Abstract

Texas Institute for Applied Environmental Research (TIAER) at Tarleton State University maintains a comprehensive sampling and analytical program in support of biological and water quality monitoring aspects of several state and federal government projects. This report addresses laboratory quality assurance and quality control reliability criteria for the period January 1, 1999 through December 31, 1999.
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Annual Laboratory Data Summary

Introduction

Texas Institute for Applied Environmental Research (TIAER) at Tarleton State University maintains a comprehensive sampling and analytical program in support of biological and water quality monitoring aspects of several state and federal government projects. Each project relating to data collection and analysis is initiated and controlled by a Quality Assurance Project Plan (QAPP), which directs all activities within the project. The TIAER laboratory provides analytical data to the various project managers under each QAPP. The laboratory manager, who is responsible for data production and quality, oversees the laboratory. The TIAER quality assurance officer is responsible for ensuring that QAPP protocols are adhered to and for auditing the overall quality program. This Annual Laboratory Data Summary and Quality Assurance Report addresses laboratory quality assurance and quality control (QA and QC) reliability criteria for the period January 1, 1999 through December 31, 1999. This document shall serve as the annual report from the TIAER laboratory manager to each project manager.

Analytical methodologies and data acceptance criteria are outlined in the QAPPs for the:

- Water Quality Monitoring and Measurement Activities Relating to the United States Department of Agriculture Bosque River Initiative (USDA)
- Environmental Measurement Activities Relating to the Leon River Watershed 319 Water Quality Demonstration Program (LEON) for the Texas State Soil and Water Conservation Board (TSSWCB)
- Demonstration of Phosphorus Best Management Practices in the North Bosque River Basin (PBMP) for the TSSWCB
- Texas Watch Volunteer Environmental Monitoring Program (TXW) for the Texas Natural Resource Conservation Commission.

Another project, the Wetlands for Nonpoint Source Pollution Control in Small Agricultural Watersheds for the TSSWCB had no samples in 1999 due to drought conditions.

The majority of water samples included in this report were collected from stream sites, Lake Waco, municipal wastewater treatment plant effluents, and from the main water body of flood retardation reservoirs in the Bosque River watershed. Other water samples were collected from the Leon River watershed, application field studies (PBMP), and limited special studies not included under any particular QAPP. In support of local industrial research, FMC Corporation sponsored a special study of their wastewater as it relates to phosphorus and organic discharge control in the Bosque River. The TIAER laboratory also supported the educational and research efforts of Tarleton State University by analysis of 88 samples from a sampling station (ENVS) maintained by a graduate student. Seventy-eight soil samples were collected from field sites (PBMP) and from lake bottom sediments (USDA).

The total number of samples received by the TIAER laboratory in 1999 was 3,196. Of this total, 2,642 samples were collected for the USDA project, 4 samples for the Leon project, 163 samples
for the PBMP project, 6 samples for TXW, 88 samples for ENVS, and 4 samples for other projects.

Of the 3,196 samples collected, 2,907 were water samples. The remaining samples included soil and sediment study samples, inter-laboratory blind comparisons, trip and field blanks, and QC unknown samples. Also, a small number of samples were assigned sample numbers but not analyzed because of leaking bottles and other reasons that precluded complete analysis. In order to ensure equal treatment of all samples with proper quality control, the laboratory staff did not know whether the samples were regular samples intended for the database or were quality control samples. In addition, internal samples representing method blanks, duplicates, spikes, standards, and field duplicates were analyzed by the TIAER laboratory.

Samples were analyzed in accordance with strict EPA protocols whenever applicable. Exceptions included chlorophyll-α (CHLA), soil samples, and bioavailable phosphorus (BAP) which did not have suitable EPA approved protocols. When samples were delivered to the laboratory, they were differentiated by a unique sample number and by the analyses required, but not by particular project identification. Samples were grouped into batches of 10 or less, according to number of samples received, and were placed in numerical order according to sample number. Quality control measures were performed on each batch by choosing the first sample in each batch for duplicate and matrix spiking analysis, thus ensuring uniform randomization by group. This nonspecific batching of samples assured anonymity within the laboratory and dissociated the analyst from specific projects to remove any future perception of bias.

Water samples were collected on both a scheduled grab basis and by automatic samplers during rainfall runoff events for most projects. Data collection and analysis provided information for numerical model validation, assessment of water quality conditions in watersheds, determination of cause and effect relationships of land-use practices to stream and reservoir water quality, and demonstration of best management practice effectiveness.

QA and QC guidelines were followed as stipulated in each QAPP. Depending on required analyses, samples received in the TIAER laboratory were divided into partial aliquots, filtered and preserved in accordance with the appropriate analytical method. EPA-approved methods were utilized for all water analyses when available. Standard Methods for the Examination of Water and Wastewater (18th Edition, 1992) was used as the source in the absence of EPA procedures with the exception of BAP, which was performed in accordance with the method developed by Andrew Sharpley, formerly with Texas A&M University, presently at the USDA Agricultural Research Service (Journal of Environmental Quality, 22:596:601, 1993). Soil analyses methods adhered to the USDA Soil Survey Laboratory Methods Manual (Report No. 42, Aug. 1992) and Methods of Soil Analysis, Soil Science Society of America, 1996. The analytical procedures used are referenced in each QAPP methods table and are presented in Appendix A.

Data Quality Objectives

Data Quality Objectives (DQOs) were developed under the supervision of each contract project manager. Each QAPP addresses specific DQOs for individual projects in Table A7-1: Data Quality Objectives for Measurement Data. While most objectives were met or exceeded, some data did not meet these goals. Sample results with data that did not comply were reanalyzed, not used, or flagged as noncompliant. Noncompliance issues are specifically addressed throughout this document.
Chemical and biological data for samples collected between January 1, 1999 and December 31, 1999 were the result of water samples, duplicate field samples, sediment, soil, and special study sample collection. This report addresses only the samples analyzed by the chemistry laboratory and does not include QA and QC for field operations, computer modeling, economics, policy, or other aspects of TIAER work within a project. This report does not include work performed by contracted or project partner laboratories. Appendix B lists the laboratory quality assurance objectives for the projects as established in Table A7-1 of each QAPP. Appendix C contains all Corrective Action Reports written for all projects in 1999 that affected data quality. Appendix D presents the complete quality control charts for 1999. The complete quality control data tables have become too voluminous to include in this report, but they are available by contacting the TIAER laboratory manager.

Sample Quality Control

Quality control criteria for sample collection are specified in each QAPP. Field criteria include use of a standard format for chain-of-custody, data record sheets, equipment calibration, and collection of grab sample duplicates. Field duplicate statistics are not included in this report, nor are any of the other field parameter information or statistics. Field blanks are not included in this year’s report because sample bottles filled with Type II ASTM deionized water were used in the laboratory as method blanks to assure cleanliness of bottles and freedom from contamination throughout the analytical and sample treatment processes. Laboratory procedures include sample spikes (fortification with analyte of interest), duplicate samples, standards and reagent blanks with appropriate statistical review to ensure an acceptable degree of accuracy and precision for each analytical procedure. The laboratory quality control charts are included in Appendix D of this report. Acceptance criteria are set to ensure validation of data. Control limits, based on standard deviation from the mean, are established to track and trend statistical performance. For the 1999 reporting year, managerial set control limits (MSCL) and acceptance limits were used, rather than control chart limits. This measure was taken to ensure a more stringent control than was delineated by the control charts. In general, managerial limits were set at 80-120 percent. In situations where only one type of sample is analyzed, Shewart control charting would be the method of choice for narrowing quality control acceptance criteria. With all the different sampling sites and types, statistical charting has not shown to be practical as a quality control mechanism. Though analytical techniques were routine and established, sample matrices were not consistent. Some samples were relatively clean while others were extremely complex in matrix, thus producing wide variance in spike recoveries and duplicate deviation. In 1995, the laboratory began to study the establishment of control charting as standard procedure in data acceptance, which has not proven to be as stringent as managerial controls. As a function of quality assurance, the laboratory manager sets control limits when control charts are inconsistent or when nonconformances arise. These limits and procedures for acceptance are taken from Standard Methods and EPA procedures.

Data Completeness

The completeness of data is a relationship of how much of the data are available for use compared to the total potential data. Ideally, 100 percent of the data should be available. However, the possibility of data becoming unavailable due to exceeded holding time, technician error, insufficient sample volume collected, or samples broken in transport must be expected. Also, emergency situations may arise or field conditions may not allow 100 percent data retrieval. Therefore, it is the general goal of TIAER that 90 percent data completeness be required for data usage. Data completeness for the referenced time frame is presented in Table
1. With the exception of bioavailable phosphorus, analytical procedures met the criteria of 90 percent or higher data completeness. Corrective actions were taken when acceptance criteria were not met for quality control measures or when conditions arose that invalidated data. Copies of Corrective Action Reports (CARs) are found in Appendix C.

Table 1  Data Completeness for Monitoring Period January 1 through December 31, 1999

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Abbreviation</th>
<th>Total Database Samples Collected</th>
<th>Samples with Valid Analytical Report</th>
<th>Percent Completeness</th>
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<tr>
<td>Ammonia as nitrogen</td>
<td>NH₃-N</td>
<td>2546</td>
<td>2544</td>
<td>99.9</td>
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<tr>
<td>Orthophosphate as phosphorus</td>
<td>OPO₄-P</td>
<td>2546</td>
<td>2514</td>
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<td>Nitrate and nitrite-nitrogen</td>
<td>NO₂NO₃-N</td>
<td>2762</td>
<td>2757</td>
<td>99.8</td>
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<td>2546</td>
<td>2539</td>
<td>99.7</td>
</tr>
<tr>
<td>Total Kjeldahl nitrogen</td>
<td>TKN</td>
<td>2546</td>
<td>2541</td>
<td>99.8</td>
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<tr>
<td>Total suspended solids</td>
<td>TSS</td>
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<td>Total dissolved solids</td>
<td>TDS</td>
<td>630</td>
<td>626</td>
<td>98.4</td>
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<td>Chemical oxygen demand</td>
<td>COD</td>
<td>798</td>
<td>791</td>
<td>99.1</td>
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<tr>
<td>Biological oxygen demand (5 day)</td>
<td>BOD</td>
<td>146</td>
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<td>Chlorophyll-α</td>
<td>CHL-α</td>
<td>363</td>
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<td>99.7</td>
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<td>Fecal coliform</td>
<td>FColi</td>
<td>622</td>
<td>604</td>
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<tr>
<td>Bioavailable phosphorus</td>
<td>BAP</td>
<td>410</td>
<td>359</td>
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</table>

Analytical Quality Control

All analytical methods for 1999 projects are referenced in the QAPPs and are taken from EPA¹, Standard Methods² or best available technology procedures. The QAPPs specify quality assurance guidelines for quality control samples including blanks, standards, spike samples, and duplicates. EPA certified samples are periodically purchased and analyzed as an added check on quality control. EPA’s Water Pollution Laboratory Performance Evaluation Study ended in 1998 with WP40, so no results are available for 1999. The State of Texas is expected to begin participation in the National Environmental Laboratory Accreditation Program (NELAP) in 2000. The TIAER laboratory will begin analyses of performance samples under this program when it is implemented.

Table 1 provides an overall review of the QA and QC data collected for each of the analytes except chlorophyll-α and fecal coliform. These analytes and more detailed explanations of other analytes are presented in Laboratory Analysis.

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Calculations

Accuracy and precision calculations for standards, spiked samples, and duplicate samples are defined in this section. Method Detection Limits (MDL) procedure is briefly outlined with references for more detailed review.

Standards

Standard solutions were evaluated with each batch of samples for each analyte measured with the exception of total suspended solids (TSS) and total dissolved solids (TDS), fecal coliform, and chlorophyll-α. Accuracy of standard readings were evaluated. The method of calculation for accuracy is as follows:

1) Accuracy

\[
\text{Percent recovery}(y) = \frac{\text{Measured}}{\text{Calculated}} \times 100
\]

The recovery acceptance limit for standard solutions was set at ± 20 percent. Standards may be used to create a calibration curve, or as continuing calibration verification (CCV) checks for an established curve. Normally, high and low range CCVs are used near the high and low calibration standard concentrations. In the case of chemical oxygen demand (COD), five CCVs are used at points throughout the curve. Due to the nature of the instrument used, the calibration curve for COD is factory installed and a full range of check standards were used to ensure continued compliance with assigned concentration parameters.

Spikes

Samples entering the lab were analyzed as received, with a portion of randomly selected samples analyzed with a known amount of the component of interest added to the sample. The sample with the known addition is a spiked sample. This procedure provides a means of checking the effect of the matrix on the analyte of interest. Spiked samples are evaluated in the following manner:

2) \[
\text{Percent recovery}(y) = \frac{\text{Measured}}{\text{Calculated}} \times 100
\]

3) \[
\text{Average Percent Recovery}(Y) = \frac{\sum y}{n}
\]

where

\[n = \text{total number of samples}\]
The control chart warning limit for spikes is equal to two standard deviations, while the control limit is equal to three standard deviations from the mean. Managerial set acceptance criteria for spike recovery is ± 20 percent for every analysis. Quality control data points that were out of control limits are included in control charts for statistical purposes, but sample data were not accepted until the samples in the batch were reanalyzed within control limits.

**Duplicate**

Evaluating duplicate sample replicates is a means of monitoring the precision of the analytical method. Duplicate samples were evaluated in the following manner:

### Table 2 Summary of January 1999 - December 1999 Quality Control Data

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- **TRIPlicate Precision (%)**: Spike accuracy (%)
where

\[ n = \text{total number of samples} \]

The control chart warning limit for duplicates is equal to two standard deviations, while the control limit is equal to three standard deviations from the mean. Managerial set acceptance criteria for duplicate percent deviation is ± 10 percent for every analysis. Quality control data points that were out of control limits are included in control charts for statistical purposes, but sample data were not accepted until the samples in the batch were reanalyzed. If a sample and its duplicate had values that were less than five times the method detection limit, the percent deviation automatically passed the acceptance criteria, regardless of its value, and the data point was included in the control chart.

**Method Detection Limits**

Method Detection Limits (MDLs) are determined using the procedure presented in *Standard Methods for the Examination of Water and Wastewater* (18th Edition, 1992, 1030 E). Briefly stated, the MDL is measured by preparing a sample in the matrix of interest near the suspected method detection limit. This solution is analyzed seven times and standard deviation of the results is calculated. For 6 degrees of freedom and 99 percent confidence, the standard deviation is multiplied by 3.14 to determine the MDL.

\[ 6) \quad \text{MDL} = \text{Standard Deviation (from 7 portions of sample)} \times 3.14 \]

**Laboratory Analysis**

Laboratory data completeness goals of 90 percent were met in 1999, except for bioavailable phosphorus, which is an experimental method under evaluation and is not EPA approved.
Sample load was very light compared to previous years due to a continued record-breaking drought. This drought did allow for a more specialized and varied group of analyses with the use of a smaller staff. Total dissolved solids were added to some site analyses as a means of site specific relationships of observed specific conductance measured in the field to laboratory determined TDS. Silica analyses were performed on Lake Waco samples for information only and were not included in the TIAER main database. *Escherichia coli*, soil, and sediment analyses were also performed for information only.

A complete set of Quality Control charts is attached in Appendix D and the individual tests are discussed below. The data and standard deviation calculations used for Appendix D control charts include data points that failed acceptance criteria. In these cases, data for the samples were not accepted until the analytical batches were reanalyzed and acceptance criteria were met. Per EPA protocol, duplicate percent deviation acceptance criteria did not include sample values that were less than five times the method detection limit for the particular analyte of interest. For most analytes, extra quality control measures were implemented that were not listed in the QAPPs. These included high, middle, and/or low range continuing calibration verification (CCV-1, CCV-2, etc.) standards, laboratory control standards (LCS) from sources other than calibration standards, lysine as an extra LCS for TKN analysis, and other matrix spikes to determine interferences.

Though control charts were used during this reporting period, managerial set upper and lower limits (MSUL and MSLL, respectively) were used for acceptance criteria. Analysts recorded when control limits were exceeded in personal laboratory notebooks; however, the automatically calculated limits were not used as a basis for acceptance criteria. Some control charts had calculated limits that were broad due to outlying data points that were not used. In general, accuracy limits for standards and matrix spikes were set at ± 20 percent recovery while precision limits were set at ± 10 percent for duplicate deviation. The following sections explain individual analyte quality control history for the reporting period. Corrective action reports that delineate problems with analysis or sample handling are found in Appendix C. Control charts are found in Appendix D.

Method Detection Limits (MDLs) for chemical constituents are dynamic in that the MDL is a function of many factors present in the laboratory, including but not limited to, the reagents used, the equipment condition, and the environment in the laboratory. MDLs are determined and recalculated on a semiannual basis to ensure satisfactory sensitivity in the procedures. The 1999 MDLs for the various analytes are found in Table 2.

### Ammonia Nitrogen

#### Standards

The average percent recovery for ammonia nitrogen CCV-1 and CCV-2 accuracy check standards in this reporting period were 97.9 and 99.1, respectively. The LCS average percent recovery was 99.7. The acceptance limit for all standards was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D.
Spikes

The average percent recovery for all ammonia nitrogen matrix spikes in this reporting period was 101.1. The acceptance limit for all spikes was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. A total of 2,546 samples were evaluated for ammonia nitrogen in the study period along with 329 matrix spikes. This represents 12.9 percent spikes. The goal, as stated in the QAPPs of 10 percent spikes, was achieved.

Duplicates

Precision of the ammonia nitrogen data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for the duplicates was ± 5.16. The acceptance limit for duplicates was ± 10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D.

A total of 2,546 samples were evaluated for ammonia nitrogen in the study period along with 329 duplicates. This represents 12.9 percent duplicates. The goal, as stated in the QAPPs of 10 percent duplicates, was achieved.

Orthophosphate Phosphorus

Standards

The average percent recovery for orthophosphate phosphorus CCV-1 and CCV-2 accuracy check standards in this reporting period were 99.7 and 102.0, respectively. The LCS average percent recovery was 101.2. The acceptance limit for all standards was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D.

Spikes

The average percent recovery for all orthophosphate phosphorus matrix spikes in this reporting period was 100.5. The acceptance limit for all spikes was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. A total of 2,546 samples were evaluated for orthophosphate phosphorus in the study period along with 345 matrix spikes. This represents 13.6 percent spikes. The goal, as stated in the QAPPs of 10 percent spikes, was achieved.

Duplicates

Precision of the orthophosphate phosphorus data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for
the duplicates was +1.13. The acceptance limit for duplicates was ±10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 2,546 samples were evaluated for orthophosphate phosphorus in the study period along with 347 duplicates. This represents 13.6 percent duplicates. The goal, as stated in the QAPPs of 10 percent duplicates, was achieved.

Nitrite- and nitrate-nitrogen

Standards

The average percent recovery for nitrite-nitrogen CCV-1 and CCV-2 accuracy check standards in this reporting period were 100.0 and 100.4, respectively. The nitrite-nitrogen LCS average percent recovery was 100.6. The average percent recovery for nitrate-nitrogen CCV-1 and CCV-2 accuracy check standards in this reporting period were 96.5 and 97.1, respectively. The nitrate-nitrogen LCS average percent recovery was 102.6. The acceptance limit for all standards was ±20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D.

Spikes

The average percent recovery for all nitrite-nitrogen matrix spikes in this reporting period was 97.0. The average percent recovery for all nitrate-nitrogen matrix spikes in this reporting period was 96.6. The acceptance limit for all spikes was ±20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. A total of 2,762 samples were evaluated for nitrite-nitrogen in the study period. This sample number is higher than with other analyses because of special studies conducted along the southern portion of the Bosque River watershed. Matrix spikes were performed on 354 samples for nitrite nitrogen. This represents 12.8 percent spikes. A total of 2,762 samples were evaluated for nitrate-nitrogen in the study period along with 349 matrix spikes. This represents 12.6 percent spikes. The goal, as stated in the QAPPs of 10 percent spikes, was achieved.

Duplicates

Precision of the nitrate-nitrite nitrogen data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for the nitrite-nitrogen duplicates was +1.62. The average percent deviation for the nitrate-nitrogen duplicates was –0.21. The acceptance limit for duplicates was ±10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 2,762 samples were evaluated for nitrate-nitrogen in the study period along with 356 duplicates. This represents 12.9 percent duplicates. A total of 2,762 samples were evaluated for nitrate-nitrogen in the study period along with 349 duplicates. This represents 12.6 percent duplicates. The goal, as stated in the QAPPs of 10 percent duplicates, was achieved.
Total Phosphorus

In 1999 the total phosphorus method was modified to extend the lower range of detection (LRTP) to better meet project needs. Additional lower range calibration and check standards were added and separate low range curves were created with separate QA and QC measures that still met EPA requirements. The analyses are essentially the same. Higher range standards were the same for LRTP and TP up to 1.0 mg/L. The low range total phosphorus (LRTP) data are included in the following text.

Standards

The average percent recovery for total phosphorus CCV-2 and CCV-3 accuracy check standards in this reporting period were 104.7 and 99.0, respectively. The LCS average percent recovery was 100.4. The LRTP average percent recoveries were 104.4 and 100.3 for CCV-1 and the LCS, respectively. The acceptance limit for all standards was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D.

Spikes

The average percent recovery for all total phosphorus (excluding LRTP) matrix spikes in this reporting period was 99.3. The LRTP average percent spike recovery was 100.5. The acceptance limit for all spikes was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. A total of 2,546 samples were evaluated for total phosphorus in the study period along with 261 matrix spikes. This represents 10.3 percent spikes. The goal, as stated in the QAPPs of 10 percent spikes, was achieved.

Duplicates

Precision of the total phosphorus data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for the duplicates was + 1.24. The LRTP average percent deviation was + 2.62. The acceptance limit for duplicates was ± 10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 2,546 samples were evaluated for total phosphorus in the study period along with 262 duplicates. This represents 10.3 percent duplicates. The goal, as stated in the QAPPs of 10 percent duplicates, was achieved.

Total Kjeldahl Nitrogen

Standards

The average percent recovery for total Kjeldahl nitrogen CCV-1 and CCV-2 standards in this reporting period were 103.6 and 97.1, respectively. The LCS average percent recovery was 98.3. The acceptance limit for standards was ± 20 percent. Lysine was used as an additional standard, for information purposes only. Lysine exhibits breakage of double carbon-nitrogen
bonds to demonstrate that complete digestion has taken place. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D.

Spikes

The average percent recovery for all total Kjeldahl nitrogen matrix spikes in this reporting period was 101.6. The acceptance limit for all spikes was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. A total of 2,546 samples were evaluated for total Kjeldahl nitrogen in the study period along with 398 matrix spikes. This represents 15.6 percent spikes. The goal, as stated in the QAPPs of 10 percent spikes, was achieved.

Duplicates

Precision of the total Kjeldahl nitrogen data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for the duplicates was –0.88. The acceptance limit for duplicates was ± 10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 2,546 samples were evaluated for total Kjeldahl nitrogen in the study period along with 388 duplicates. This represents 15.2 percent duplicates. The goal, as stated in the QAPPs of 10 percent duplicates, was achieved.

Total Suspended and Dissolved Solids

Standards and Spikes

Standards and spikes are not applicable for total suspended and dissolved solids analyses.

Duplicates

Precision of the total suspended and dissolved solids data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for total suspended solids duplicates was –2.07. The average percent deviation for total dissolved solids duplicates was +1.78. The acceptance limit for duplicates was ± 10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 2,538 samples were evaluated for total suspended solids in the study period along with 334 duplicates. This represents 13.2 percent duplicates. A total of 630 samples were evaluated for total dissolved solids in the study period along with 66 duplicates. This represents 10.5 percent duplicates. The goal, as stated in the QAPPs of 10 percent duplicates, was achieved for total suspended solids. Total dissolved solids were not included in any QAPP and were analyzed for information only.
Chemical Oxygen Demand

Standards

The average percent recovery for chemical oxygen demand CCV-2 and CCV-4 accuracy check standards in this reporting period were 98.3, and 98.5, respectively. The addition of three other CCVs as extra checks on accuracy were added last year for information only, but these values are not included in Table 2. The LCS average percent recovery was 105.4. The acceptance limit for all standards was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D.

Spikes

The average percent recovery for all chemical oxygen demand matrix spikes in this reporting period was 105.0. The acceptance limit for all spikes was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. A total of 798 samples were evaluated for chemical oxygen demand in the study period along with 121 matrix spikes. This represents 15.2 percent spikes. The goal, as stated in the QAPPs of 10 percent spikes, was achieved.

Duplicates

Precision of the chemical oxygen demand data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for the duplicates was +4.42. The acceptance limit for duplicates was ± 10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 798 samples were evaluated for chemical oxygen demand in the study period along with 121 duplicates. This represents 15.2 percent duplicates. The goal, as stated in the QAPPs of 10 percent duplicates, was achieved.

Five-Day Biochemical Oxygen Demand

Standards

The average concentration recovery for biochemical oxygen demand (BOD₅) glucose and glutamic acid accuracy check standards in this reporting period was 166mg/L. Since a precise concentration standard is not used by the procedure, no percent recoveries or CCVs are used for BOD. The control limits established for acceptance during this reporting period were 72.0–261. No data were outside of the control chart. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. If analytical or sampling errors were made, samples could not be reanalyzed due to holding time restraints.
Spikes and Blanks

Matrix spiking is not applicable to biochemical oxygen demand. Seeded and unseeded dilution water blanks were analyzed with each daily analysis. Seeded blanks are tabulated and control charted in Appendix D. Only one blank was recognized as presenting a problem. A study was performed to determine the source of contamination and the deionized water source was suspected. A new system with a reversed osmosis unit preceding the demineralizers was installed and the problem appeared to be corrected.

Duplicates

Precision of the biochemical oxygen demand data was determined by evaluating glucose and glutamic acid standards in duplicate. Laboratory duplicates on samples involved various dilutions of the same sample aliquot and were not tabulated as a separate entity. The average percent deviation for the standard duplicates was +0.28. The acceptance limit for duplicates was −16.0 to 16.6 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 146 samples were evaluated for biochemical oxygen demand in the study period along with 12 glucose and glutamic acid duplicates. The glucose and glutamic acid duplicates were analyzed on a daily basis as required by the EPA, rather than on each batch of ten samples. A goal for duplicate frequency was not stated in the QAPPs.

Chlorophyll-α

Standards and Spikes

Spikes and standards were not applicable for chlorophyll-α. A spinach derived chlorophyll-α source was found from one supply company and was used for part of the year to determine applicability as a standard in the future. This and other sources are still under evaluation for usefulness, reliability, and reproducibility. At present determination, the standards do not appear to be stable enough to be of use.

Duplicates

Laboratory precision was not determined for chlorophyll-α. Every sample was performed in duplicate and the two values for each sample were averaged to obtain the chlorophyll-α level.

Silica

Silica analyses, as combined silicates (SiO₂), were added to the Lake Waco portion of the USDA project. Silica was not defined in the QAPP and is not added to the database, so an accurate count of samples used in silica analysis has not been made. Though used for information purposes only, EPA protocols were used with appropriate QA and QC measures employed.
Standards

The average percent recovery for silica CCV-1 and CCV-2 accuracy check standards in this reporting period were 107.0 and 95.8 respectively. The LCS average percent recovery was 94.0. The acceptance limit for all standards was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D.

Spikes

The average percent recovery for all silica matrix spikes in this reporting period was 97.5. The acceptance limit for all spikes was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. Since silica was not entered into the TIAER database, no calculations of spike frequency to sample number received were made. However, greater than 10 percent frequency was achieved since these QC measures are performed on every batch of 10 samples or less.

Duplicates

Precision of the silica data was determined by evaluating samples in duplicate. Only laboratory duplicates are described here. The average percent deviation for the duplicates was + 0.60. The acceptance limit for duplicates was ± 10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. Since silica was not entered into the TIAER database, no calculations of duplicate frequency to sample number received were made. However, greater than 10 percent frequency was achieved since these QC measures are performed on every batch of 10 samples or less.

Bioavailable Phosphorus

An additional test of bioavailable phosphorus was introduced in 1998 on an experimental basis. The method, as proposed by Sharpley, 1993\(^3\) involves adsorption of phosphorus onto ferric chloride impregnated, oxidized strips and estimates the phosphorus that is readily available for biological use in an ecosystem. Standard protocols for QA and QC were employed, though the method is only an estimate. Percent completeness was less than 90 percent, but no goal was stated in the QAPP, and BAP was not an analysis of interest in any of the latest QAPP revisions. BAP values were stored in a separate database for use as nonessential information only.

Standards

The average percent recovery for bioavailable phosphorus CCV-1 and CCV-2 accuracy check standards in this reporting period were 107.7 and 100.2, respectively. The LCS average percent recovery was 97.8. The acceptance limit for all standards was ± 20 percent. Data outside of the

Annual Laboratory Data Summary

Spikes

The average percent recovery for all bioavailable phosphorus matrix spikes in this reporting period was 94.9. The acceptance limit for all spikes was ± 20 percent. Data outside of the acceptance limit were not used and the samples were reanalyzed. The data reported were within the criteria specified in the QAPPs. Control charts are found in Appendix D. A total of 410 samples were evaluated for bioavailable phosphorus in the study period along with 107 matrix spikes. This represents 26.1 percent spikes. The method by Sharpley does not call for matrix spikes, but the TIAER laboratory manager patterns almost all analytical procedures after EPA type protocols. No goal was stated in the QAPPs for bioavailable phosphorus.

Duplicates

Precision of the bioavailable phosphorus data was determined by evaluating samples in duplicate. Laboratory duplicates only are described here. The average percent deviation for the duplicates was + 1.68. The acceptance limit for duplicates was ± 10 percent deviation. Data outside of the acceptance limit were not used and the samples were reanalyzed. Control charts are found in Appendix D. A total of 410 samples were evaluated for bioavailable phosphorus in the study period along with 107 duplicates. This represents 26.1 percent. The method by Sharpley does not call for duplicate analyses, but the TIAER laboratory manager patterns almost all analytical procedures after EPA type protocols. No goal was stated in the QAPPs for bioavailable phosphorus.

Soils

Some other elements of the PBMP and other programs involved the edge-of-field collection of water samples from field plots receiving dairy waste for irrigation, reservoir and lake sediments, and row crop field runoff samples. Only water samples collected from runoff on these fields are included in the quality control measurements tabulated in Appendix D. Soil analyses are not included in the quality control charts due to the limited number of samples and complex matrix differences, which make comparisons to water inappropriate. Soil testing was conducted in the field plots to provide data for testing of computer models and to increase understanding of the edge-of-field data. Soils were analyzed for various combinations of total Kjeldahl nitrogen, extractable nitrate-nitrogen, percent organic carbon, extractable (weak Bray) phosphorus, total phosphorus, and metals. All data reported met the precision and accuracy requirements listed in Table A-7 of the USDA QAPP. Statistical information is not provided here because the information collected was not for data reporting purposes and is not contained in the TIAER database. For information purposes, these data are available on request from TIAER.

Other Chemical Analyses

Other analyses were performed in the laboratory for special studies and for comparisons to other parameters, but were not used as data for any project. These included, but were not limited to, chloride, metals, total residual chlorine, pH, and conductivity. The number of
samples did not warrant control charting or statistical analysis for quality control. The tests that were performed did meet or exceed EPA protocols where warranted.

Bacteriological Testing

Bacteriological analyses were separated from chemical testing for statistical purposes since the methods and applications differ so significantly. Fecal coliform analyses were performed more frequently during this reporting period than in previous years. On more turbid samples, interference was usually noted. Both cream colored and yellow colored colonies appeared with the characteristic blue fecal coliform colonies on sample plates of turbid samples. These other colonies have been found to be atypical coliform bacteria. Since the yellow and cream colored colonies did not meet the criteria of the Standard Methods procedure, they were not included in colony number determinations, but have been noted on data sheets for future reference. A MDL for fecal coliform is 40 colonies per 100 ml as presented in the QAPPs. Blanks were analyzed at the beginning and ending of the filtering and plating procedure. At no time during any analysis did the blanks show contamination, which indicated that proper sterile technique was used throughout the reporting period. Though the QAPPs did not require standards, duplicates, or spikes for bacterial analyses, laboratory duplicates were analyzed for fecal coliform for information purposes only, but not charted statistically. Bacteriological duplicate percent deviation is being evaluated as a means for data acceptance criteria.

*Escherichia coli* analyses were also performed to make a preliminary determination of suitability for use as a water quality indicator. *E. coli* were determined from fecal coliform plates after colony counting and were taken as a comparison study for information only.

Performance and Systems Audit

TIAER is committed to providing accurate, precise measurements for all monitoring investigations. To ensure this, approved instrumentation and EPA-approved analytical techniques are used, whenever they exist, for all measurements. Quality of data is also assured through periodic verification that instruments, installations, and methods are correctly used and all instruments are operating properly. Field performance audits were conducted throughout the monitoring period by TIAER supervisors. These audits included verification that sampling and measurement equipment was in proper working order. TIAER supervisors performed a careful review of each automated sampler installation in 1999. The supervisors observed sampling procedures to verify that suitable techniques were used and that field staff received adequate training. All new monitoring personnel underwent training and supervised field performance prior to conducting monitoring. Supervisors reviewed field and laboratory notebooks and data entry forms to ensure that personnel provided acceptable documentation of sample and data collection and field observations. Entry of all data into the TIAER databases was verified to minimize errors from data entry. Field duplicate and data collection activities are beyond the scope of this laboratory report.
# APPENDIX A

## Laboratory Analysis

### Table A–1

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<td>Total phosphorus—soils</td>
<td>USGS p.367</td>
<td>Acid digestion, colorimetric</td>
</tr>
</tbody>
</table>


## Laboratory Quality Assurance Objectives

Table B–1  Laboratory Quality Assurance Objectives  
*Vary between QAPPs due to date of submission: are determined semiannually by the laboratory**Determined at the time of analysis, not established by the EPA

<table>
<thead>
<tr>
<th>Test</th>
<th>Estimated Precision Limits (Deviation)</th>
<th>Estimated Accuracy Limits</th>
<th>Method Detection Limits 1999 May/November</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonia as nitrogen</td>
<td>10%</td>
<td>80-120%</td>
<td>0.030/0.037 mg/L</td>
</tr>
<tr>
<td>Orthophosphate as phosphorus</td>
<td>10%</td>
<td>80-120%</td>
<td>0.003/0.006 mg/L</td>
</tr>
<tr>
<td>Nitrite as nitrogen</td>
<td>10%</td>
<td>80-120%</td>
<td>0.007/0.004 mg/L</td>
</tr>
<tr>
<td>Nitrate as nitrogen</td>
<td>10%</td>
<td>80-120%</td>
<td>0.010/0.013 mg/L</td>
</tr>
<tr>
<td>Total Kjeldahl nitrogen</td>
<td>10%</td>
<td>80-120%</td>
<td>0.150/0.122 mg/L</td>
</tr>
<tr>
<td>Total phosphorus</td>
<td>10%</td>
<td>80-120%</td>
<td>0.053/0.00048 mg/L</td>
</tr>
<tr>
<td>Residue, nonfilterable</td>
<td>10%</td>
<td>80-120%</td>
<td>4/6 mg/L</td>
</tr>
<tr>
<td>Residue, total dissolved</td>
<td>10%</td>
<td>80-120%</td>
<td>10/10 mg/L</td>
</tr>
<tr>
<td>Chemical oxygen demand</td>
<td>10%</td>
<td>80-120%</td>
<td>4/4 mg/L</td>
</tr>
<tr>
<td>5-day biochemical oxygen demand</td>
<td>10%</td>
<td>na</td>
<td>2.2 mg/L (fixed)</td>
</tr>
<tr>
<td>Fecal coliform bacteria</td>
<td>na</td>
<td>na</td>
<td>40 col/100 mL (fixed)</td>
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<tr>
<td>Chlorophyll-a</td>
<td>na</td>
<td>na</td>
<td>0.99/2.46 MG/M³</td>
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<tr>
<td>Silica</td>
<td>10%</td>
<td>80-120%</td>
<td>1.0 MG/L (fixed)</td>
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<tr>
<td>Bioavailable phosphorus</td>
<td>10%</td>
<td>80-120%</td>
<td>0.045/0.039 mg/L</td>
</tr>
<tr>
<td>Soils—total Kjeldahl nitrogen</td>
<td>10%</td>
<td>80-120%</td>
<td>na</td>
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<tr>
<td>Soils—extractable nitrate</td>
<td>10%</td>
<td>80-120%</td>
<td>na</td>
</tr>
<tr>
<td>Soils—extractable phosphorus</td>
<td>10%</td>
<td>80-120%</td>
<td>na</td>
</tr>
<tr>
<td>Soil—total phosphorus</td>
<td>10%</td>
<td>80-120%</td>
<td>na</td>
</tr>
<tr>
<td>Soil—estimated organic carbon</td>
<td>10%</td>
<td>80-120%</td>
<td>na</td>
</tr>
</tbody>
</table>
APPENDIX D

Control Charts

LCL= Lower control limit
LWL= Lower warning limit
UWL= Upper warning limit
UCL= Upper control limit
MSLR= Manager set limit range
StDev= Standard deviation

Figure D–1  Ammonia Nitrogen Control Charts

CCV1

<table>
<thead>
<tr>
<th>%</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>97.95</td>
</tr>
<tr>
<td>StDev</td>
<td>10.62</td>
</tr>
<tr>
<td>LCL</td>
<td>66.07</td>
</tr>
<tr>
<td>LWL</td>
<td>76.70</td>
</tr>
<tr>
<td>UWL</td>
<td>119.19</td>
</tr>
<tr>
<td>UCL</td>
<td>129.82</td>
</tr>
<tr>
<td>MSLR</td>
<td>80-120</td>
</tr>
</tbody>
</table>

NH3-N CCV1 Recovery

CCV2

<table>
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<tr>
<th>%</th>
<th></th>
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<tbody>
<tr>
<td>Mean</td>
<td>99.06</td>
</tr>
<tr>
<td>StDev</td>
<td>5.48</td>
</tr>
<tr>
<td>LCL</td>
<td>82.61</td>
</tr>
<tr>
<td>LWL</td>
<td>88.09</td>
</tr>
<tr>
<td>UWL</td>
<td>110.03</td>
</tr>
<tr>
<td>UCL</td>
<td>115.51</td>
</tr>
<tr>
<td>MSLR</td>
<td>80-120</td>
</tr>
</tbody>
</table>

NH3-N CCV2 Recovery
### LCS %
- **Mean:** 99.68
- **StDev:** 4.60
- **LCL:** 85.86
- **LWL:** 90.47
- **UWL:** 108.88
- **UCL:** 113.49
- **MSLL:** 80-120

### Spikes %
- **Mean:** 101.11
- **StDev:** 26.74
- **LCL:** 20.89
- **LWL:** 47.63
- **UWL:** 154.59
- **UCL:** 181.32
- **MSLL:** 80-120

### Duplicates %
- **Mean:** 5.17
- **StDev:** 20.61
- **LCL:** -56.64
- **LWL:** -36.04
- **UWL:** 46.38
- **UCL:** 66.99
- **MSLL:** -10 to +10
Figure D-2 Orthophosphate Phosphorus Control Charts

CCV1 %
Mean= 99.70
StDev= 12.99
LCL= 60.72
LWL= 73.71
UWL= 125.68
UCL= 138.67
MSLR= 80-120

CCV2 %
Mean= 101.97
StDev= 9.34
LCL= 73.94
LWL= 83.28
UWL= 120.66
UCL= 130.00
MSLR= 80-120

LCS %
Mean= 101.15
StDev= 3.11
LCL= 91.81
LWL= 94.92
UWL= 107.37
UCL= 110.48
MSLR= 80-120
Figure D-3  Low Level Special Study Orthophosphate Phosphorus Control Charts
### Appendix D Control Charts

#### CCV2

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Mean</td>
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</tr>
<tr>
<td>StDev</td>
<td>1.00</td>
</tr>
<tr>
<td>LCL</td>
<td>96.76</td>
</tr>
<tr>
<td>LWL</td>
<td>97.76</td>
</tr>
<tr>
<td>UWL</td>
<td>101.76</td>
</tr>
<tr>
<td>UCL</td>
<td>102.76</td>
</tr>
<tr>
<td>MSLR</td>
<td>80-120</td>
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</table>

#### LCS

<table>
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<tr>
<td>StDev</td>
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</tr>
<tr>
<td>LCL</td>
<td>95.45</td>
</tr>
<tr>
<td>LWL</td>
<td>96.87</td>
</tr>
<tr>
<td>UWL</td>
<td>102.55</td>
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<tr>
<td>UCL</td>
<td>103.97</td>
</tr>
<tr>
<td>MSLR</td>
<td>80-120</td>
</tr>
</tbody>
</table>

#### Spikes

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>99.97</td>
</tr>
<tr>
<td>StDev</td>
<td>1.57</td>
</tr>
<tr>
<td>LCL</td>
<td>95.27</td>
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<tr>
<td>LWL</td>
<td>96.83</td>
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<tr>
<td>UWL</td>
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<tr>
<td>UCL</td>
<td>104.68</td>
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<tr>
<td>MSLR</td>
<td>80-120</td>
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</table>
Figure D-4  Nitrate Nitrogen Control Charts

Duplicates  %
Mean= 0.97  
StDev= 5.13  
LCL= -14.43  
LWL= -9.29  
UWL= 11.23  
UCL= 16.37  
MSLR= 80-120

Low Level PO4-P Duplicate Deviation

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<th>% deviation</th>
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<tr>
<td>40</td>
<td>10</td>
</tr>
<tr>
<td>50</td>
<td>20</td>
</tr>
</tbody>
</table>

CCV1  %
Mean= 96.49  
StDev= 12.80  
LCL= 58.09  
LWL= 70.89  
UWL= 122.08  
UCL= 134.88  
MSLR= 80-120

NO3-N CCV1 Recovery

<table>
<thead>
<tr>
<th>measurement</th>
<th>% recovery</th>
</tr>
</thead>
<tbody>
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<td>0</td>
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</tr>
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<td>50</td>
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<tr>
<td>500</td>
<td>100</td>
</tr>
<tr>
<td>550</td>
<td>110</td>
</tr>
</tbody>
</table>

CCV2  %
Mean= 97.11  
StDev= 7.31  
LCL= 75.18  
LWL= 82.49  
UWL= 111.73  
UCL= 119.04  
MSLR= 80-120

NO3-N CCV2 Recovery

<table>
<thead>
<tr>
<th>measurement</th>
<th>% recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
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<td>500</td>
<td>100</td>
</tr>
<tr>
<td>550</td>
<td>110</td>
</tr>
</tbody>
</table>
### LCS

- **Mean:** 102.55
- **StDev:** 4.69
- **LCL:** 88.47
- **LWL:** 93.16
- **UWL:** 111.94
- **UCL:** 116.63
- **MSLR:** 80-120

### Spikes

- **Mean:** 96.62
- **StDev:** 11.13
- **LCL:** 63.22
- **LWL:** 74.36
- **UWL:** 118.88
- **UCL:** 130.02
- **MSLR:** 80-120

### Duplicates

- **Mean:** -0.21
- **StDev:** 20.58
- **LCL:** -61.96
- **LWL:** -41.38
- **UWL:** 40.96
- **UCL:** 61.54
- **MSLR:** -10 to +10
Figure D–5  Nitrate Nitrogen Control Charts

CCV1  
Mean= 100.04  
StDev= 7.45  
LCL= 77.69  
LWL= 85.14  
UWL= 114.94  
UCL= 122.39  
MSLR= 80-120

CCV2  
Mean= 100.44  
StDev= 3.78  
LCL= 89.11  
LWL= 92.89  
UWL= 107.99  
UCL= 111.77  
MSLR= 80-120

LCS  
Mean= 100.55  
StDev= 2.91  
LCL= 91.82  
LWL= 94.73  
UWL= 106.38  
UCL= 109.29  
MSLR= 80-120
Appendix D Control Charts

Figure D–6  Total Phosphorus High Range Control Charts
**Annual Laboratory Data Summary**

**CCV3 %**
- **Mean**: 99.00
- **StDev**: 5.76
- **LCL**: 81.71
- **LWL**: 87.47
- **UWL**: 110.53
- **UCL**: 116.29
- **MSLR**: 80-120

**TP High CCV3 Recovery**

**LCS %**
- **Mean**: 100.43
- **StDev**: 4.92
- **LCL**: 85.66
- **LWL**: 90.58
- **UWL**: 110.28
- **UCL**: 115.20
- **MSLR**: 80-120

**TP High LCS Recovery**

**Spike %**
- **Mean**: 99.34
- **StDev**: 19.59
- **LCL**: 40.58
- **LWL**: 60.17
- **UWL**: 138.51
- **UCL**: 158.10
- **MSLR**: 80-120

**TP High Spike Recovery**
Figure D–7  Total Phosphorous Low Range Control Charts

**Duplicates %**
- Mean = 1.24
- StDev = 10.82
- LCL = -31.23
- LWL = -20.41
- UWL = 22.88
- UCL = 33.70
- MSLR = 80-120

**CCV1 %**
- Mean = 104.42
- StDev = 23.31
- LCL = 34.50
- LWL = 57.81
- UWL = 151.04
- UCL = 174.35
- MSLR = 80-120

**CCV2 %**
- Mean = 103.47
- StDev = 11.32
- LCL = 69.52
- LWL = 80.84
- UWL = 126.10
- UCL = 137.42
- MSLR = 80-120
LCS %
Mean= 100.28
StDev= 14.95
LCL= 55.44
LWL= 70.39
UWL= 130.18
UCL= 145.12
MSLR= 80-120

Spike %
Mean= 100.46
StDev= 19.96
LCL= 40.58
LWL= 60.54
UWL= 140.39
UCL= 160.35
MSLR= 80-120

Duplicates %
Mean= 2.62
StDev= 20.12
LCL= -57.75
LWL= -37.63
UWL= 42.86
UCL= 62.98
MSLR= -10 to +10
Figure D–8 Total Kjeldahl Nitrogen Control Charts

**CCV2 %**
- Mean= 103.57
- StDev= 15.49
- LCL= 57.09
- LWL= 72.59
- UWL= 134.56
- UCL= 150.05
- MSLR= 80-120

**TKN CCV2 Recovery**

<table>
<thead>
<tr>
<th>Measurement</th>
<th>% recovery</th>
</tr>
</thead>
<tbody>
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</tr>
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<td>500</td>
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<tr>
<td>600</td>
<td>600</td>
</tr>
</tbody>
</table>

**CCV3 %**
- Mean= 99.20
- StDev= 6.41
- LCL= 79.95
- LWL= 86.37
- UWL= 112.02
- UCL= 118.44
- MSLR= 80-120

**TKN CCV3 Recovery**

<table>
<thead>
<tr>
<th>Measurement</th>
<th>% recovery</th>
</tr>
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<tbody>
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<td>500</td>
<td>500</td>
</tr>
<tr>
<td>600</td>
<td>600</td>
</tr>
</tbody>
</table>

**LCS %**
- Mean= 98.28
- StDev= 3.91
- LCL= 86.57
- LWL= 90.47
- UWL= 106.10
- UCL= 110.00
- MSLR= 80-120

**TKN LCS Recovery**

<table>
<thead>
<tr>
<th>Measurement</th>
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</tr>
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<tbody>
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<td>500</td>
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<tr>
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</tbody>
</table>
### Lysine

- **Mean:** 97.92
- **StDev:** 5.63
- **LCL:** 81.04
- **LWL:** 86.67
- **UWL:** 109.17
- **UCL:** 114.80
- **MSLR:** 80-120

### Spike

- **Mean:** 101.56
- **StDev:** 18.16
- **LCL:** 47.08
- **LWL:** 65.24
- **UWL:** 137.87
- **UCL:** 156.03
- **MSLR:** 80-120

### Duplicates

- **Mean:** -0.88
- **StDev:** 9.68
- **LCL:** -29.91
- **LWL:** -20.23
- **UWL:** 18.48
- **UCL:** 28.16
- **MSLR:** 80-120
Figure D–9 Solids (Residue) Control Charts

Duplicates %
Mean= 1.78
StDev= 13.40
LCL= -38.43
LWL= -25.02
UWL= 28.59
UCL= 41.99
MSLR= -10 to +10

Figure D–10 Chemical Oxygen Demand Control Charts

Duplicates %
Mean= -2.07
StDev= 16.08
LCL= -50.32
LWL= -34.24
UWL= 30.10
UCL= 46.19
MSLR= -10 to +10
Annual Laboratory Data Summary

CCV2 %
Mean= 98.28
StDev= 6.98
LCL= 77.32
LWL= 84.31
UWL= 112.24
UCL= 119.23
MSLR= 80-120

COD CCV2 Recovery

CCV3 %
Mean= 98.30
StDev= 9.33
LCL= 70.31
LWL= 79.64
UWL= 116.96
UCL= 126.29
MSLR= 80-120

COD CCV3 Recovery

CCV4 %
Mean= 98.46
StDev= 2.52
LCL= 90.89
LWL= 93.41
UWL= 103.50
UCL= 106.03
MSLR= 80-120

COD CCV4 Recovery
Appendix D  Control Charts

**CCV5**  
Mean= 100.28  
StDev= 6.72  
LCL= 80.11  
LWL= 86.83  
UWL= 113.73  
UCL= 120.45  
MSLR= 80-120

**LCS**  
Mean= 105.28  
StDev= 8.72  
LCL= 79.13  
LWL= 87.84  
UWL= 122.72  
UCL= 131.43  
MSLR= 80-120

**Spike**  
Mean= 105.14  
StDev= 10.24  
LCL= 74.41  
LWL= 84.65  
UWL= 125.63  
UCL= 135.87  
MSLR= 80-120
Figure D–11  Biochemical Oxygen Demand Control Charts

### Duplicates

- **Mean**: 4.42
- **StDev**: 36.80
- **LCL**: -105.98
- **LWL**: -69.18
- **UWL**: 78.02
- **UCL**: 114.82
- **MSLR**: -10 to +10

### Seed Blank

- **mean**: 0.41
- **SD**: 0.26
- **LCL**: -0.36
- **LWL**: -0.10
- **UWL**: 0.92
- **UCL**: 1.17
- **MSLR**: 80-120

### Glucose/Glutamic Acid

- **mean**: 0.28
- **SD**: 5.43
- **LCL**: -16.00
- **LWL**: -10.57
- **UWL**: 11.14
- **UCL**: 16.56
- **MSLR**: N/A
Appendix D  Control Charts

Figure D–12  Silica Control Charts

**G/G average**

- **mean**: 166.51
- **SD**: 31.52
- **LCL**: 71.96
- **LWL**: 103.47
- **UWL**: 229.54
- **UCL**: 261.05
- **MSLR**: 100 to 250

**Glucose/Glutamic Acid Readings**

![Glucose/Glutamic Acid Readings](image)

**CCV1**

- **Mean**: 106.99
- **StDev**: 45.58
- **LCL**: -29.74
- **LWL**: 15.84
- **UWL**: 198.14
- **UCL**: 243.72
- **MSLR**: 80-120

**Silica CCV1 Recovery**

![Silica CCV1 Recovery](image)

**CCV2**

- **Mean**: 95.75
- **StDev**: 12.03
- **LCL**: 59.65
- **LWL**: 71.68
- **UWL**: 119.82
- **UCL**: 131.85
- **MSLR**: 80-120

**Silica CCV2 Recovery**

![Silica CCV2 Recovery](image)
**LCS**

Mean = 94.05  
StDev = 6.44  
LCL = 74.72  
LWL = 81.17  
UWL = 106.94  
UCL = 113.39  
MSLR = 80-120

**Silica LCS Recovery**

![Silica LCS Recovery Chart]

**Spikes**

Mean = 97.50  
StDev = 9.28  
LCL = 69.65  
LWL = 78.94  
UWL = 116.07  
UCL = 125.35  
MSLR = 80-120

**Silica Spike Recovery**

![Silica Spike Recovery Chart]

**Duplicates**

Mean = 0.60  
StDev = 10.39  
LCL = -30.56  
LWL = -20.18  
UWL = 21.37  
UCL = 31.76  
MSLR = 80-120

**Silica Duplicate Deviation**

![Silica Duplicate Deviation Chart]
Figure D-13  Bioavailable Phosphorus Control Charts

**CCV1 %**
- Mean = 107.69
- StDev = 52.24
- LCL = -49.02
- LWL = 3.22
- UWL = 212.16
- UCL = 264.40
- MSLR = 80-120

**CCV2 %**
- Mean = 100.17
- StDev = 6.07
- LCL = 81.97
- LWL = 88.04
- UWL = 112.31
- UCL = 118.38
- MSLL = 80-120

**LCS %**
- Mean = 97.85
- StDev = 6.17
- LCL = 79.34
- LWL = 85.51
- UWL = 110.19
- UCL = 116.36
- MSLL = 80-120
### Spikes %

- **Mean:** 94.89
- **StDev:** 21.00
- **LCL:** 31.88
- **LWL:** 52.89
- **UWL:** 136.90
- **UCL:** 157.90
- **MSLL:** 80-120

![BAP Spike Recovery Graph]

### Duplicates %

- **Mean:** 1.68
- **StDev:** 20.63
- **LCL:** -60.20
- **LWL:** -39.57
- **UWL:** 42.94
- **UCL:** 63.56
- **MSLL:** 80-120

![BAP Duplicate Deviation Graph]