tasks for the project.

Nancy Easterling, TIAER Quality Assurance Officer

Responsible for coordinating development and implementation of TIAER's QA program. Responsible for writing and maintaining QAPPs and monitoring their implementation. Ensures the data collected for the project are of known and acceptable quality and adhere to the specifications of the QAPP. Responsible for identifying, receiving, and maintaining project quality assurance records. Responsible for coordinating with the TWRI QAO to resolve QA-related issues. Notifies the TIAER Program Manager (Larry Hauck) of particular circumstances which may adversely affect the quality of data. Implements or ensures implementation of corrective actions needed to resolve nonconformances noted during assessments. Conducts in-house audits to ensure compliance with written SOPs and to identify potential problems.

Mark Murphy, TIAER Laboratory Manager

Responsible for supervising TIAER chemistry laboratory personnel involved in generating analytical data for this project. Responsible for ensuring that laboratory personnel involved in generating analytical data have adequate training and a thorough knowledge of the QAPP and all SOPs specific to the analysis or task performed and/or supervised. Responsible for oversight of all laboratory operations and ensuring that all quality assurance-quality control requirements are met. Responsible for documentation related to laboratory analyses. Enforces corrective action, as required. Develops and facilitates laboratory system audits with TIAER QA officer. Performs validation and verification of data before report is sent to primary contractor.

**SWFTL** – Soil, Water and Forages Testing Laboratory, Texas Cooperative Extension, Texas A&M University, College Station, Texas. Laboratory Support. Responsible for analysis and reporting of the turfgrass demonstration conducted in the project.

Tony Provin, State Soil Chemist, TCE, SWFTL Director

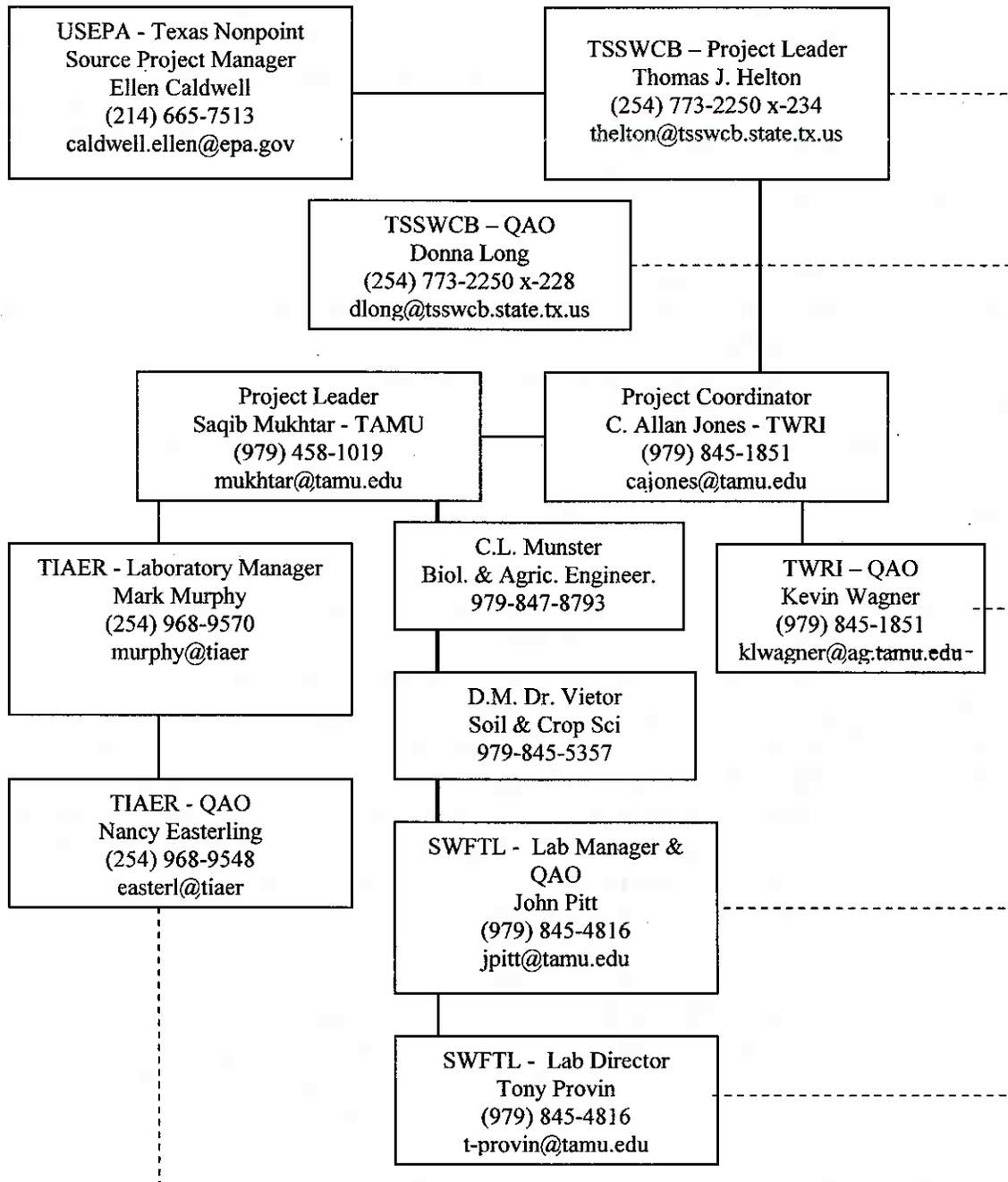
Responsible for directing TCE Soil, Water and Forage Testing Laboratory personnel involved in generating analytical data for this project. Responsible for ensuring that laboratory personnel involved in generating analytical data have adequate training and a thorough knowledge of the TCE Soil, Water and Forage Testing Laboratory SOPs specific to the analysis or task performed and/or supervised. Responsible for oversight of all laboratory operations and ensuring that all SOPs 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 and manuscript development.

John Pitt, TCE, SWFTL Manager and QAO

Assumes all responsibilities TCE Laboratory in the absence of the laboratory director. Monitors the implementation of the QAM and this QAPP within the TCE-SWFTL to ensure complete compliance with QA objectives as defined by the contract and in the QAPP in reference to soil and water samples. Conducts internal audits to identify potential problems and ensure compliance with written SOPs. Responsible for supervising and verifying all aspects of the QA/QC in the laboratory. Performs validation and verification of data before the report is sent to the contractor. Insures that all QA reviews are conducted in a timely manner.

**Figure A4-1. Project Organization Chart**

Dashed lines indicate communication only



## **Section A5: Problem Definition/Background**

In 1998, segments 1226 and 1255 (corresponding to the North Bosque River and Upper North Bosque River segments) were deemed "impaired segments" on the State of Texas Clean Water Act (CWA) Section 303(d) under water quality standards related to nutrients and aquatic plant growth. Recent studies conducted or sponsored by the Texas Commission on Environmental Quality (TCEQ), TSSWCB, TIAER, TWRI and others have demonstrated that high levels of phosphorus (P) and other nutrients from point and nonpoint sources degrade water quality in the North Bosque River. Nonpoint sources such as dairy waste application fields (WAF) and point sources such as municipal wastewater treatment plants are the major controllable sources of P in the watershed.

These findings led to the USEPA approval for the two Total Maximum Daily Loads (TMDLs) for P in the North Bosque River. In December 2002, the TCEQ approved the implementation plan for the two TMDLs, and the TSSWCB approved them in January 2003. The goal of these TMDLs is to achieve a reduction of total annual loading and annual average concentrations of soluble reactive P (SRP) by approximately 50%. The Bosque River Advisory Committee expects that both point and nonpoint sources will have to make significant reductions in their P contributions to achieve this goal. It is anticipated that SRP reductions of this magnitude will reduce the potential for problematic algal growth in the North Bosque River and Lake Waco.

There are roughly 41,000 dairy cows in the Bosque River watershed. An adjacent watershed, the Leon River, contains approximately 50,000 dairy cows. Runoff from production areas such as feedlots and feed lanes is regulated as point source. Runoff from WAFs is not regulated, and therefore is treated as a nonpoint source. It is anticipated that the measures to control SRP loading from WAFs may include a combination of dairy confined animal feeding operations (CAFOs) regulated for land application of manure and wastewater, as well as voluntary programs. Several permitted dairies, in both watersheds, use some kind of best management practices (BMPs) to reduce nutrients in the effluent being applied to the WAFs. In most cases, these include separation of solids from liquid manure by either gravitational (settling basins) or mechanical (screen separators) methods to remove as much as 40% of solids from liquid dairy manure. While separating solids does reduce total P, as much as 90% of the SRP remains in the effluent to be stored in a basin or lagoon and then land applied to the WAFs. Low-cost, highly efficient and easy-to-adopt technologies in the form of BMPs that will reduce total P and SRP from dairy effluent that is applied on the WAFs will contribute significantly to the overall goal of 50% SRP reduction in annual loading for the two TMDLs in the North Bosque River. In recent years, techniques such as electrocoagulation (a process where electrical current passing between metal electrodes is used to remove dissolved and suspended constituents from wastewater), and chemical precipitation of contaminants with metal coagulants (aluminum sulfate, also known as alum and similar compounds) have been promoted as ways to remove nutrients from dairy wastes. Other methods that have been noted include polymers (such as synthetic long chain polyacrylamides), deep aeration of

lagoon using micro-bubblers, and geotextile materials. In addition, use of special microbial additives may help precipitate suspended solids (with associated P) and reduce odors.

The North Bosque River and Leon River watersheds contain areas that have concentrated numbers of dairy operations. Dairy lagoons are designed and built to catch and contain process water and certain amounts of rainwater on most dairies. The lagoons must be dewatered from time to time, which adds phosphorus to the receiving soils. Historical dairy waste application fields that are subject to lagoon dewatering activities have been identified as potentially significant sources of phosphorus entering the waterways. In order to alleviate phosphorus loading on these WAFs, it would be beneficial to utilize new technologies. It is the purpose of this project to demonstrate reductions in phosphorus from many of these innovative, phosphorus-based methods.

## **Section A6: Project/Task Description**

The implementation of this project consists of evaluation of five new technologies that will be demonstrated on cooperators' dairy farms by the providers; electrocoagulation system, a geotextile solids separation system, and three technologies that use proprietary bacterial treatments to all claim that they can reduce the amount of P in a dairy waste stream. The new technologies are tested and utilized in municipal waste treatment systems, dredging and sediment recovery from streams, and oil and gas industry but they have not been adequately tested or demonstrated for treating animal waste. This is especially true for testing these technologies for the reduction of P from land applied liquid dairy manure in the Bosque river watershed. This project will also conduct a demonstration that evaluates the impacts and effects of growing turfgrass on soils amended with residual material contained inside of a geotextile solids separation system. A brief description of example technologies and the turfgrass demonstration is provided.

**Electrocoagulation System:** This technology is based upon the principle of electrical precipitation using cations from metal (aluminum and/or iron electrodes) as coagulants to remove phosphorus from liquid waste in an electrically energized state. The primary action is combining of orthophosphates with the metal ions but polyphosphates and organic phosphorous compounds are also removed by being absorbed or entrapped in the floc particles. The floc is then removed using a number of removal techniques, including filtration, dissolved air floatation, and skimming methods. The provider for this technology indicates that their system can treat liquid waste at a rate of up to 100 gallons per minute and remove significant amounts of P and other heavy metals as well as reduce pathogenic activity in the treated effluent. Preliminary studies by manufacturers will be the basis for demonstration of an optimized process using dairy effluent.

**Geotextile Solids Separation System:** This system is comprised of a large porous tube made from a heavy duty polypropylene fabric, with a large circumference (up to 45 feet) and variable lengths (up to 400 feet) to remove solids from slurry pumped into the tube. This system has been used in erosion control and sediment removal from streams. Lagoon effluent from the animal feeding operations can be pumped into the tube and as the liquid leaves the tube due to its porous structure, solids larger than the pore size of the tube are trapped. This process can be repeated until the tube is full. The liquid (effluent out of the tube) with reduced amount of phosphorus may be routed back to the lagoon or to a waste application field. The removed solids, now lower in moisture, can be hauled to a relatively longer distance to fields with low soil phosphorous or composted on site and then land applied to distant fields with low soil phosphorous. The use of geotextile tubes needs to be demonstrated at full scale in the dairy industry.

**L4DB Microbial Treatment:** This technology uses proprietary microbes mixture named L4DB. This mixture consists of an enzyme and microorganism mix extracted from the natural destructive fermentation process of a proprietary mix of natural organic plant

material under controlled conditions. The project will collect baseline data from the chosen dairy's lagoons before the initial inoculation with the L4DB microbes. Lagoons will continue to be inoculated with the microbe mixture as seen fit by the applicators. During the first five months of the demonstration, samples of sludge and liquid will be taken from the lagoon; after the fifth month, sampling will be reduced to every other month. This extended sampling period is being used to capture the effects of the treatment across an entire year. Two tanks will also be used to evaluate the performance of the product and compare it to a controlled tank. This tank set-up will also allow for calculating a mass balance of tank influent and effluent. Results of this demonstration will show the ability of this product to reduce P in waters that will be applied to waste application fields and its potential to help reduce the P loading problem in the Bosque and Leon River watersheds.

**NBT-100 *Lactobacillus* Treatment:** This technology will demonstrate the use of specially prepared *lactobacillus* (NBT-100) that consumes pathogens and nutrients at a faster rate than regular *lactobacillus*. Initial tests will be conducted to evaluate the composition of the influent entering the lagoon and the waste that is stored before inoculation. Inoculation will occur at a 1 to 10,000 ratio (microbes to lagoon liquid) under the surface of the lagoon. The technology provider claims that effects will immediately be seen after inoculation has occurred. Samples will be collected on a periodic basis to analyze the effects that the treatment process has on P and other constituent in the lagoons. A 90 day project period is planned for this demonstration; the technology provider suggests that the 50% P reduction will be met before the 90<sup>th</sup> day of the demonstration.

**Bacterial Stimulation and Oxygenation:** This demonstration uses a combination of products that stimulate and oxygenate a lagoon to increase the capacity of organisms to breakdown the nutrients and pathogens in the lagoon. Initial samples will be collected to evaluate the make-up of effluent currently stored in the lagoon and the waste stream entering the lagoon. A dosage rate will be calculated based on the average daily loading of the lagoon and will be automatically applied daily. After inoculation, samples will be collected on a periodic basis to determine its impact on reducing nutrients stored in the lagoon. A 60 day demonstration was requested by the technology provider. During this time, a 50 to 75% reduction in P is expected.

**Turfgrass Grown on Residue from Geotextile Solids Separation System:** This demonstration will evaluate the effects of geotube residue on soil properties and the growth response of Tifway bermudagrass turf planted in column lysimeters within a greenhouse at Texas A&M. The turf will be planted on four replications of contrasting soil types with and without incorporation of 12.5% and 25% by volume of geotube residue. Turfgrass growth and concentrations of total N and P and cations will be monitored over a 90-d period under irrigated conditions in column lysimeters. In addition, pH and loss of N and P forms, organic carbon, and cations in leachate will be monitored for one pore volume of percolate 45 and 90 d after planting of turfgrass. A potential use of geotube

residue for turfgrass production near CAFOs, and potential nutrient export from the Bosque River Watershed through turfgrass sod will be demonstrated. The residue recovered from geotextile solids separation systems contain trapped nutrients and organic matter that could be composted or directly applied to soil as an amendment to enhance production of a turfgrass crop. Yet,  $Al_2SO_4$  and polymers were injected to coagulate P and particles during pumping of wastewater through geotubes, which could be detrimental to growth of turfgrass and other crop plants. Once the turfgrass is harvested, it can be sold outside of the watershed and will effectively remove a significant portion of the P that was applied to the soil as an amendment. As an added benefit, sod produced in this manner has been shown to need no additional P fertilizer for at least 10 years after it has been harvested and transplanted.

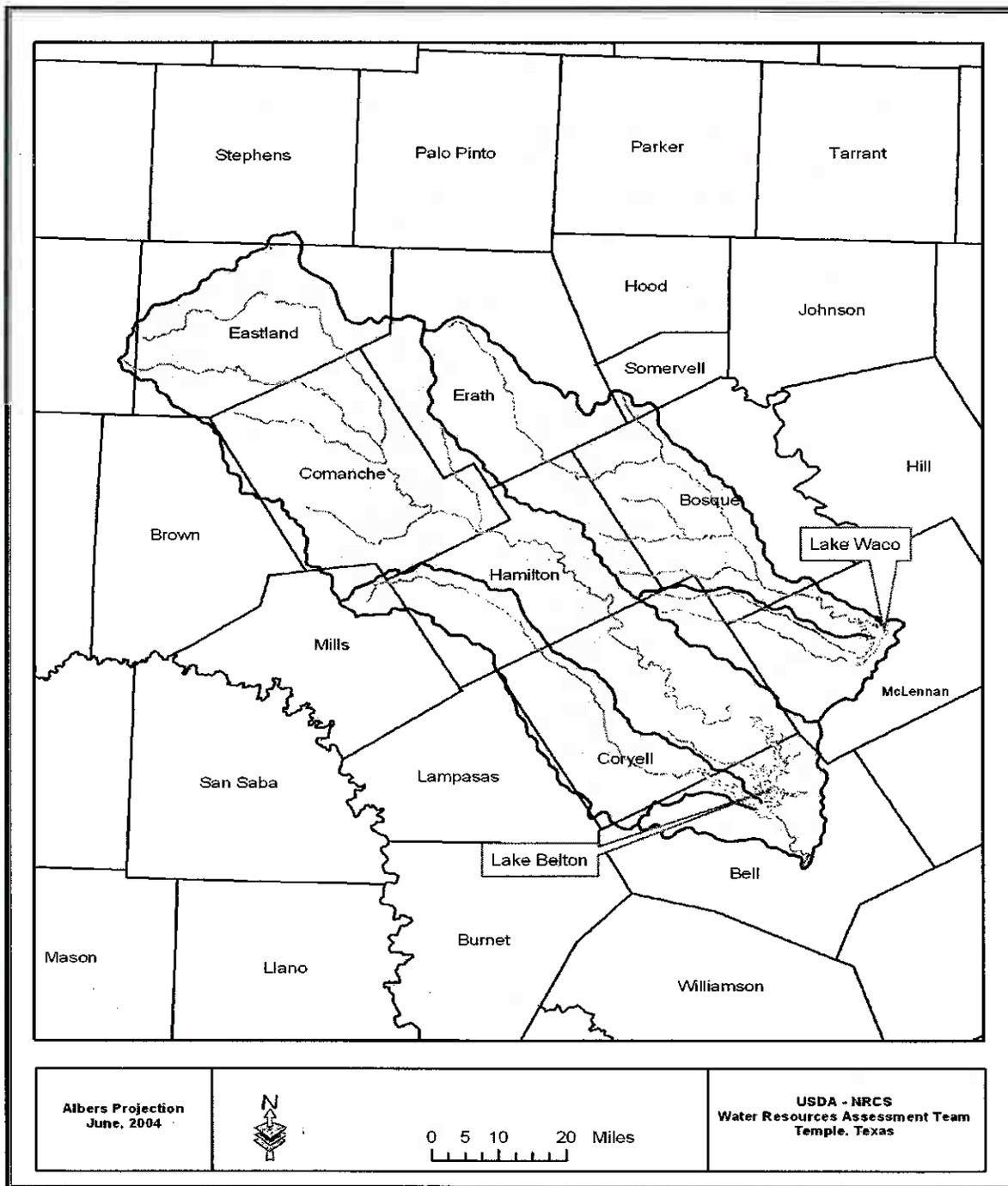
An advisory committee will comprise Texas A&M University System scientists, engineers and extension agents, personnel from the dairy industry, Texas Farm Bureau (TFB), EPA Region 6, the TSSWCB, Texas Cooperative Extension (TCE), the TCEQ, the Brazos River Authority (BRA), the USDA-NRCS, and the Texas Agricultural Experiment Station (TAES), dairy operators and owners, and technology providers will provide:

- Selection of specific pilot systems to be evaluated,
- Selection of dairy facilities on which to conduct the evaluations,
- Protocols and procedures to be used in the evaluation, and
- Publications, field demonstrations, and other documentation to be prepared.

Each technology will be evaluated for its efficacy to reduce total P and SRP, and other nutrients and metals, by sampling and analyzing the raw and treated effluent. Cost effectiveness, treatment efficiency, and ease of adoption of the technology, as a BMP will be evaluated. Each technology will be demonstrated for a period of at least three months. The advisory committee will be consulted to provide input on the priority and order of the evaluation/demonstration. The committee will also provide assistance in identifying and interacting with companies interested in demonstrating and evaluating the efficacy of the technologies. TIAER, an independent analytical laboratory that performs work under numerous quality assurance project plans approved by TCEQ and EPA, will be used for sample analyses.

The turfgrass demonstration will be conducted in the turfgrass greenhouses at Texas A&M in College Station, TX. It will be evaluated to determine the effects of the incorporation of residual biosolids materials on the growth of turfgrass and groundwater water quality. The SWFTL and Dr. D.M. Vietor at Texas A&M will conduct leachate sampling and analysis.

**Figure A6-1. Bosque River and Leon River Watersheds.**



Tasks for this project include the following:

1. Identification of potential technology providers.
2. Identification of dairy cooperators in the North Bosque and Leon watershed areas that use a flush system and lagoons to remove, store and treat, land-apply effluent and are willing to participate in these demonstrations.
3. On-site installation and start-up of the five pilot-scale technologies to be demonstrated.
4. Set up and demonstration of turfgrass and soil responses in column lysimeters under greenhouse conditions with and without soil amendments of residual material from a geotextile solids separation system.
5. QAPP preparation; field data collection and analysis.
6. Development of reports and outreach education materials on effectiveness of the innovative technologies.
7. Development of a final report assessing preexisting and post implementation effects of the project.

Subtasks are outlined in Table A6-1 along with a listing of the responsible agency or agencies and an activity schedule.

**Table A6-1. Project Plan Milestones**

TASK	PROJECT MILESTONES	AGENCY	START	END
1.1a	TCE set up advisory committee consisting of members from: TCE, TWRI, TFB, dairy industry representatives, EPA Region 6, TSSWCB, TCEQ, BRA, NRCS and TAES	TCE, TAMU	Dec03	Jan04
1.1b	Advisory committee will identify potential technology providers, select promising technologies and prioritize demonstrations	TCE, TAMU	Jan04	Mar07
1.2	TCE, TSSWCB and TFB will identify dairy cooperators in the North Bosque watershed that use a flush type system and lagoons to remove, store, treat and land-apply effluent (manure and process-generated wastewater)	TCE, TSSWCB, TFB	Jan04	Mar07
1.3	Each technology provider (with dairy cooperator permission) will prepare and install each of the five pilot-scale technologies chosen to be demonstrated , including turfgrass in column lysimeters.	TAMU, TCE	Aug03	Aug07
1.4a	TCE develop DQOs and QAPP and submit to EPA for approval	TCE, TAMU	Mar04	Sept04
1.4b	EPA approve QAPP	TSSWCB, EPA	Sept04	Oct04
1.5a	TCE in cooperation with TWRI will develop reports and outreach education materials	TCE, TWRI		
1.5b	TCE and TWRI in cooperation will submit quarterly progress reports to TSSWCB	TCE, TWRI, TSSWCB	Jan04	Jan08
1.5c	TCE and TWRI will submit draft final report to TSSWCB	TCE, TWRI, TSSWCB	Dec07	Jan08
1.5d	Submit final report		Jan08	Mar08

## **Section A7: Data Quality Objectives for Measurement Data**

The objectives of the water quality monitoring implemented for this project are as follows:

- 1) To reduce nonpoint source NPS pollution of P by removing total TP and SRP from dairy Lagoon effluent applied to waste application fields. This will be accomplished by evaluation and demonstration of at least six new technologies, over a period of three years, which purport to remove P from the dairy waste stream.
- 2) To demonstrate the impact and effectiveness of each new technology, including the effects of geotextile solid separation system residual material amendments on turfgrass growth and water quality, to producers within the watershed via field days, educational brochures and publications. Dairy producers, media representatives, scientists, engineers, regulatory personnel, representatives of commodity groups, and the general public in the Upper Leon River, North Bosque River and Upper North Bosque basins and surrounding areas will be invited to view the systems in operation. A guidance document for operators will be offered through the TCE online bookstore.

Liquid, slurry, and solid samples will be collected and analyzed for the presence of nitrite + nitrate--nitrogen ( $\text{NO}_2+\text{NO}_3\text{-N}$ ), total Kjeldahl nitrogen (TKN), orthophosphate phosphorus ( $\text{PO}_4\text{-P}$ ), total phosphorus (TP), non-critical macro and micro nutrients- (i.e.-K, Ca, Mg, Na, Mn, Fe, Cu), total suspended solids (TSS), total solids (TS), volatile solids (VS) and Aluminum (Al). These parameters will be measured because they are good indicators of water quality with respect to nutrient NPS pollution. The major data quality objective is to demonstrate reductions in phosphorus from innovative, phosphorus based, BMPs.

Samples will be analyzed if they meet preservation requirements and holding times. All samples will be analyzed within the estimated accuracy and precision limits of measured parameters to insure data quality (Table A7-1).

Database checks for validity will be performed on an on-going basis. Data will be reviewed for abnormalities or any unusual results, e.g., a sample with a concentration of orthophosphate-phosphorus higher than the concentration of total phosphorus, prior to entry into the database. 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 data will be discarded.

The Project Leader will coordinate with the TIAER Laboratory Manager, the SWFTL Laboratory Manager, and TAMU Research staff to ensure that proper protocols are utilized.

The measurement performance specifications to support the project objectives for a minimum data set are specified in Table A7.1 and in the text following.

**Table A7.1 – TIAER Measurement Performance Specifications**

PARAMETER	Matrix	PARAMETER CODE	UNITS <sup>3</sup>	METHOD	AWRL	RL (Lab Reporting Limit)	RECOVERY at Reporting Limit (%)	PRECISION (RPD of LCS/LCSD mean) <sup>1</sup>	BIAS (%Rec. of LCS/LCSD mean) <sup>2</sup>
Total Kjeldahl N	Solid		mg/Kg	EPA 351. <sup>3</sup> , Modified <sup>4</sup>	NA	4	NA	20	80-120
Total Kjeldahl N	Aqueous	00625	mg/L	EPA 351. <sup>3</sup> Modified <sup>4</sup>	0.2	0.2	75-125	20	80-120
Nitrate/nitrite-N, extractable	Solid		mg/Kg	SSSA 38-1148	NA	1	NA	20	80-120
Nitrate/nitrite-N, dissolved	Aqueous	00631	mg/L	EPA 353.2 <sup>3</sup>	0.04	0.04	75-125	20	80-120
Soluble Phosphorus	Solid		mg/Kg	SSSA 32-891	NA	1	NA	20	80-120
O-phosphate-P, dissolved, lab filtered	Aqueous	70507	mg/L	EPA 365.2 <sup>3</sup>	0.04	0.005	75-125	20	80-120
Total phosphorus	Solid		mg/Kg	EPA 365.4 <sup>3</sup> , Modified <sup>4</sup>	NA	Estimated 1	75-125	20	80-120
Total phosphorus	Aqueous	00665	mg/L	EPA 365.4 <sup>3</sup> , Modified <sup>4</sup>	0.06	0.06	75-125	20	80-120
Potassium	Solid		mg/Kg	EPA 200.7 <sup>5</sup>	NA	6	NA	20	80-120
Potassium	Aqueous	00937	mg/L	EPA 200.7 <sup>5</sup>	0.3	0.3	60-120	20	80-120
Calcium	Solid		mg/Kg	EPA 200.7 <sup>5</sup>	NA	6	NA	20	80-120
Calcium	Aqueous	00916	mg/L	EPA 200.7 <sup>5</sup>	0.01	0.01	60-120	20	80-120
Magnesium	Solid		mg/Kg	EPA 200.7 <sup>5</sup>	NA	6	NA	20	80-120
Magnesium	Aqueous	00927	mg/L	EPA 200.7 <sup>5</sup>	0.02	0.02	60-120	20	80-120
Sodium	Solid		mg/Kg	EPA 200.7 <sup>5</sup>	NA	6	NA	20	80-120
Sodium	Aqueous	00929	mg/L	EPA 200.7 <sup>5</sup>	0.03	0.03	60-120	20	80-120
Manganese	Solid		Mg/Kg	EPA 200.7 <sup>5</sup>	0.05	.05	60-120	20	80-120
Manganese	Aqueous	01055	mg/L	EPA 200.7 <sup>5</sup>	0.05	0.05	75-125	20	80-120
Iron	Solid		mg/Kg	EPA 200.7 <sup>5</sup>	NA	1	NA	20	80-120
Iron	Aqueous	01045	mg/L	EPA 200.7 <sup>5</sup>	0.03	0.03	60-120	20	80-120
Copper	Solid		mg/Kg	EPA 200.7 <sup>5</sup>	NA	.05	NA	20	80-120
Copper	Aqueous	01042	mg/L	EPA 200.7 <sup>5</sup>	0.003	0.003	60-120	20	80-120
Aluminum	Solid		mg/Kg	EPA 200.7 <sup>5</sup>	NA	4	NA	20	80-120
Aluminum	Aqueous	01105	mg/L	EPA 200.7 <sup>5</sup>	0.2	0.2	60-120	20	80-120

PARAMETER	Matrix	PARAMETER CODE	UNITS <sup>3</sup>	METHOD	AWRL	RL (Lab Reporting Limit)	RECOVERY at Reporting Limit (%)	PRECISION (RPD of LCS/LCSD mean) <sup>1</sup>	BIAS (%Rec. of LCS/LCSD mean) <sup>2</sup>
TSS	Aqueous	00530	mg/L	EPA 160.2	4.0	4.0	NA	20	80-120
Percent Solids	Solids		%	SM 2540B	NA	0.1	NA	NA	NA
TS	Aqueous	00500	mg/L	SM 2540B	10.0	10.0	NA	NA	20
TVS	Solid		%	SM 2450G	NA	0.1	NA	NA	NA
TVS	Aqueous	00505	mg/L	EPA 160.4	10.0	10.0	NA	NA	NA
Potential Hydrogen (pH)	Solid		pH std. units	EPA 9045C	NA	NA	NA	NA	NA
Potential Hydrogen (pH)	Aqueous	00403	pH std. units	EPA 150.1	NA	NA	NA	NA	NA
Conductivity	Solid		mg/Kg	EPA 9050A	NA	NA	NA	NA	NA
Conductivity	Aqueous	00095	μS/cm	EPA 120.1	NA	NA	NA	NA	NA

- 1 Precision results will not be used as acceptance criteria if values below the practical quantification limit.
- 2 mg/Kg (ppm) for solids is based on a dry weight basis.
- 3 In case of equipment malfunction and resulting holding time issues, the alternate back-up analytical method for total phosphorus will be EPA 365.4, modified in the same way; EPA 351.1-4 for TKN; EPA 300.0, EPA 352.1, EPA 353.1-3, and EPA 354.1 for NO<sub>2</sub>-N+NO<sub>3</sub>-N, EPA 300.0 and EPA 365.2 and lab-filtered EPA 365.2 (code 70507) for PO<sub>4</sub>-P, EPA 365.4 (modified as per footnote 4) for total P. EPA 351.2 for TKN states that the digestate may also be used for total P. If an alternative method is necessitated, all QC, AWRLs, recovery, precision, and bias limits required by TCEQ will be followed.
- 4 Modification of the total phosphorus method involves using CuSO<sub>4</sub> instead of HgSO<sub>4</sub>. Documentation of TIAER's ability to achieve acceptable performance using the modification is kept by the TIAER analytical laboratory. EPA 351.2 for TKN states that the digestate may also be used for total P.
- 5 The calibration curve for metals analyzed by the ICP, using EPA 200.7 may not include a standard at the AWRL. All other QC for the procedure will be performed. Affected data will be flagged. EPA 200.7 revision 5.0 (1998), which includes solids, will be used. There is no difference between EPA methods 200.7 and 6010B. Method 200.7 is a newer version and will yield the same results.
- 6 AWRL levels will not apply for metals on these samples.

**Table A7.2 – Estimated SWFTL Accuracy and Precision Limits**

PARAMETER	Precision (RPD) <sup>b</sup>	Limits <sup>a</sup>	Bias <sup>a</sup> and % Recovery Limits	MDL <sup>c</sup>	Reporting Limits
<b>Soil</b>					
Total N	20%		80-120%	0.1 mg/kg <sup>d</sup>	1.0 mg/kg
Total P	20%		80-120%	0.1 mg/kg	1.0 mg/kg
pH	NA		± 0.2	NA	0.2 pH units
Extractable P	20%		80-120%	0.1 mg/kg	1.0 mg/kg
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	20%		80-120%	0.1 mg/kg	1.0 mg/kg
NH <sub>4</sub> -N	20%		80-120%	0.1 mg/kg	1.0 mg/kg
Water soluble P	20%		80-120%	0.1 mg/kg	1.0 mg/kg
Potassium	20%		80-120%	0.1 mg/kg	5.0 mg/kg
Calcium	20%		80-120%	0.1 mg/kg	1.0 mg/kg
Magnesium	20%		80-120%	0.1 mg/kg	5.0 mg/kg
Sodium	20%		80-120%	2.4 mg/kg	10.0 mg/kg
Aluminum	20%		80-120%	0.1 mg/kg	1.0 mg/kg

Organic Carbon	1%	± 1%	NA	0.5%
<b>Water</b>				
Total N	20%	80-120%	0.1 mg/L <sup>f</sup>	1.0 mg/L
Total P	20%	80-120%	0.1 mg/L	1.0 mg/L
pH	NA	± 0.2	NA	0.2 pH units
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	20%	80-120%	0.5 mg/L	0.25 mg/L
NH <sub>4</sub> -N	20%	80-120%	0.5 mg/L	0.5 mg/L
Water soluble P	20%	80-120%	0.5 mg/L	0.5 mg/L
Potassium	20%	80-120%	0.01 mg/L	5.0 mg/L
Calcium	20%	80-120%	0.01 mg/L	10.0 mg/L
Magnesium	20%	80-120%	0.01 mg/L	5.0 mg/L
Sodium	20%	80-120%	0.02 mg/L	10.0 mg/L
<b>Geotube residue</b>				
Total N	20%	80-120%	0.1mg/kg	1.0 mg/kg
Total P	20%	80-120%	0.1 mg/kg	1.0 mg/kg
pH	NA	± 0.2	NA	0.2 pH units
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	20%	80-120%	0.1 mg/kg	1.0 mg/kg
NH <sub>4</sub> -N	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Water soluble P	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Potassium	20%	80-120%	0.1 mg/kg	5.0 mg/kg
Calcium	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Magnesium	20%	80-120%	0.1 mg/kg	5.0 mg/kg
Sodium	20%	80-120%	2.4 mg/kg	10.0 mg/kg
Aluminum	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Organic Carbon	1%	± 1%	NA	0.5%
Zinc	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Iron	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Manganese	20%	80-120%	0.1 mg/kg	3.0 mg/kg
Copper	20%	80-120%	0.3 mg/kg	3.0 mg/kg
<b>Turfgrass</b>				
Total N	20%	80-120%	0.1 mg/kg	200 mg/kg
Total P	20%	80-120%	0.5 mg/kg	200 mg/kg
Potassium	20%	80-120%	1.5 mg/kg	200 mg/kg
Calcium	20%	80-120%	2.2 mg/kg	200 mg/kg
Magnesium	20%	80-120%	1.1 mg/kg	200 mg/kg
Sodium	20%	80-120%	55 mg/kg	200 mg/kg
Aluminum	20%	80-120%	0.1 mg/kg	3.0 mg/kg
Zinc	20%	80-120%	0.2 mg/kg	3.0 mg/kg
Iron	20%	80-120%	0.1 mg/kg	3.0 mg/kg
Manganese	20%	80-120%	0.1 mg/kg	3.0 mg/kg
Copper	20%	80-120%	0.3 mg/kg	3.0 mg/kg

<sup>a</sup> Precision and bias are determined by analysis of laboratory media standards

<sup>b</sup> RPD = relative percent difference    <sup>c</sup>MDL = method detection limit    <sup>d</sup>NA = Not applicable

<sup>e</sup>mg/kg = milligrams/kilograms    <sup>f</sup>mg/L = milligrams per liter

**Table A7.3. Estimated accuracy and precision limits for Dr. Viotor's laboratory.**

Variable	Precision	Bias <sup>a</sup> and % Recovery	MDL <sup>c</sup>	Reporting Limits
	Limits <sup>a</sup> (RPD) <sup>b</sup>	Limits		
<b>Soil</b>				
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	20%	80-120%	0.1 mg/kg <sup>e</sup>	1.0 mg/kg
NH <sub>4</sub> -N	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Soluble reactive P	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Bulk Density	10%	NA <sup>d</sup>	0.01 g/L	0.1 g/L
Water content	20%	NA	0.1 mL/L	1.0 mL/L
<b>Water</b>				
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	20%	80-120%	0.05 mg/L <sup>f</sup>	0.25 mg/L
NH <sub>4</sub> -N	20%	80-120%	0.05 mg/L	0.5 mg/L
Soluble reactive P	20%	80-120%	0.01 mg/L	0.05 mg/L
Dissolved organic C	20%	80-120%	0.1 mg/L	1.0 mg/L
<b>Geotube residue</b>				
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	20%	80-120%	0.1 mg/kg	1.0 mg/kg
NH <sub>4</sub> -N	20%	80-120%	0.1 mg/kg	1.0 mg/kg
Soluble reactive P	20%	80-120%	0.1 mg/kg	1.0 mg/kg

<sup>a</sup> Precision and bias are determined by analysis of laboratory media standards

<sup>b</sup> RPD = relative percent difference

<sup>c</sup>MDL = method detection limit

<sup>d</sup>NA = Not applicable

<sup>e</sup>mg/kg = milligrams/kilogram

<sup>f</sup>mg/L = milligrams per liter

### **Precision**

The precision of data is a measure of the reproducibility of a measurement when an analysis is repeated. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions.

TIAER laboratory precision is assessed by comparing replicate analyses of laboratory control standards (LCS/LCSD) and/or sample/duplicate pairs. Performance limits for laboratory duplicates are defined in the Table A7.1.

Field splits are used to assess the variability of sample handling, preservation, and storage, as well as the analytical process, and are prepared by splitting samples in the field. Performance limits for field splits are defined in Section B5.

Precision in SWFTL and Dr. Vietor's laboratories is assessed by replicate analyses of laboratory media standards. The definition of SWFTL laboratory media standards can be found in Section B5. Soil, media standard results within a batch are compared to the statistical known mean value of the laboratory media standard for each analytical procedure used. Water and vegetation laboratory media standard results within a batch are compared to the known value of the laboratory media standard for each analytical procedure used. These comparison results are used to evaluate the SWFTL's analytical performance, specifically to obtain the SWFTL relative percent difference. Performance limits for relative percent difference are listed in Table A7-2.

### **Bias**

Bias is a statistical measurement of correctness and includes components of systemic error. A measurement is considered unbiased when the value reported does not differ from the true value. Bias is verified through the analysis of laboratory control standards and blank samples. Performance limits for the mean results of LCS/LCSD and results of calibration control standards at laboratory reporting limits (RLs), the lowest concentration at which the laboratory will report quantitative data within a specified recovery range, are specified in Table A7.1. Performance limits for blank analyses are discussed in Section B5.

### **Representativeness**

Site selection and sampling of all pertinent media (liquid, slurry, and solids) and use of only approved analytical methods will assure that the measurement data represents the conditions at the site. Representativeness also depends on the number of samples taken to accurately reflect the technological effectiveness at a given site. The goal for meeting total representation for effectiveness of each technology is tempered by the potential funding for complete representativeness.

**Comparability**

Confidence in the comparability of data sets from this project to those for similar uses is based on the commitment of project staff to use only approved sampling and analysis methods and QA/QC protocols in accordance with quality system requirements and as described in this QAPP and project SOPs. Comparability is also guaranteed by reporting data in standard units, by using accepted rules for rounding figures, and by reporting data in a standard format.

**Completeness**

The completeness of the data is basically a relationship of how much of the data is available for use compared to the total potential data. Ideally, 100 percent of the data should be available. However, the possibility of unavailable data due to accidents, insufficient sample volume, broken or lost samples, etc. is to be expected. Therefore, it will be a general goal of this project that 90 percent data completion is achieved. Should less than 90 percent data completeness occur, the Laboratory Manager will initiate corrective action. Data completeness will be calculated as a percent value.

**Section A8: Special Training Requirements/Certification**

There are no special requirements for staff training or certification for this project, however; laboratory analysts have a combination of experience, education, and training to demonstrate a knowledge of their function. Experience, education and training are retained in the respective laboratory personnel files and can be made available during a monitoring systems audit.

### **Section A9: Documentation and Records**

Hard copies of all field data sheets, general maintenance (GM) records for field equipment, will be archived by the TAMU-Bio./Ag. Engineering Department for at least five years. Electronic copies and/or hard copies of all GM records for laboratory equipment, chain of custody forms (COCs), laboratory data entry sheets, calibration logs, and laboratory corrective action reports (CARs) will be archived by SWFTL for at least five years. In addition, the TAMU-Bio. /Ag. Engineering Department will archive electronic forms of all project data for at least five years. A CAR form is presented in Appendix A, a copy of the COC is presented in Appendix B, and a copy of the field data sheet is presented in Appendix C.

Quarterly progress reports will be generated by TWRI and will note activities conducted in connection with the water quality monitoring program, items or areas identified as potential problems, and any variations or supplements to the QAPP; these will be made available on the project website (<http://twri.tamu.edu/project-info/NewTechnologies/>). CARs will be utilized when necessary (Appendix A). CARs will be maintained in an accessible location for reference at TAMU Bio./Ag. Engineering Department. 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. All quarterly progress reports and QAPP revisions will be distributed to personnel listed in Section A3 by the TWRI. TWRI will also be responsible for submitting the final report for this project.

The TSSWCB may elect to take possession of records (or copies thereof) at the conclusion of the specified retention period.

## **Section B1: Sampling Process Design (Experimental Design)**

This project is designed to evaluate the efficacy of five new technologies to reduce total P, SRP, and other nutrients by sampling and analyzing the raw and treated effluent from selected dairy lagoons in the North Bosque River watershed. These technologies have been tested and are utilized in municipal waste treatment systems, dredging and sediment recovery from streams, and oil and gas industry but they have not been adequately tested or demonstrated for treating animal waste. Cost effectiveness, treatment efficiency, and ease of adoption of the technology as an agricultural BMP will also be evaluated. This project will also conduct a demonstration that evaluates the impacts of growing turfgrass on soils amended with residual material contained inside of a geotextile solids separation system. A brief description of example technologies and turfgrass demonstration is provided in Section A6.

An advisory committee comprises Texas A&M University System scientists, engineers and extension agents, personnel from the dairy industry, Texas Farm Bureau (TFB), EPA Region 6, the TSSWCB, Texas Cooperative Extension (TCE), the TCEQ, the Brazos River Authority (BRA), the USDA-NRCS, and the Texas Agricultural Experiment Station (TAES), dairy operators and owners, and technology providers will provide:

- Selection of specific pilot systems to be evaluated,
- Selection of dairy facilities on which to conduct the evaluations,
- Protocols and procedures to be used in the evaluation, and
- Publications, field demonstrations, and other documentation to be prepared.

Each technology will be evaluated for its efficacy to reduce total P and SRP, and other nutrients and metals, by sampling and analyzing the raw and treated effluent. Cost effectiveness, treatment efficiency, and ease of adoption of the technology, as a BMP will be evaluated. Each technology will be demonstrated for a period of at least three months. The advisory committee will be consulted to provide input on the priority and order of the evaluation/demonstration. The committee will also provide assistance in identifying and interacting with companies interested in demonstrating and evaluating the efficacy of the technologies.

Liquid, slurry, and solid samples will be collected and analyzed for the presence of  $\text{NO}_2 + \text{NO}_3$ -N, TKN,  $\text{PO}_4$ -P, TP, non-critical macro and micro nutrients (i.e.-K, Ca, Mg, Na, Mn, Fe, Cu), total solids (TS), volatile solids (VS) and Aluminum (Al). In addition, liquid samples will also be analyzed for total suspended solids (TSS). A complete listing of the waterborne constituents that will be measured for various new technologies are shown in Table B1-1. These parameters will be measured because they are good indicators of water quality with respect to nutrient NPS pollution and will facilitate the demonstration of reductions in phosphorus from innovative technologies.

Sample collection will commence when technology reaches optimum operational capacity. Four samples of influent, four samples of effluent and two samples of by-product will be collected each sampling event. Total volume of each sample collected will be one liter.

Sampling events will occur at specified intervals and each technology will be evaluated for a minimum of three months. Phosphorous species, solids, conductivity and pH parameters will be run for each sampling event. Nitrogen species parameters will similarly be analyzed for each sampling event. Total metals will be run as specified for sampling events.

A turfgrass demonstration will be conducted at Texas A&M University in the turfgrass laboratory greenhouse. Turfgrass will be planted in column lysimeters that are 10 cm in diameter and 30 cm in depth on contrasting soil textures (Westwood Sandy Clay Loam and Windthorst fine sandy loam). Four replications of a randomized complete block design will comprise two rates of geotextile solids separation system residue (12.5% and 25% by volume). Both rates will be compared to controls of each soil without geotextile solids separation system residue.

The physical and chemical properties of residues from dairy lagoon wastewater in the geotextile solids separation system will be analyzed for two replicate samples from each of two tubes before being incorporated into the column lysimeters for the turfgrass demonstration. One half of each sample will be dried and sieved to determine bulk density and gravimetric water content. Chemical analyses will determine the amounts of total organic C, total N, total P, molybdate-reactive P, K, Ca, Mg, Na, Al, Fe, Mn, Cu, Zn, NO<sub>3</sub>-N and NH<sub>4</sub>-N.

Filtered and unfiltered leachate from the column lysimeters will be collected at 45 and 90 days after the turfgrass is planted. The leachate will be analyzed to evaluate the losses of dissolved organic C, total N, NO<sub>3</sub>-N, NH<sub>4</sub>-N, total dissolved or water-soluble P, soluble-reactive P (SRP), K, Ca, Mg, Na, Al, Fe, Mn, Cu, Zn and organic P (difference between total dissolved P and SRP). Statistical analyses will then be conducted to evaluate the variation of soil physical, chemical and biological properties among main effects and for interactions between soil type and rates of geotextile solids separation residual material.

**Table B1-1. Experimental variables**

<b>Parameter</b>	<b>Status</b>	<b>Reporting Units</b>	<b>Sampling Frequency</b>
Total Nitrogen	Critical	mg/L (ppm)	As specified
Nitrite+Nitrate-Nitrogen	Critical	mg/L (ppm)	As specified
Sol. Reactive Phosphorus	Critical	mg/L (ppm)	As specified
Total Phosphorus	Critical	mg/L (ppm)	As specified
Potassium	Non-critical	mg/L (ppm)	As specified
Calcium	Non-critical	mg/L (ppm)	As specified
Magnesium	Non-critical	mg/L (ppm)	As specified
Sodium	Non-critical	mg/L (ppm)	As specified
Manganese	Non-critical	mg/L (ppm)	As specified
Iron	Non-critical	mg/L (ppm)	As specified
Copper	Non-critical	mg/L (ppm)	As specified
Zinc	Non-critical	mg/L (ppm)	As specified
Total Suspended Solids	Critical	mg/L (ppm)	As specified
Total Solids	Critical	mg/L (ppm)	As specified
Dissolved Organic C	Critical	mg/L (ppm)	45 day interval
Volatile Solids	Critical	mg/L (ppm)	As specified
Potential Hydrogen (pH)	Non-critical	pH standard units	As specified
Conductivity	Non-critical	microseimens per centimeter( $\mu$ S/cm)	As specified
Aluminum	Non-critical	mg/L (ppm)	As specified
Water content	Critical	mL/L (%)	7 day interval
Bulk density	Critical	g/L	90 day interval

The TAMU Bio. /Ag. Engineering Department's research associate will serve as the field technician and will transport properly prepared field samples to the TIAER and SWFTL laboratories for analysis. TIAER, SWFTL, and Dr. Vietor's laboratory will analyze the samples for the variables listed in Table B1-1. These will be the standard analyses for all project samples. For turfgrass and soil responses to geotube residue, Mr. Ronnie Schnell and Drs. Munster and Vietor of TAMU's Soil and Crop Sciences Department will transport properly prepared field samples to the SWFTL and Dr. Vietor's laboratories for analysis. The SWFTL will analyze nitrogen and phosphorus forms, cations including aluminum, pH, and conductivity for soil, turfgrass, geotube, or leachate samples. Dr. Vietor's lab will measure nitrate/nitrite nitrogen, soluble reactive or orthophosphate phosphorus, dissolved organic C in soil and geotube residue extracts and leachate samples. In addition, Vietor's laboratory will measure bulk density and water content of geotube residue and soil.

## **Section B2: Sampling Method Requirements**

All field sampling will follow appropriate protocols set forth in the *TCEQ Surface Water Quality Monitoring Procedures Volume 1: Physical and Chemical Monitoring Methods for Water, Sediment and Tissue* (September 2003). Field sampling activities are documented on field data reporting forms as presented in Appendix C. All sample information will be logged into a field log. The following will be recorded for all sampling:

- station ID / location
- sampling time
- date
- sample collector's name/signature
- COC number

Detailed observational data are recorded including weather, specific sample information, and days since last significant rainfall. Field splits will be required in order to satisfy standard QA/QC protocols for TIAER, SWFTL, and Dr. Vietor's laboratories. Upon collection, all samples will be transported within 3 hr to the laboratory for analysis. Leachate and water samples will be kept on ice or refrigerated during transport. Sample processing and preservation, other than temperature reduction by ice, will be performed in the laboratory.

### **Recording Data**

For the purposes of this section and subsequent sections, all field and laboratory personnel follow the basic rules for recording information as documented below:

- Legible writing with no modifications, write-overs or cross-outs;
- Correction of errors with a single line followed by an initial and date;
- Close-outs on incomplete pages with an initialed and dated diagonal line.

### **Failures in Sampling Methods Requirements and/or Deviations from Sample Design and Corrective Action**

Examples of failures in sampling methods and/or deviations from sample design requirements include but are not limited to such things as sample container problems such as inadequate sample volume due to spillage or container leaks, contamination of a sample bottle during collection, failure to preserve samples appropriately, storage temperature and holding time exceedance, sampling at the wrong site, etc. Any deviations may require corrective action. Corrective action may include for samples to be discarded and re-collected. It is the responsibility of the Project Leader, in consultation with the TWRI QAO, to ensure that the actions and resolutions to the problems are documented and that records are maintained in accordance with this QAPP. The Project Leader will determine if the deviation from the QAPP compromises the validity of the resulting data. The Project Leader, in consultation with the TWRI QAO and TSSWCB QAO will decide to accept or reject data associated with the sampling event, based on best professional judgment. Resolution of the situation will be reported to the TSSWCB in the quarterly report.

## **Section B3: Sample Handling and Custody Requirements**

### **Chain-of -Custody**

Proper sample handling and custody procedures ensure the custody and integrity of samples beginning at the time of sampling and continuing through transport, sample receipt, preparation, and analysis. The COC form is used to document sample handling during transfer from the field to the laboratory, and among subcontract laboratories. The sample number, location, date, 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. At the laboratory, samples are inventoried against the accompanying COC. Any discrepancies will be noted at that time and the COC will be signed for acceptance of custody. At TIAER, SWFTL, and Dr. Vietor's laboratories, sample numbers will then be recorded into a laboratory sample log, where the laboratory staff member who receives the sample will sign it. A copy of TIAER and SWFTL forms, which are also used by Dr. Vietor's lab, are located in Appendix B.

### **Sample Labeling**

Samples are labeled on the container with an indelible, waterproof marker. Label information includes the site identification / location, the date, the sampler's initials, time of sampling, sample type, and the preservative added, if applicable. The COC form will accompany all sets of sample containers.

### **Sample Handling**

Following collection, samples are placed on ice in an insulated cooler for transport to the laboratory. At the laboratory, samples are placed in a refrigerated cooler dedicated to sample storage. The Laboratory Manager has the responsibility to ensure that holding times are met with water samples. The holding time is documented on the COC. Any problems will be documented with a corrective action report.

After samples are received at the laboratory, 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 laboratory sample log and samples will be checked for preservation (as allowed by the specific analytical procedure), filtered or pretreated as necessary, and placed in a refrigerated cooler dedicated to sample storage, where required. Table B3-1 delineates the method, sample container, and holding time information for parameters of interest in this project.

**Table B3-1. TIAER Sample Procedures and Handling Methods**

Laboratory Parameters	Method	Container	Holding Time	Sample Volume	Field Preservation
Nitrite+Nitrate-Nitrogen	EPA 353.2	plastic	28 days	200 mL	filtered, pH<2 with H <sub>2</sub> SO <sub>4</sub> , 4° C
Nitrate/Nitrite-Nitrogen, extractable	SSSA 38-1148	plastic	28 days	200 g	4° C
Total Kjeldahl Nitrogen	EPA 351.2	plastic	28 days	200 mL	pH<2 with H <sub>2</sub> SO <sub>4</sub> , 4° C
Total Kjeldahl Nitrogen	EPA 351.2	plastic	28 days	200 g	4° C
Potassium	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Potassium	EPA 200.7	plastic	6 months	200 g	4° C
Calcium	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Calcium	EPA 200.7	plastic	6 months	200 g	4° C
Magnesium	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Magnesium	EPA 200.7	plastic	6 months	200 g	4° C
Sodium	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Sodium	EPA 200.7	plastic	6 months	200 g	4° C
Manganese	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Manganese	EPA 200.7	plastic	6 months	200 g	4° C
Iron	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Iron	EPA 200.7	plastic	6 months	200 g	4° C
Copper	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Copper	EPA 200.7	plastic	6 months	200 g	4° C
Orthophosphate Phosphorus (lab filtered)	EPA 365.2	plastic	48 hours	200 mL	4° C
Soluble Phosphorus	SSSA 32-891	plastic	6 months	200 g	4° C
Total Phosphorus	EPA 365.4	plastic	28 days	200 mL	pH<2 with H <sub>2</sub> SO <sub>4</sub> , 4° C
Total Phosphorus	EPA 365.4	plastic	6 months	200 g	4° C
Total Suspended Solids	EPA 160.2	plastic	7 days	400 mL	4° C
Total Solids	SM 2540B	plastic	7 days	400 mL	4° C
Percent Solids	SM 2540 B	plastic	6 months	200 g	4° C
Volatile Solids	EPA 160.4	plastic	7 days	400 mL	4° C
Volatile Solids	SM 2450G	plastic	6 months	200 g	4° C
Potential Hydrogen (pH)	EPA 150.1	plastic	NA	200 mL	4° C
Potential Hydrogen (pH)	SWEPA 9045C	plastic or glass	NA	50 g	4° C
Conductivity	EPA 120.1	plastic	NA	200 mL	4° C
Conductivity, slurry in DI water	SWEPA 9050A	plastic or glass	NA	50 g	4° C
Aluminum	EPA 200.7	plastic	6 months	1 Liter (L)	pH<2 with HNO <sub>3</sub> , 4° C
Aluminum	EPA 200.7	plastic	6 months	200 g	4° C

EPA = *Methods for Chemical Analysis of Water and Wastes*, March 1983

Water and solids samples digested for total recoverable metals per EPA 200.7, rev. 5.0. There is no difference between EPA methods 200.7 and 6010B. Method 200.7 is a newer version and will yield the same results.

SWEPA – *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846, 3rd Ed.)*, EPA, 1997

Solid samples digested for total recoverable metals per EPA 3050B.

SSSA= *Methods of Soil Analysis*, Part 3, Soil Science Society of America, 1996 (with section and page number)

SM = *Standard Methods for the Treatment of Water and Wastewater*; 2002

HNO<sub>3</sub> = Nitric acid

H<sub>2</sub>SO<sub>4</sub> = Sulfuric acid

NA = not applicable

**Table B3-2. SWFTL Sample Procedures and Handling Methods**

<b>Laboratory Parameters</b>	<b>SWFTL<sup>a</sup> Code</b>	<b>Container</b>	<b>Preservation</b>	<b>Holding Time</b>
<b>Soil</b>				
Total N	0073 & 0068	TCE D-592 <sup>b</sup>		90 days
Total P	0011	TCE D-592		90 days
pH	0015	TCE D-592		90 days
Extractable P	0079 & 0081	TCE D-592		90 days
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	0014	TCE D-592		90 days
NH <sub>4</sub> -N	0068	TCE D-592		90 days
Water soluble P	0064 & 0037	TCE D-592		90 days
Potassium	0079 & 0081	TCE D-592		90 days
Calcium	0079 & 0081	TCE D-592		90 days
Magnesium	0079 & 0081	TCE D-592		90 days
Sodium	0079 & 0081	TCE D-592		90 days
Aluminum		TCE D-592		90 days
Organic Carbon	0055	TCE D-592		90 days
<b>Water</b>				
Total N	0073	HDPE <sup>c</sup>	4°C	7 days
Total P	0037	HDPE	4°C	7 days
pH	0041	HDPE	4°C	24 hours
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	0038	HDPE	4°C	48 hours
NH <sub>4</sub> -N	0068	HDPE	4°C	48 hours
Water soluble P	0064 & 0037	HDPE	4°C	72 hours
Potassium	0037	HDPE	4°C, HNO <sub>3</sub>	28 days
Calcium	0037	HDPE	4°C, HNO <sub>3</sub>	28 days
Magnesium	0037	HDPE	4°C, HNO <sub>3</sub>	28 days
Sodium	0037	HDPE	4°C, HNO <sub>3</sub>	28 days
<b>Geotube residue</b>				
Total N	0073 & 0068	TCE - 592		90 days
Total P	0035 & 0011	TCE - 592		90 days
pH	0015	TCE -592		90 days
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	0014	TCE -592		90 days
NH <sub>4</sub> -N	0068	TCE -592		90 days
Water soluble P	0064 & 0037	TCE -592		90 days
Potassium	0035 & 0074	TCE -592		90 days
Calcium	0035 & 0074	TCE -592		90 days
Magnesium	0035 & 0074	TCE -592		90 days
Sodium	0035 & 0074	TCE -592		90 days
Copper	0035 & 0074	TCE -592		90 days
Aluminum		TCE -592		90 days
Organic Carbon	0055	TCE -592		90 days

Zinc	0035 & 0074	TCE -592		90 days
Iron	0035 & 0074	TCE -592		90 days
Manganese	0035 & 0074	TCE -592		90 days
<b><i>Turfgrass</i></b>				
Total N	0073 & 0068	DFSB <sup>d</sup>		90 days
Total P	0035 & 0011	DFSB		90 days
Potassium	0035 & 0074	DFSB		90 days
Calcium	0035 & 0074	DFSB		90 days
Magnesium	0035 & 0074	DFSB		90 days
Sodium	0035 & 0074	DFSB		90 days
Aluminum		DFSB		90 days
Organic Carbon	0055	DFSB		90 days
Zinc	0035 & 0054	DFSB		90 days
Iron	0035 & 0054	DFSB		90 days
Manganese	0035 & 0054	DFSB		90 days
Copper	0035 & 0054	DFSB		90 days

<sup>a</sup>SWFTL Code = Soil, Water, and Forage Testing Laboratory SOP code

<sup>b</sup> D592 = TCE soil sampling bags    <sup>c</sup> HDPE = High density polyethylene bottles

<sup>d</sup>DFSB = Disposable Forage Sample Bag

**Table B3-3. Dr. Victor's Lab Sample Procedures and Handling Methods**

Laboratory Parameters	SWFTL <sup>a</sup> Code	Container	Preservation	Holding Time
<b><i>Soil</i></b>				
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	0014	TCE D-592		90 days
NH <sub>4</sub> -N	0068	TCE D-592		90 days
Soluble reactive P	0061 & 0062	TCE D-592		90 days
Bulk Density		TCE D-592		90 days
Water content		TCE D-592		24 hours
<b><i>Water</i></b>				
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	0038	HDPE	4°C	48 hours
NH <sub>4</sub> -N	0068	HDPE	4°C	48 hours
Soluble reactive P	0061 & 0062	HDPE	4°C	24 hours
Dissolved organic C		HDPE	4°C	72 hours
<b><i>Geotube residue</i></b>				
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	0014	TCE -592		90 days
NH <sub>4</sub> -N	0068	TCE -592		90 days
Soluble reactive P	0061 & 0062	HDPE	4°C	24 hours

**Failures in Chain-of-Custody and Corrective Action**

All failures associated with chain-of-custody procedures, as described in this QAPP, are immediately reported to the Project Leader. These include such items as delays in transfer, resulting in holding time violations; violations of sample preservation requirements; incomplete documentation, including signatures; possible tampering of samples; broken or spilled samples, etc.

The Project Leader in conjunction with the TWRI QAO will determine if the procedural violation may have compromised the validity of the resulting data. Any failures that potentially compromise data validity will invalidate data, and the sampling event should be repeated. The resolution of the situation will be reported to the TSSWCB in the quarterly progress report. Corrective action reports will be maintained by the Project Leader.

**Section B4: Analytical Methods Requirements**

The parameters listed in Table B3-2 will be analyzed by SWFTL at College Station, Texas. A listing of analytical methods and equipment is provided in Table B4-1. The parameters listed in Table B3-3 will be analyzed by Dr. Vietor's laboratory at Texas A&M University in College Station, Texas. A listing of analytical methods and equipment is provided in Table B4-2 and B4-3. Standard operating procedures have been established for all procedures undertaken by staff that concerns sample monitoring and analysis, and copies of the TIAER and SWFTL SOPs are available upon request.

**Table B4-1. TIAER Laboratory Analytical Methods**

Parameter	Method	Equipment Used
Nitrite+Nitrate Nitrogen	EPA 353.2 and SSSA 38-1148	Perstorp® or Lachat® QuickChem Autoanalyzer
Total Kjeldahl Nitrogen	EPA 353.2, modified	Perstorp® or Lachat® QuickChem Autoanalyzer
Potassium	EPA 200.7	Spectro ® ICP
Calcium	EPA 200.7	Spectro ® ICP
Magnesium	EPA 200.7	Spectro ® ICP
Sodium	EPA 200.7	Spectro ® ICP
Manganese	EPA 200.7	Spectro ® ICP
Iron	EPA 200.7	Spectro ® ICP
Copper	EPA 200.7	Spectro ® ICP
Orthophosphate Phosphorus	EPA 365.2	Beckman® DU 640 Spectrophotometer
Soluble Phosphorus	SSSA 32-891	Beckman® DU 640 Spectrophotometer
Total Phosphorus	EPA 365.4, modified	Perstorp® or Lachat® QuickChem Autoanalyzer
Total Suspended Solids	EPA 160.2	Sartorius® AC210P or Mettler® AT261 analytical balance, oven
Percent Solids	SM 2540B	Sartorius® AC210P or Mettler® AT261 analytical balance, oven
Total Solids	SM 2540C	Sartorius® AC210P or Mettler® AT261 analytical balance, oven
Total Volatile Solids	SM 2450G	Sartorius® AC210P or Mettler® AT261 analytical balance, oven, muffle furnace
Volatile Solids	EPA 160.4	Sartorius® AC210P or Mettler® AT261 analytical balance, oven, muffle furnace
Potential Hydrogen	EPA 150.1 and EPA 9045A	Accument® AB15 Plus pH meter
Conductivity	EPA 120.1 and EPA 9050A	YSI® 3200 conductivity meter
Aluminum	EPA 200.7	Spectro ® ICP

EPA = Methods for Chemical Analysis of Water and Wastes, March 1983 and version 2, June 1999.  
There is no difference between EPA methods 200.7 and 6010B. Method 200.7 is a newer version and will yield the same results.

**Table B4-2. SWFTL Laboratory Analytical Methods**

<b>Parameter</b>	<b>Method</b>	<b>Equipment Used</b>
<b><i>Soil</i></b>		
Total N	0073 & 0068	Elementar Vario Max CN Analyzer
Total P	0011	Spectro Radial Modula ICP
pH	0015	Orion Model 410A pH Meter
Extractable P	0079 & 0081	Spectro Radial Modula ICP
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	0014	FIA Labs 2500 Flow Injection Analyzer
NH <sub>4</sub> -N	0068	Technicon Autoanalyzer II
Water soluble P	0064 & 0037	Spectro Radial Modula ICP
Potassium	0079 & 0081	Spectro Radial Modula ICP
Calcium	0079 & 0081	Spectro Radial Modula ICP
Magnesium	0079 & 0081	Spectro Radial Modula ICP
Sodium	0079 & 0081	Spectro Radial Modula ICP
Aluminum		Spectro Radial Modula ICP
Organic Carbon	0055	Elementar Vario Max CN
<b><i>Water(Leachate)</i></b>		
Total N	0073	Technicon autoanalyzer II
Total P	0037	Spectro Radial Modula ICP
pH	0041	Orion Model 410A pH Meter
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	0038	FIA Labs 2500 Flow Injection Analyzer
NH <sub>4</sub> -N	0068	Technicon Autoanalyzer II
Water soluble P	0064 & 0037	Spectro Axial Ciros ICP
Potassium	0037	Spectro Axial Ciros ICP
Calcium	0037	Spectro Axial Ciros ICP
Magnesium	0037	Spectro Axial Ciros ICP
Sodium	0037	Spectro Axial Ciros ICP
<b><i>Geotube residue</i></b>		
Total N	0073 & 0068	Elementar Vario Max CN Analyzer
Total P	0035 & 0011	Spectro Radial Modula ICP
pH	0015	Orion Model 410A pH Meter
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	0014	FIA Labs 2500 Flow Injection Analyzer
NH <sub>4</sub> -N	0068	Technicon Autoanalyzer II
Water soluble P	0064 & 0037	Spectro Radial Modula ICP
Potassium	0035 & 0074	Spectro Radial Modula ICP
Calcium	0035 & 0074	Spectro Radial Modula ICP
Magnesium	0035 & 0074	Spectro Radial Modula ICP
Sodium	0035 & 0074	Spectro Radial Modula ICP
Aluminum		Spectro Radial Modula ICP

Organic carbon	0055	Elementar Vario Max CN
Zinc	0035 & 0074	Spectro Radial Modula ICP
Iron	0035 & 0074	Spectro Radial Modula ICP
Manganese	0035 & 0074	Spectro Radial Modula ICP
Copper	0035 & 0074	Spectro Radial Modula ICP
<b><i>Turfgrass</i></b>		
Total N	0073 & 0068	Elementar Rapid N III
Total P	0035 & 0011	Spectro Axial Ciros ICP
Potassium	0035 & 0074	Spectro Axial Ciros ICP
Calcium	0035 & 0074	Spectro Axial Ciros ICP
Magnesium	0035 & 0074	Spectro Axial Ciros ICP
Sodium	0035 & 0074	Spectro Axial Ciros ICP
Aluminum		Spectro Axial Ciros ICP
Organic carbon	0055	Elementar Vario Max CN
Zinc	0035 & 0054	Spectro Axial Ciros ICP
Iron	0035 & 0054	Spectro Axial Ciros ICP
Manganese	0035 & 0054	Spectro Axial Ciros ICP
Copper	0035 & 0054	Spectro Axial Ciros ICP

<sup>a</sup>SWFTL Code = Soil, Water, and Forage Testing Laboratory SOP code

**Table B4-3. Dr. Viator's Laboratory Analytical Methods**

Parameter	Method	Equipment Used
<b><i>Soil</i></b>		
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	Lopez & Vargas-Albores	DynaTech MRX Microplate Reader
NH <sub>4</sub> -N	Lopez & Vargas-Albores	DynaTech MRX Microplate Reader
Soluble reactive P	Murphy & Riley	DynaTech MRX Microplate Reader
<b><i>Water(Leachate)</i></b>		
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	Lopez & Vargas-Albores	DynaTech MRX Microplate Reader
NH <sub>4</sub> -N	Lopez & Vargas-Albores	DynaTech MRX Microplate Reader
Soluble reactive P	Murphy & Riley	DynaTech MRX Microplate Reader
Dissolved organic C		OI Analytical Model 700 Organic C analyzer
<b><i>Geotube residue</i></b>		
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	Lopez & Vargas-Albores	DynaTech MRX Microplate Reader
NH <sub>4</sub> -N	Lopez & Vargas-Albores	DynaTech MRX Microplate Reader
Soluble reactive P	Murphy & Riley	DynaTech MRX Microplate Reader

**Failures in Measurement Systems and Corrective Actions**

In the event of a failure in the analytical system, the Project Leader will be notified. Failures in laboratory measurement systems involve, but are not limited to such things as instrument malfunctions, failures in calibration, blank contamination, quality control samples outside QAPP defined limits, etc. In many cases, lab analyst will be able to correct the problem. If the problem is resolvable by the laboratory manager or lab analyst, then they will document the problem on the laboratory record, or CAR and complete the analysis. If the problem is not resolvable, then the Laboratory Manager (and their QAO, if applicable) of the affected Lab, in conjunction with the Project Leader and TWRI QAO will then determine if the existing sample integrity is intact, if re-sampling can and should be done, or if the data should be omitted. The situation and agreed resolution will be reported to the TSSWCB in the quarterly progress report.

## **Section B5: Quality Control Requirements**

The use of approved sampling and analytical methods will ensure that measured data accurately represent conditions at each monitoring site. 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.

TAMU Biological and Agricultural Engineering Department sampling site audits, and quality assurance of field sampling methods will be conducted by the TWRI QAO. In addition, laboratory audits, sampling site audits, and quality assurance of field sampling methods will be conducted by the TSSWCB QAO or their designee.

It is the responsibility of the Project Leader to verify that the data are representative. The chemistry data's precision, accuracy, and comparability will be the responsibility of the Laboratory Manager. The Project Leader has the responsibility of determining that the 90 percent completeness criteria is met, or will justify acceptance of a lesser percentage. All incidents requiring corrective action will be documented through use of Corrective Action Reports (Appendix A). Corrective action reports will be maintained by the Project Leader and the TSSWCB PM.

The TIAER laboratory practices new quality control guidelines, as set forth by the *TCEQ Surface Water Quality Monitoring Procedures Volume 1: Physical and Chemical Monitoring Methods for Water, Sediment and Tissue* (September 2003), for all water quality data collection and verification. TIAER laboratory quality control procedures are explained below under their respective headings.

### **TIAER Sampling Quality Control Requirements and Acceptability Criteria**

Field splits - A field split is a single sample subdivided by field staff immediately following collection and submitted to the laboratory as two separate, identified samples according to procedures specified in the SWQM Procedures Manual. Split samples are preserved, handled, shipped, and analyzed identically and are used to assess variability in all of these processes. Field splits apply to conventional samples only and are collected on a 10% basis or one per batch whichever is greater. The precision of field split results is calculated by relative percent difference (RPD) using the following equation:

$$\text{RPD} = \{ (X_1 - X_2) / (X_1 + X_2) / 2 \} * 100$$

A 30% RPD criteria will be used to screen field split results as a possible indicator of excessive variability in the collection and analytical system. If it is determined that meaningful quantities of constituent (> RL) were measured and analytical variability can be eliminated as a factor, then variability in field split results will primarily be used as a trigger for discussion with field staff to ensure samples are being handled correctly in the field.

Some sample results or batches of samples may be invalidated based on the examination of all extenuating information. Professional judgment during data validation will be relied upon to interpret the results and take appropriate action. The qualification (i.e., invalidation) of data will be documented on the Data Summary. Deficiencies will be addressed as specified in this section under Failures in Quality Control and Corrective Action.

### **TIAER Laboratory Measurement Quality Control Requirements and Acceptability Criteria**

Detailed laboratory QC requirements are contained within each individual method and laboratory quality assurance manuals (QAMs). Lab QC sample results are reported with the laboratory data report.

Laboratory duplicate - Laboratory duplicates are used to assess precision. A laboratory duplicate is prepared by splitting aliquots of a single sample (or a matrix spike or a laboratory control standard) in the laboratory. Both samples are carried through the entire preparation and analytical process. Laboratory duplicates are performed at a rate of one per batch. They are used to assess precision.

Precision is calculated by the relative percent deviation (RPD) of duplicate results as defined by 100 times the difference (range) of each duplicate set, divided by the average value (mean) of the set. For duplicate results,  $X_1$  and  $X_2$ , RPD is calculated from the following equation:

$$\text{RPD} = \{ (X_1 - X_2) / (X_1 + X_2) / 2 \} * 100$$

Performance limits and control charts are used to determine the acceptability of duplicate analyses.

Laboratory Control Standard (LCS)/Laboratory Control Standard Duplicate (LCSD) - LCS/LCSD pairs are analyte-free water samples spiked with the analyte of interest prepared from standardized reference material. The LCS/LCSD pairs are generally spiked into laboratory pure water at a level less than or equal to the mid-point of the calibration curve for each analyte. They are carried through the complete preparation and analytical process. The LCS/LCSD pairs are used to document the bias of the method due to the analytical process. Bias can be assessed by measuring the percent recovery of LCSs and LCSDs, and precision can be assessed by comparing the results of LCS/LCSD pairs. An LCS/LCSD pair is prepared (e.g., distilled, extracted) along with 20 samples or once per day, whichever is greater. If no preparation is required for a test, one LCS/LCSD pair must be analyzed per day. Acceptability criteria for bias are laboratory specific and usually based on results of past laboratory data (i.e., control charts). Precision and bias criteria for LCS/LCSD pairs are specified in Table A7.1. Laboratory-specific control limits and charts are calculated and maintained by laboratory staff on a periodic basis.

Bias of LCSs and LCSDs is expressed by percent recovery (%R) where SR is the observed spiked sample concentration, and SA is the spike added:

$$\%R = SR/SA * 100$$

The mean bias of LCS/LCSD pairs is expressed by %R<sub>mean</sub>, where %R<sub>LCS</sub> is the percent recovery of the LCS and %R<sub>LCSD</sub> is the percent recovery of the LCSD:

$$\%R_{\text{mean}} = (\%R_{\text{LCS}} + \%R_{\text{LCSD}})/2$$

Precision between LCS/LCSD pairs is expressed by RPD. For LCS/LCSD results, X<sub>1</sub> and X<sub>2</sub>, the RPD is calculated from the following equation:

$$\text{RPD} = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} * 100$$

Matrix spikes (MS) - A matrix spike is an aliquot of sample spiked with a known concentration of the analyte of interest. Percent recovery of the known concentration of added analyte is used to assess accuracy of the analytical process. The spiking occurs prior to sample preparation and analysis. Matrix spike samples are routinely prepared and analyzed at a rate of 10% of samples processed or one per batch whichever is greater. The MS may be spiked at a level less than or equal to the midpoint of the calibration or analysis range for each analyte. The MS is used to document the accuracy of a method due to sample matrix and not to control the analytical process. Percent Recovery (%R) is defined as 100 times the observed concentration, minus the sample concentration, divided by the true concentration of the spike. MS recoveries are indicative of matrix-specific biases and are plotted on control charts maintained by the laboratory. Measurement performance specifications for matrix spikes are not specified in this document, and MS data should be evaluated on a case-by-case basis.

The formula used to calculate percent recovery, where %R is percent recovery; SSR is the observed spiked sample concentration; SR is the sample concentration; and, SA is the spike added; is:

$$\%R = (SSR - SR)/SA * 100$$

AWRL /Reporting Limit Verification - The laboratory's reporting limit will be at or below the AWRL. To demonstrate ongoing ability to recover at the reporting limit, the laboratory will analyze a calibration standard (if applicable) at or below the reporting limit on each day samples are analyzed. Two acceptance criteria will be met or corrective action will be implemented. First, calibrations including the standard at the reporting limit will meet the calibration requirements of the analytical method. Second, the instrument response (e.g., absorbency, peak area, etc.) for the standard at the reporting limit will be treated as a response for a sample by use of the calibration equation (e.g., regression curve, etc.) in calculating an apparent concentration of the standard.

The calculated and reference concentrations for the standard will then be used to calculate percent recovery (%R) at the reporting limit using the equation:

$$\%R = CR/SA * 100$$

where CR is the calculated result and SA is the actual or reference concentration for the standard. Recoveries must be within 75-125% of the reference concentration.

When daily calibration is not required (e.g., EPA Method 624), or a method does not use a calibration curve to calculate results, the laboratory will analyze a check standard at the reporting limit on each day samples are analyzed. The check standard does not have to be taken through sample preparation, but must be recovered within 75-125% of the reference concentration for the standard. The percent recovery of the check standard is calculated using the following equation in which %R is percent recovery, SR is the sample result, and SA is the reference concentration for the check standard:

$$\%R = SR/SA * 100$$

If the calibration (when applicable) or the recovery of the calibration or control standard is not acceptable, corrective actions (e.g., re-calibration) will be taken to meet the specifications before proceeding with analyses of samples.

Method Blank- A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing and analyzed with each batch. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination from the analytical process. The analysis of method blanks should yield values less than the laboratory's reporting limit. Method blanks exceeding the RL will be evaluated on a case-by-case basis to determine possible contamination sources, but will not necessarily invalidate sample results. A CAR will be completed to document the exceedence and corrective actions. The CAR information, conclusions, corrective actions, and affected tag numbers will be included with data submittal to TSSWCB. For very high level analyses, blank value should be less than 5% of the lowest value of the batch or corrective action will be implemented.

Additional method specific QC requirements - Additional QC samples are run (e.g., surrogates, internal standards, continuing calibration samples, interference check samples) as specified in the methods. The requirements for these samples, their acceptance criteria, and corrective action are method-specific.

#### **Laboratory Measurement Quality Control Requirements and Acceptability Criteria for SWFTL and Dr. Vietor's laboratories.**

Detailed laboratory QC requirements are contained within each individual method and laboratory QAMs. Lab QC sample results are reported with the laboratory data report.

Field Duplicate – A field duplicate is defined as a second sample from the same location, collected in immediate succession, using identical techniques. This applies to all sample types, soil, water, vegetation and compost. One duplicate sample will be taken during each sampling event. For soil, the duplicate sample will be collected by selecting one of the depth increments in one treatment, collecting two times as much sample as needed for lab analyses, mixing the sample in the field, and submitting the sample under a different numbers. For leachate samples, the duplicate sample will be collected by selecting one treatment for each type of sample, collecting two times as much sample as needed for lab analysis, thoroughly mixing the sample in the field and submitting the sample under a different number. The duplicate sample will be labeled, tracked, and analyzed under a different number.

Duplicate samples are sealed, handled, stored, shipped, and analyzed in the same manner as the primary sample. Precision of duplicate results for most parameters is calculated by the RPD as defined by 100 times the difference (range) of each duplicate set, divided by the average value (mean) of the set. For duplicate results,  $X_1$  and  $X_2$ , the RPD is calculated using the following equation:

$$RPD = (X_1 - X_2) / \{(X_1 + X_2) / 2\} * 100$$

Performance limits are used to determine the acceptability of field duplicate analyses.

Laboratory Matrix Blank – A laboratory matrix blank is a sample that does not contain the specific media but rather the reagent matrix of the procedure and is used to determine the bias and precision of laboratory instrument analytical abilities. The laboratory matrix blank is run through the exact same procedure as the blind samples and its results are compared to the lowest calibration point of the calibration standard curve. If a laboratory matrix blank is greater than the reporting limit, then data for the ‘batch’ is rejected and samples are reanalyzed in a new ‘batch’.

For soil and water, a laboratory matrix blank is included with each ‘batch’ of samples, which is at most every 30 samples.

Laboratory Media Standard – For water, a laboratory media standard is a sample of known concentration used to determine bias and percent recovery for the laboratory procedures and analytical methods. For soil, a laboratory media standard is a sample with a known statistical mean value that was determined through replicated analysis over time.

For soil, the laboratory media standard is obtained by collecting a large volume of one soil and replicating analyses on the soil sample. For water, National Institute for Standards and Testing (NIST) traceable standards, which are manufactured by a company that has verified standard results against NIST standards are used to create laboratory media standards within deionized water.

For soil and water, a laboratory media standard is included with each ‘batch’ of samples, which is at most every 30 samples. Using the historical average of the replicated results for soil and the reported results for water, the laboratory assesses bias and percent recovery from the individual laboratory media standards run during each batch of sample analysis.

Calibration Standards – All instruments or devices used in obtaining environmental measurement data will be calibrated prior to use. Laboratory equipment and devices needing calibration and recalibration are numerous and varied. Thus, each instrument has a specialized procedure for calibration and a specific type of standard used to verify calibration. Generally, calibrations are performed with a minimum of four standards of increasing concentrations and a calibration blank. Instrument calibration for each analyte will achieve an  $r^2$  value of 0.990 or higher. The frequency of calibration recommended by the equipment manufacturer or as stated in the SOPs, as well as any instructions specified by applicable analytical methods, will be followed. Calibration shall be verified immediately after a set of standards is analyzed and continuously throughout an analytical run, after every sample batch, and at the end of an analysis to verify that the instrument or method has not drifted or changed since calibration. The initial calibration verification (ICV) and continuing calibration verification (CCV) will be matched to the generated standard curve and screened for acceptability. If an ICV or CCV fails, the instrument is recalibrated and all samples are rerun since the last passing ICV or CCV. Laboratory standards will be checked to verify that the concentrations are those which are prescribed for the analytical method. 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 laboratory will meet or exceed the calibration frequencies published in the test methods used for this project. Additional calibration procedures will 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-A and Table B4-B.

#### **TIAER, SWFTL, and Dr. Vietor's laboratory Failures in Quality Control and Corrective Action**

In that differences in field split sample results are used to assess the entire sampling process, the arbitrary rejection of results based on pre-determined limits is not practical. Therefore, the professional judgment of the TIAER, SWFTL, and TAMU Program Managers, Laboratory Managers, and QAOs will be relied upon in evaluating results. Rejecting sample results based on wide variability is a possibility. Notations of field split excursions and blank contamination are noted in the quarterly report to TWRI and in the final QC Report.

Corrective action will involve identification of the cause of the failure where possible. Response actions will typically include re-analysis of questionable samples. In some cases, TIAER, SWFTL, or Dr. Vietor's lab may require the TAMU Bio. /Ag. Engineering Department field technician to have a site re-sampled in order to achieve project goals.

Laboratory measurement quality control failures are evaluated by the laboratory staff. The dispositions of such failures and conveyance to the TSSWCB are discussed in Section B4 under Failures in Management Systems and Corrective Actions.

**Deficiencies, Nonconformances and Corrective Action Related to Quality Control**

Deficiencies are defined as unauthorized deviations from procedures documented in the QAPP. Nonconformances are deficiencies, which affect quality and render the data unacceptable or indeterminate. Deficiencies related to quality control include but are not limited to field and laboratory quality control sample failures.

The TWRI Project Leader, in consultation with the TWRI QAO, will determine if the deficiency constitutes a nonconformance of the QAPP. If it is determined the activity or item in question does not affect data quality and therefore, is not a valid nonconformance, it will be added as part of the normal quarterly reporting requirement with no other documentation needed. If it is determined a nonconformance does exist, the TWRI Project Leader, in consultation with the TWRI QAO and pertinent field or laboratory personnel, will determine the disposition of the nonconforming activity or item and necessary corrective action(s). Results will be documented by the TWRI QAO, Project Leader and field technician, or the TIAER or SWFTL QAO and Laboratory Manager as appropriate, by completion of a Corrective Action Report.

CARs document: root cause(s); impact(s); specific corrective action(s) to address the deficiency; action(s) to prevent recurrence; individual(s) responsible for each action; the timetable for completion of each action; and, the means by which completion of each corrective action will be documented. CARs will be included with quarterly progress reports. In addition, significant conditions (i.e., situations which, if uncorrected, could have a serious effect on safety or on the validity or integrity of data) will be reported to the TSSWCB immediately, both verbally and in writing.

### **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 (see Table B4-1 for list of equipment). All laboratory tool, gauge, instrument, and equipment testing and maintenance requirements are contained within laboratory SOPs. Records of all tests, inspections, and maintenance will be maintained and log sheets kept showing time, date, and analyst signature. These records will be available for inspection by the TSSWCB.

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, where possible, in the case that a piece of equipment fails during use, so measurement tasks may 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.

To minimize downtime of all measurement systems, spare parts for laboratory equipment will be kept in the TIAER, SWFTL, and Dr. Vietor's laboratories and will be maintained in a working condition. All field and laboratory equipment will be tested, maintained, and inspected in accordance with manufacturer's instructions and recommendation in Standard Methods for the Examination of Water and Wastewater, 20<sup>th</sup> Edition. Maintenance and inspection logs will be kept on each piece of laboratory equipment.

Failures in any testing, inspections, or calibration of equipment will result in a CAR and resolution of the situation will be reported to the TSSWCB in the quarterly report. Copies of corrective action reports will be maintained by the Project Leader.

## **Section B7: Instrument Calibration and Frequency**

All instruments or devices used in obtaining environmental measurement data will be used according to appropriate laboratory or field practices (see Tables B4-1, B4-2, and B4-3 for lists of equipment). Written copies of standard operating procedures in TIAER, SWFTL and Dr. Viotor's laboratories will be available for review upon request.

Standards used for instrument or method calibrations shall be of known purity and be 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 calibration 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, after every sample batch, and at the end of an analysis to verify that the instrument or method has not drifted or changed since calibration. The initial calibration verification and continuing calibration verification will be matched to the generated standard curve and screened for acceptability. 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 environmental measurement 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. 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 a field audit.

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 Tables B4-1 and B4-2 in Section B-4 "Analytical Methods Requirements." All instruments that require calibration prior to use will be calibrated before each day's analysis. Calibration is normally performed with a 5 point standard curve. The analytical balance for TSS requires no calibration other than class "S" weights to check the balance

**Section B8: Inspection/Acceptance Requirements for Supplies and Consumables**

All supplies and consumables received by the TIAER, SWFTL, and Dr. Vietor's laboratories 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, initialed by staff member and marked with receipt date. Volumetric glassware is inspected to ensure class "A" classification, where required. All supplies will be stored as per manufacturer labeling and discarded past expiration date.

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, then rinsed at least three times in type II ASTM (American Society for Testing and Materials) water, i.e., water with conductivity of less than 2 microsiemens per centimeter. No phosphate-based detergents are used in the cleaning process. The hydrochloric acid (HCl) is used only once and is rinsed down the drain after neutralization or dilution with the tap water. For certain analyses, cleaning with solvents and oven drying may be required. Glassware is never rinsed with compounds of the constituent being analyzed.

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

Water quality determinations at sampling sites will be based upon data collected during the time frame of this project. However, data collected within the Bosque River or Leon River watersheds, under approved QAPPs, from other state or federal projects will be used as supplemental information to meet data quality objectives (see Section A7). Data collected at sites along the North Bosque River and provided to TCEQ's TRACS Database will be used to assess the impact of potential reductions, from new technology strategies, on the water quality of the North Bosque River. The data collected under approved QAPPs from other projects will be referred to as historical data.

## **Section B10: Data Management**

### **Field Collection and Management of Routine Samples**

Field staff will visit sampling sites and experiments on scheduled dates to collect samples. Site identification, date and time, personnel, and any comments concerning weather or conditions at the site are noted on a field data sheet. One field data sheet is filled out in the field for each site visited. An example of a field data sheet is shown in Appendix C.

Samples are collected at the field site or experiment and an identification number (either a sample identification number or a site code) is written in marker on the outside of the plastic/HDPE sample bottles or soil sampling bags. The samples are then placed in an iced chest for transportation to the TIAER, SWFTL, or Dr. Vietor's laboratory. The samples are filtered and/or acidified or weighed wet or dry as appropriate, according the particular variable (s) of interest for the collection period (*see* Tables B1-1, B3-1, B3-2, and B3-3). Samples are refrigerated, frozen, or stored on laboratory shelves, as appropriate for the variable being measured, until analysis in the SWFTL or Dr. Vietor's laboratory.

Sample ID numbers are recorded on the COC forms. Sample bottles or bags being processed are typically placed in order of collection time, so the order of the sample bottles matches the order of the field data and the COC sample ID numbers, reducing transcription errors. Site name, time of collection, comments, and other pertinent data are copied from the field data sheets to the COC. The COC and accompanying sample bottles or bags are submitted to laboratory analysts, with relinquishing and receiving personnel both signing and dating the COC. A copy of a blank COC form used on this project is included as Appendix B.

### **Chain of Custody Forms**

A chain of custody (COC) form is used to record water sample identification parameters and to document the submission of samples from the field staff to the analytical laboratory staff or from the initiating laboratory to a sub-contracted laboratory. Each COC has space to record data for at least 15 separate samples. 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 will be kept in three-ring binders in the TIAER, SWFTL, and Dr. Vietor's lab offices. The Project leader will also keep a copy of the COCs at the TAMU Biological and Agricultural Engineering Department at Texas A&M University for at least five years.

### **Laboratory Analysis and Data Collection**

Aliquots of each sample are used by the laboratory staff in running the various analytical procedures. The sample number is marked on all containers to which aliquots are transferred. Aliquots are filtered, as necessary, and analyzed as per standard operating procedures. Data pertaining to analyte measurements are recorded in bound personal logbooks, which are specific to each procedure and analyst. Measurement data are entered into the water quality database from the laboratory notebooks by laboratory personnel.

### **Water Quality Data Entry**

All COC, field, and laboratory information will be entered into spreadsheet software. Field data and COC information will be verified by the Project Leader. The Project Leader will review site names, appropriateness of data values, completeness of data, dates and times, bottle numbers, comments and all other data within the Access data table. Any questions or abnormalities will be investigated, relying largely on field data and general maintenance records, field technicians, laboratory notebooks, and laboratory personnel. As appropriate, corrections will be made to the spreadsheet table with appropriate documentation maintained.

### **Systems Design**

TAMU- Bio. /Ag. Engineering Department, TWRI, TIAER, SWFTL, and Dr. Vietor's lab will use laptop personal computers and desktop personal computers. The computers run Windows operating system and databases include Microsoft® Excel, Microsoft® Access database, and a SAS database management system. The TIAER, SWFTL, and Dr. Vietor's analytical laboratories collect data using a variety of automated equipment.

### **Backup and Disaster Recovery**

The network server is backed up daily to a tape drive. In the event of a catastrophic systems failure, the tapes can be used to restore the data in less than one day's time. Data generated on the day of the failure may be lost, but can be reproduced from raw data in most cases.

### **Archives and Data Retention**

Original data recorded on paper files are stored for at least five years. Data in electronic format are stored on tape drives in a climate controlled, fire-resistant storage area on the Texas A&M University campus.

### **Information Dissemination**

TIAER, SWFTL, and Dr. Vietor's data for this project will be sent to the Project Leader at TAMU-Bio./Ag. Engineering Department. Summaries of the data will be presented to TSSWCB in the final project report.

## Section C1: Assessments and Response Actions

The commitment to use approved equipment and approved methods when obtaining environmental samples and when producing field or laboratory measurements requires periodic verification that the equipment and methods are, in fact, being employed and being employed properly. This verification will be provided through a field and laboratory performance audit performed by the TSSWCB QA officer or contracted entity. 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.

**Table C.1-1. Assessments and Response Actions**

Assessment Activity	Approximate Schedule	Responsible Party(ies)	Scope	Response Requirements
Status Monitoring Oversight, etc.	Continuous	TWRI, TAMU- Bio. /Ag. Engineering Department.	Monitoring of the project status and records to ensure requirements are being fulfilled. Monitoring and review of contract laboratory performance and data quality	TAMU- Bio. /Ag. Engineering Department and TWRI will report to TSSWCB PM via quarterly report.
Laboratory Inspections	Minimum of one during the course of this project.	SWFTL-QAO, TWRI-QAO, and TSSWCB QAO	Analytical and QC procedures employed at the laboratory and in the field.	TAMU- Bio. /Ag. Engineering Department has 30 days to respond in writing to the TSSWCB QAO to address corrective actions
Monitoring Systems Audit	Minimum of one during the course of this project.	TSSWCB QAO	The assessment will be tailored in accordance with objectives needed to assure compliance with the QAPP. Field sampling, handling and measurement; facility review; and data management as they relate to the project	TAMU- Bio. /Ag. Engineering Department has 30 days to respond in writing to the TSSWCB QAO to address corrective actions

All laboratory analyses will have the precision and accuracy of data determined on the particular day that the data were generated. The specific requirements are presented in Section B5 of the QAPP.

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.

Backup equipment or common spare parts will be made available, where possible, in the case that a piece of equipment fails during use, so that measurement tasks may be resumed in a timely manner.

The Project Leader is responsible for implementing and tracking corrective action procedures as a result of audit findings. Records of audit findings and corrective actions are maintained by the Project Leader and the TSSWCB QAO.

If audit findings and corrective actions cannot be resolved, then the authority and responsibility for terminating work is specified in the TSSWCB QMP and in agreements or contracts between participating organizations.

## **Section C2: Reports to Management**

Quarterly progress reports will be generated by TWRI personnel and will note activities conducted in connection with the water quality monitoring program, items or areas identified as potential problems, and any variations or supplements to the QAPP. Corrective action report forms will be utilized when necessary (Appendix A). CARs that concern field operations will be maintained in an accessible location for reference at TAMU Bio. / Ag. Engineering Department. CARs that concern laboratory operations will be maintained in an accessible location for reference in TIAER, SWFTL, and Dr. Viotor's laboratories. CARs that result in any changes or variations from the QAPP will be made known to pertinent project personnel, documented in an update or amendment to the QAPP and distributed to personnel listed in Section A3.

The field and laboratory 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. Corrective Action Reports will be filled out to document the problems and the remedial action taken.

Copies of all Corrective action reports for this project will also be included with TWRI's TAMU-Bio. /Ag. Engineering Department's final report. The final report will contain a quality assurance section to address TIAER's, SWFTL's, Dr. Viotor's, and TAMU-Bio. /Ag. Engineering Department's accuracy, precision and completeness of the measurement data. The final report will also discuss any problems encountered and solutions made. The final report is the responsibility of the Project Leader and TWRI Quality Assurance Officer.

## **Section D1: Data Review, Validation and Verification**

For the purposes of this document, verification means the processes taken to determine compliance of data with project requirements, including documentation and technical criteria. Validation means those processes taken independently of the data-generation processes to determine the usability of data for its intended use(s). Integrity means the processes taken to assure that no falsified data will be reported.

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." Only those data that are supported by appropriate QC 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 Project Leader is responsible for ensuring that any pertinent field data is properly reviewed, verified, and submitted in the required format for the project database. The Laboratory Manager is responsible for ensuring that laboratory data are scientifically valid, defensible, of acceptable precision and accuracy, and reviewed for integrity. The data is then submitted to the Project Leader in the required format for the project database.

## **Section D2: Validation and Verification Methods**

All field and laboratory data will be reviewed, verified and validated to ensure they conform to project specifications and meet the conditions of end use as described in Section A7. A *Data Review Checklist* is included in Appendix D. The staff and management of the respective field, laboratory, and data management tasks, as listed in this project, are responsible for the integrity, validation and verification of the data each task generates or handles throughout each process. The field and laboratory tasks ensure the verification of raw data, electronically generated data, and data on chain-of-custody forms and hard copy output from instruments.

Microsoft Excel will be used for general spreadsheet computation and laboratory control charting of quality control parameters. The TIAER, SWFTL, and Dr. Vietor's laboratories will employ various data handling software on IBM compatible personal computer stations for data on many of the analyzed variables. Specific software and/or hardware handle data for other parameters. Orthophosphate phosphorus is analyzed on the Beckman DU-640 Spectrophotometer using Quant II Linear software at TIAER, on a Technicon Autoanalyzer at SWFTL, and a Dynatech MRX Microplate reader in Dr. Vietor's laboratory. Nitrite+nitrate-nitrogen is measured on a Lachat QuickChem Autoanalyzer at TIAER, a FIA Labs 2500 Flow Injection Analyzer at SWFTL, and a Dynatech MRX Microplate reader in Dr. Vietor's laboratory. Total Kjeldahl nitrogen data are generated on the Lachat QuickChem Autoanalyzer at TIAER and on a Elementar Vario Max CN Analyzer or Elementar Rapid N III Analyzer in the SWFTL. Macro and micro nutrient values, including total phosphorus will be analyzed using a Dionex ICP Autoanalyzer at TIAER and a Spectro Radial Modula ICP in the SWFTL. Dissolved organic C in leachate will be analyzed on an OI Analytical Model 700 total organic C analyzer.

The respective Laboratory Managers are responsible for review of calculations and charts made by these programs. Statistical analyses are performed with SAS programs. The Project Leader and TWRI QAO, as appropriate, are responsible for validating that the verified data are scientifically valid, defensible, of known precision, accuracy, integrity, meet the data quality objectives of the project, and are reportable to the TSSWCB.

### **Section D3: Reconciliation with Data Quality Objectives**

Respective Laboratory Managers shall be responsible for reviewing raw data produced by the TIAER, SWFTL, and Dr. Vietor's laboratories. The Laboratory Manager shall check calculations to verify that data are entered into the database correctly and be responsible for internal lab error corrections. Corrective Action Reports will be initiated in cases where invalid or incorrect data have been detected.

Representativeness and comparability of data, while unique to each individual collection site, is the responsibility of the Project Leader. 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 Leader will review the final data to ensure that it meets the requirements as described in this QAPP. Data that have been reviewed, verified, and validated will be summarized for each site individually, as well as all sites collectively, for their ability to meet the data quality objectives of the project and the informational needs of water quality agency decision-makers. These summaries will be included in the final report.

## References:

- APHA, American Public Health Association, American Water Works Association, and Water Environment Federation. 1992 & 1999. Standard Methods for the Examination of Water and Wastewater, 18th & 20<sup>th</sup> editions.
- Avila-Segura, M., J.W. Lyne, J.M. Meyer, and P. Barak. 2004. Rapid spectrophotometric analysis of soil phosphorus with a microplate reader. *Comm. Soil Sci. Plant Anal.* 35:547-557.
- EPA, United States Environmental Protection Agency. *Methods for Chemical Analysis of Water and Wastes*. March 1983. Washington, D.C.: EPA, Office of Water.
- Kleinman, P.J.A., A.N. Sharpley, Ann M. Wolf, D.B. Beegle, and P.A. Moore, Jr. Measuring Water-Extractable Phosphorus in Manure as an Indicator of Phosphorus in Runoff. *Soil Sci Soc Am J* 2002 66: 2009-2015.
- Lopez, J.H., and F. Vargas-Albores. 2003. A microplate technique to quantify nutrients ( $\text{NO}_2^-$ ,  $\text{NO}_3^-$ ,  $\text{NH}_4^+$ ,  $\text{PO}_4^{3-}$ ) in seawater. *Aquaculture Research* 35:1201-1204.
- Murphy, J., and J.P. Riley. 1962. A modified single solution method for determination of phosphate in natural waters. *Anal. Chim. Acta* 27:31-36.
- Ott, L. 1984. *An Introduction to Statistical Methods and Data Analysis*, 2nd Edition. Duxbury Press, Boston., Massachusetts.
- Sims, G.K., T.R. Ellsworth, and R.L. Mulvaney. 1995. Microscale determination of inorganic nitrogen in water and soil extracts. *Commun. Soil Sci. Plant Anal.* 26:303-316.
- Spooner, J., R.P. Maas, S.A. Dressing, M.D. Smolen, and F.J. Humenik. 1985. Appropriate Designs for Documenting Water Quality Improvements from Agricultural NPS Control Programs, pp. 30-34. In: *Perspectives on Nonpoint Source Pollution*. EPA 440/5-85-001.
- SCS, United States Department of Agriculture Soil Conservation Service. 1985. *National Engineering Handbook, Section 4 - Hydrology*. Engineering Division. Washington, D.C. Report No. SCS/ENG/NEH-4-2.
- TCE (Texas Cooperative Extension). August 2005. Quality assurance project plan for the Marketing Composted Manure to Public Entities project. Texas A&M University, College Station, Texas.
- TCEQ (Texas Commission on Environmental Quality). September 2003. *Surface Water Quality Monitoring Procedures Volume 1: Physical and Chemical Monitoring Methods for Water, Sediment and Tissue*. Austin, Texas.
- TCEQ (Texas Commission on Environmental Quality). September 2003. Clean Rivers Program (CRP) Quality Assurance Project Plan (QAPP) Shell for 2004-2005. Austin, Texas.
- TIAER, Texas Institute for Applied Environmental Research. 1993. Quality assurance project plan for the National Pilot Project. Tarleton State University, Stephenville, Texas.

TIAER, Texas Institute for Applied Environmental Research. 2002. Quality assurance project plan for Technical and Financial Assistance to Dairy Producers and Landowners of the North Bosque River Watershed within the Cross Timbers Soil and Water Conservation District and the Upper Leon Soil and Water Conservation District. Tarleton State University, Stephenville, Texas.

TIAER, Texas Institute for Applied Environmental Research. 1996, 1997-99 and 1999-2000. Quality assurance project plan for the United States Department of Agriculture Lake Waco-Bosque River Initiative. Tarleton State University, Stephenville, Texas.

TIAER, Texas Institute for Applied Environmental Research. 2003, 2004. Quality assurance project plan for the Upper Oyster Creek (Segment 1245) Dissolved Oxygen TMDL Project Quality Assurance Project Plan. Tarleton State University, Stephenville, Texas.

Ward, R.C., J.C. Loftis, H.P. DeLong, and H.F. Bell. 1988. Groundwater quality: A data analysis protocol. *Journal of the Water Pollution Control Federation* 60:1938-1945.

**Appendix A**  
**TIAER Corrective Action Report**  
SOP-Q-105

CAR #: \_\_\_\_\_

Report Initiation Date: \_\_\_\_\_ Reported by: \_\_\_\_\_ Sampling Station: \_\_\_\_\_

Analyte: \_\_\_\_\_ Procedure or QC Type : \_\_\_\_\_

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

Affected sample #s / date(s) of sample collection <sup>1</sup>: \_\_\_\_\_

Project(s): \_\_\_\_\_ Attached documentation: COC FDS SampLink Flow8 Logbook QC Table

Possible Causes: \_\_\_\_\_

Corrective Actions Taken: \_\_\_\_\_

Suggested Corrected Actions: \_\_\_\_\_

CAR routed to: \_\_\_\_\_ Date: \_\_\_\_\_

**Supervisor:** Circle one: **Tier 1** (does not affect final data integrity) **Tier 2** (data accepted but flag required<sup>2</sup>) **Tier 3** (possibly affects final data integrity)

Corrective actions taken for specific incident:

Corrective actions taken to prevent recurrences: \_\_\_\_\_

Corrective actions to be taken \_\_\_\_\_

Responsible Party<sup>3</sup> \_\_\_\_\_ Proposed completion date \_\_\_\_\_

Effect on data quality: \_\_\_\_\_

Responsible Supervisor: \_\_\_\_\_ Date: \_\_\_\_\_

**Concurrence:** Program/Project Manager: \_\_\_\_\_ Date: \_\_\_\_\_  
(Tier 3 CARs only)

Quality Assurance Officer: \_\_\_\_\_ Date: \_\_\_\_\_

<sup>1</sup> For storm samples, use date of the beginning bottle of affected sample instead of the date it was retrieved.

<sup>2</sup> Method blanks, matrix spikes, and field splits that do not meet criteria fall into this category

<sup>3</sup> Party responsible for implementing corrective action is also responsible for notifying QAO of completion and outcome of corrective action.





# 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 (See options listed below)	
Laboratory # (For Lab Use)	Your Sample ID	Source of Water:		Water Use:			
		<input type="checkbox"/> Public <input type="checkbox"/> Private	<input type="checkbox"/> Well <input type="checkbox"/> Pond <input type="checkbox"/> Lake <input type="checkbox"/> Stream <input type="checkbox"/> Processing plant <input type="checkbox"/> Animal feedlot	<input type="checkbox"/> Wastewater treatment <input type="checkbox"/> Other	<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"/> Private	<input type="checkbox"/> Well <input type="checkbox"/> Pond <input type="checkbox"/> Lake <input type="checkbox"/> Stream <input type="checkbox"/> Processing plant <input type="checkbox"/> Animal feedlot	<input type="checkbox"/> Wastewater treatment <input type="checkbox"/> Other	<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"/> Private	<input type="checkbox"/> Well <input type="checkbox"/> Pond <input type="checkbox"/> Lake <input type="checkbox"/> Stream <input type="checkbox"/> Processing plant <input type="checkbox"/> Animal feedlot	<input type="checkbox"/> Wastewater treatment <input type="checkbox"/> Other	<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) \$20 per sample  
 (Conductivity, pH, Na, Ca, Mg, K, CO<sub>3</sub><sup>2-</sup>, HCO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, Cl<sup>-</sup>, B, Nitrate-N, Hardness, and SAR)
- 2. R + Metals \$30 per sample  
 In addition to Routine Analysis includes:  
 (Zn, Fe, Cu, Mn, and P)
- 3. R + Metals + Heavy Metals (Heavy) + Fluoride \$50 per sample  
 In addition to Routine and Metal analyses includes:  
 (As, Ba, N, Cd, Pb, Cr, and Fluoride)

- 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) \$20 per sample  
 (Total N, P, K, Ca, Mg, Na, Zn, Mn, Fe, and Cu)



## Appendix D

### Data Review Checklist

Y, N, or N/A  
( Y=yes N=no, N/A=not  
applicable)

#### Field Data Review

- A. QC samples (field splits) collected for all analytes as prescribed in the TCEQ SWQM Procedures Manual? \_\_\_\_\_
- B. Field documentation includes the following:
  - (1) Identification of individual collecting samples(s)? \_\_\_\_\_
  - (2) Sample ID number and site location? \_\_\_\_\_
  - (3) Sample collection date and time? \_\_\_\_\_
  - (4) Site observations (i.e. weather, etc.)? \_\_\_\_\_
  - (5) Unusual occurrences that may affect sample? \_\_\_\_\_
  - (6) Sample collection problems? \_\_\_\_\_
- C. Chain of custody record properly filled out and available for review? \_\_\_\_\_

#### Data Format and Structure

- A. Are there any duplicate sample ID numbers? \_\_\_\_\_
- B. Are station location numbers assigned? \_\_\_\_\_
- C. Are sampling dates in the correct format, DD/MM/YYYY? \_\_\_\_\_
- D. Are samples listed in the correct units? \_\_\_\_\_
- E. Is the sampling time entered? \_\_\_\_\_

#### Data Quality Review

- A. Appropriate holding times confirmed? \_\_\_\_\_
- B. MDLs consistent with those in the QAPP? \_\_\_\_\_
- C. Outliers confirmed and documented? \_\_\_\_\_
- D. Documentation (verified error log) provided to TSSWCB? \_\_\_\_\_
- E. Checks on correctness of analysis or data reasonableness performed? (i.e. - Is ortho-phosphorus greater than total phosphorus?) \_\_\_\_\_
- F. Have at least 10% of the data in the database been reviewed against the data sheets? \_\_\_\_\_

Explain any answers that may indicate a problem with the data (attach another page if necessary): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Data Range: \_\_\_\_\_

Data Source: \_\_\_\_\_

Project Leader Signature: \_\_\_\_\_ Date: \_\_\_\_\_

## Appendix E

### SWFTL Methodology References

Parameter	SWFTL Method	Reference
<b>Soil</b>		
Total Kjeldahl N	0073 & 0068	Parkinson, J., and S. Allen. 1975. Comm. Soil Sci. and Plant Anal. 6:1-11.
Total P	0011	
pH	0015	Thomas, G. 1996. In Methods of Soil Analysis, Part 3. SSSA.
Extractable P	00079 & 00081	Mehlich, A. 1984. Commun. Soil Sci. Plant Anal. 15:1409-1416
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	0014	Dorich, R.A., and D.W. Nelson. 1984. Soil Sci. Soc. Am. J. 48:72-75
NH <sub>4</sub> -N	0068	Dorich, R.A., and D.W. Nelson. 1983. Soil Sci. Soc. Am. J. 47:833-836.
Water soluble P	0064 & 0037	Kleinman et al., 2002. Soil Sci. Soc. Am. J. 66:2009-2015 Avila-Segura, M., et al., 2004. Commun. Soil Sci. Plant Anal 35:547-557.
Potassium	0079 & 0081	Mehlich, A. 1984. Commun. Soil Sci. Plant Anal. 15:1409-1416
Calcium	0079 & 0081	Mehlich, A. 1984. Commun. Soil Sci. Plant Anal. 15:1409-1416
Magnesium	0079 & 0081	Mehlich, A. 1984. Commun. Soil Sci. Plant Anal. 15:1409-1416
Sodium	0079 & 0081	Mehlich, A. 1984. Commun. Soil Sci. Plant Anal. 15:1409-1416
Aluminum		Bertsch, P., & P. Bloom. 1996. Methods of Soil Analysis, Part 3, SSSA.
Organic carbon	0055	Nelson, D., and L. Sommers. 1996. Methods of Soil Analysis, Part 3, SSSA.
<b>Water</b>		
Total Kjeldahl N	0073	Parkinson, J., and S. Allen. 1975. Comm. Soil Sci. and Plant Anal. 6:1-11.
Total P	0037	
pH	0041	
NO <sub>3</sub> <sup>-</sup> -N/NO <sub>2</sub> <sup>-</sup> -N	0038	Dorich, R.A., and D.W. Nelson. 1984. Soil Sci. Soc. Am. J. 48:72-75

NH <sub>4</sub> -N	0068	Dorich, R.A., and D.W. Nelson. 1983. Soil Sci. Soc. Am. J. 47:833-836.
Water soluble P	0064 & 0037	Avila-Segura, M., et al., 2004. Commun. Soil Sci. Plant Anal 35:547-557.
Soluble reactive P	0061 & 0062	Murphy, J, and J.P. Riley. 1962. Anal. Chim. Acta 27:31-36.
Potassium	0037	
Calcium	0037	
Magnesium	0037	
Sodium	0037	
<b>Geotube residue</b>		
Total Kjeldahl N	0073 & 0068	Parkinson, J., and S. Allen. 1975. Comm. Soil Sci. and Plant Anal. 6:1-11.
Total P	0035 & 0011	
pH	0015	Thomas, G. 1996. In Methods of Soil Analysis, Part 3. SSSA.
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	0014	Dorich, R.A., and D.W. Nelson. 1984. Soil Sci. Soc. Am. J. 48:72-75
NH <sub>4</sub> -N	0068	Dorich, R.A., and D.W. Nelson. 1983. Soil Sci. Soc. Am. J. 47:833-836.
Water soluble P	0064 & 0037	Kleinman et al., 2002. Soil Sci. Soc. Am. J. 66:2009-2015 Avila-Segura, M., et al., 2004. Commun. Soil Sci. Plant Anal 35:547-557.
Potassium	0035 & 0074	
Calcium	0035 & 0074	
Magnesium	0035 & 0074	
Sodium	0035 & 0074	
Aluminum		Bertsch, P., & P. Bloom. 1996. Methods of Soil Analysis, Part 3, SSSA.
Organic carbon	0055	Nelson, D., and L. Sommers. 1996. Methods of Soil Analysis, Part 3, SSSA
Zinc	0035 & 0074	
Iron	0035 & 0074	
Manganese	0035 & 0074	
<b>Turfgrass</b>		
Total Kjeldahl N	0073 & 0068	Parkinson, J., and S. Allen. 1975. Comm. Soil Sci. and Plant Anal. 6:1-11.
Total P	0035 & 0011	

Potassium	0035 & 0074
Calcium	0035 & 0074
Magnesium	0035 & 0074
Sodium	0035 & 0074
Aluminum	
Organic carbon	0055
Zinc	0035 & 0054
Iron	0035 & 0054
Manganese	0035 & 0054
Copper	0035 & 0054

Bertsch, P., & P. Bloom. 1996. Methods of  
Soil Analysis, Part 3, SSSA.  
Nelson, D., and L. Sommers. 1996.  
Methods of Soil Analysis, Part 3, SSSA

## Appendix F

### Dr. Viotor's and Schnell Methodology References

Variable	Reference
NO <sub>3</sub> <sup>-1</sup> -N/NO <sub>2</sub> <sup>-1</sup> -N	Lopez, J.H., and F. Vargas-Albores. 2003. Aquaculture Research 35:1201-1204. Sims, G.K., T.R. Ellsworth, and R.L. Mulvaney. 1995. commun. Soil Sci. Plant Anal. 26:303-316.
NH <sub>4</sub> -N	Lopez, J.H., and F. Vargas-Albores. 2003. Aquaculture Research 35:1201-1204.
Water soluble P	Kleinman et al., 2002. Soil Sci. Soc. Am.J. 66:2009-2015 Avila-Segura, M., et al., 2004. Commun. Soil Sci. Plant Anal 35:547-557.
Soluble Reactive P	Murphy, J., and J.P Riley. 1962. Anal. Chim. Acta 27:31-36.