Environmental Technology Verification Program Advanced Monitoring Systems Center

Test/QA Plan for Verification of Nutrient Analyzers at a Wastewater Treatment Plant



TEST/QA PLAN

for

Verification of Nutrient Analyzers at a Wastewater Treatment Plant

April 22, 2005

Prepared by

Battelle 505 King Avenue Columbus, OH 43201-2693

Nutrient Analyzers Test/QA Plan Page 2 of 52 Version 1.0 April 22, 2005

A2 TABLE OF CONTENTS

Section

А	PROJECT MANAGEMENT	
A1	Title Page	1
A2	Table of Contents	2
A3	Distribution List	5
A4	Verification Test Organization	6
A5	Background 1	3
A6	Verification Test Description and Schedule 1	4
A7	Quality Objectives	1
A8	Special Training/Certification	2
A9	Documentation and Records 2	2
В	MEASUREMENT AND DATA ACQUISITION	
B1	Experimental Design	4
B2	Sampling Requirements	8
B3	Sample Handling and Custody Requirements	9
B4	Laboratory Reference Methods 4	0
B5	Quality Control	2
B6	Instrument/Equipment Testing, Inspection, and Maintenance 4	2
B7	Instrument Calibration and Frequency 4	2
B8	Inspection/Acceptance of Supplies and Consumables 4	3
B9	Non-Direct Measurements 4	3
B10	Data Management	4
С	ASSESSMENT AND OVERSIGHT	
C1	Assessments and Response Actions 4	7
C2	Reports to Management 4	9
D	DATA VALIDATION AND USABILITY	
D1	Data Review, Validation, and Verification Requirements 5	1
D2	Validation and Verification Methods 5	1
D3	Reconciliation with User Requirements 5	1
E	REFERENCES	2
	APPENDIX A A-	1

Page

List of Figures

<u>Page</u>

Figure 1	Organization Chart	7
Figure 2	Generalized Test Site Layout	20

List of Tables

Table 1	Planned Verification Schedule	16
Table 2	Planned Weekly Test Activities During the Field Period	18
Table 3	Effluent Characteristics at the Test Site	19
Table 4	Verification Test Performance Parameters	25
Table 5	Approximate Nutrient Concentrations and Suggested Order for	
	Multi-point Challenges	27
Table 6	Additional Standards for Total Nitrogen and Total Phosphorus Analyzers	28
Table 7	Approximate Sample Specifications for Off-line Evaluation of Matrix Effects	31
Table 8	Nutrient Sample Summary	33
Table 9	Reference Method Sample Summary	39
Table 10	Reference Methods	41
Table 11	Summary of Data Recording Process	45

Nutrient Analyzers Test/QA Plan Page 4 of 52 Version 1.0 April 22, 2005

ETV Advanced Monitoring Systems Center

Test/QA Plan for Verification of Nutrient Analyzers at a Wastewater Treatment Plant

Version 1.0

April 22, 2005

VENDOR ACCEPTANCE:

Name _____

Company _____

Date _____

Nutrient Analyzers Test/QA Plan Page 5 of 52 Version 1.0 April 22, 2005

A3 DISTRIBUTION LIST

Ms. Elizabeth A. Betz U.S. Environmental Protection Agency-HEASD National Exposure Research Laboratory E205-01 USEPA Mailroom Research Triangle Park, NC 27711

Mr. Robert Fuerst U.S. Environmental Protection Agency-HEASD National Exposure Research Laboratory D205-05 USEPA Mailroom Research Triangle Park, NC 27711

Verification Test Collaborators: Mr. Kenneth N. Wood DuPont Company Brandywine Building 9212 1007 Market Street Wilmington, DE 19898

Mr. L. Waverly Smith DuPont - Spruance Plant Spot No. 912 5401 Jefferson Davis Highway Richmond, VA 23234

Ms. Karen Riggs Dr. Ann Louise Sumner Ms. Amy Dindal Mr. Michael Rectanus Mr. Zachary Willenberg Battelle 505 King Ave. Columbus, OH 43201 Nutrient Analyzer Vendor Dr. Robert Clifford Shimadzu Scientific Instruments, Inc. 7102 Riverwood Drive Columbia, MD 21046

Nutrient Analyzer Vendor Mr. F. Joseph Bussell ZAPS Technologies 131 NW 4th Street Corvallis, OR 97330

SECTION A

PROJECT MANAGEMENT

A4 VERIFICATION TEST ORGANIZATION

The verification test will be conducted under the auspices of the U.S. Environmental Protection Agency (EPA) through the Environmental Technology Verification (ETV) Program. It will be performed by Battelle, which is managing the ETV Advanced Monitoring Systems (AMS) Center through a cooperative agreement with EPA. The scope of the AMS Center covers verification of monitoring technologies for contaminants and natural species in air, water, and soil.

The day to day operations of this verification test will be coordinated and supervised by Battelle personnel, with the participation of the vendors who will be having the performance of their nutrient analyzers verified. The testing will be conducted in collaboration with DuPont Company at their industrial wastewater treatment facility at the Spruance Plant in Richmond, Virginia. Staff from DuPont and their contractor, Operations Management International, Inc. (OMI), will support this test by helping to install the analyzers to be tested, providing infrastructure at the test site, and overseeing operation of the analyzers during periods of routine operation. Each analyzer vendor will install their respective analyzer, operate the analyzer through portions of the test (unless they give written consent for Battelle, DuPont, and/or OMI staff to operate it), and repair or maintain their analyzer during the test. Reference measurements of nutrients in wastewater and quality control samples will be carried out by a local commercial laboratory as coordinated by DuPont. Quality Assurance (QA) oversight will be provided by the Battelle Quality Manager and also by the EPA AMS Center Quality Manager, at her discretion. The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with the verification test. Roles and responsibilities are defined further below.

Nutrient Analyzers Test/QA Plan Page 7 of 52 Version 1.0 April 22, 2005



Figure 1. Organization Chart

A4.1 Battelle

<u>Dr. Ann Louise Sumner</u> is the AMS Center's Verification Test Coordinator for this test. In this role, Dr. Sumner will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, Dr. Sumner will:

- Prepare the draft test/QA plan, verification reports, and verification statements.
- Establish a budget for the verification test and manage staff to ensure the budget is not exceeded.
- Revise the draft test/QA plan, verification reports, and verification statements in response to reviewers' comments.
- Assemble a team of qualified technical staff to conduct the verification test.
- Direct the team (Battelle, DuPont, and OMI) in performing the verification test in accordance with this test/QA plan.
- Hold a kick-off meeting approximately one week prior to the start of the verification test to review the critical logistical, technical, and administrative aspects of the verification test. Responsibility for each aspect of the verification test will be confirmed.
- Ensure that all quality procedures specified in this test/QA plan and in the AMS Center Quality Management Plan¹ (QMP) are followed.
- Serve as the primary point of contact for vendor representatives.
- Ensure that confidentiality of sensitive vendor information is maintained.
- Participate in required safety training at the DuPont test site.
- Assist vendors as needed during the analyzer installation and verification testing.
- Become familiar with the operation and maintenance of the nutrient analyzers through instruction by the vendors, if needed.
- Prepare nutrient samples and perform testing activities and data acquisition on the nutrient analyzers as specified in this test/QA plan.
- Perform sample collection as detailed in this test/QA plan and follow procedures set forth by DuPont for submission of samples to the reference laboratory for analysis.

- Respond to any issues raised in assessment reports, audits, or from Battelle, DuPont, or OMI test staff observations, and institute corrective action as necessary.
- Coordinate distribution of the final test/QA plan, verification reports, and verification statements.

<u>Ms. Amy Dindal</u> is a Verification Testing Leader for the AMS Center. As such, Ms. Dindal will provide technical guidance and oversee the various stages of verification testing. She will:

- Support Dr. Sumner in preparing the test/QA plan and organizing the testing.
- Review the draft and final test/QA plan.
- Attend the verification test kick-off meeting.
- Review the draft and final verification reports and verification statements.

Ms. Karen Riggs is Battelle's manager for the AMS Center. Ms. Riggs will:

- Review the draft and final test/QA plan.
- Review the draft and final verification reports and verification statements.
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test.
- Ensure that confidentiality of sensitive vendor information is maintained.
- Support Dr. Sumner in responding to any issues raised in assessment reports and audits.
- Maintain communication with EPA's technical and quality managers.
- Issue a stop work order if Battelle or EPA QA staff discovers adverse findings that will compromise test results.

<u>Battelle Technical Staff</u> will support Dr. Sumner in planning and conducting the verification test. The responsibilities of the technical staff will be to:

• Assist in planning for the test, and making arrangements for the installation of the analyzers.

- Attend the verification test kick-off meeting.
- Participate in required safety training at the DuPont test site if working at the test site.
- Assist vendor staff as needed during the analyzer installation and verification testing.
- Perform testing activities and data acquisition on the nutrient analyzers as specified in this test/QA plan.
- Perform sample collection as detailed in this test/QA plan and follow procedures set forth by DuPont for submission of samples to the reference laboratory for analysis.
- Report any observed testing activities that deviate from this test/QA plan to the Verification Test Coordinator and assist in resolving issues as needed.
- Perform statistical calculations specified in this test/QA plan on the nutrient analyzer data as needed.
- Provide results of statistical calculations and associated discussion for the verification reports as needed.
- Support Dr. Sumner in responding to any issues raised in assessment reports and audits related to statistics and data reduction as needed.

<u>Mr. Zachary Willenberg</u> is Battelle's Quality Manager for the AMS Center. Mr. Willenberg will:

- Review the draft and final test/QA plan.
- Attend the verification test kick-off meeting.
- Participate in safety training at the DuPont test site, if required.
- Conduct a technical systems audit once during the verification test, or designate other QA staff to conduct the audit.
- Audit at least 10% of the verification data or designate other QA staff to conduct the data audit.
- Prepare and distribute an assessment report for each audit.
- Verify implementation of any necessary corrective action.

- Request that Battelle's AMS Center Manager issue a stop work order if audits indicate that data quality is being compromised.
- Provide a summary of the QA/QC activities and results for the verification reports.
- Review the draft and final verification reports and verification statements.
- Assume overall responsibility for ensuring that the test/QA plan is followed.

A4.2 Nutrient Analyzer Vendors

The responsibilities of the nutrient analyzer vendors are as follows:

- Review and provide comments on the draft test/QA plan.
- Accept (by signature of a company representative) the final test/QA plan prior to test initiation (see page 4).
- Provide a nutrient analyzer for evaluation during the verification test.
- Provide all other equipment/supplies/reagents/consumables needed to operate their analyzer for the duration of the verification test.
- Participate in required safety training at the DuPont test site before installation of the nutrient analyzer.
- Supply a representative to install and maintain their technology, and to operate it in portions of the test specified in this test/QA plan, or provide written consent and instructions for Battelle, DuPont, and OMI staff to carry out these activities.
- Provide written instructions for routine operation of their analyzer, including a daily checklist of diagnostic and/or maintenance activities (see Section B1).
- Provide maintenance and repair support for their analyzer, on-site if necessary, throughout the duration of the verification test.
- Review and provide comments on the draft verification report and statement for their respective analyzer.

Nutrient Analyzers Test/QA Plan Page 12 of 52 Version 1.0 April 22, 2005

A4.3 EPA

EPA's responsibilities in the AMS Center are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (EPA QMP).² The roles of specific EPA staff are as follows:

<u>Ms. Elizabeth Betz</u> is EPA's AMS Center Quality Manager. For the verification test, Ms. Betz will:

- Review the draft test/QA plan.
- Perform at her option one external technical systems audit during the verification test.
- Notify the EPA AMS Center Manager of the need for a stop work order if the external audit indicates that data quality is being compromised.
- Prepare and distribute an assessment report summarizing results of the external audit.
- Review draft verification reports and verification statements.

Mr. Robert Fuerst is EPA's manager for the AMS Center. Mr. Fuerst will:

- Review the draft test/QA plan.
- Approve the final test/QA plan.
- Review the draft verification reports and verification statements.
- Oversee the EPA review process for the test/QA plan, verification reports, and verification statements.
- Coordinate the submission of verification reports and verification statements for final EPA approval.

A4.4 DuPont Company

This test will be conducted in collaboration with DuPont, who will provide in-kind support and a site for this test. The responsibilities of personnel from DuPont and their contractor, OMI, include the following:

- Coordinate use of the test site for the purposes of ETV testing, assuring access to the test site for Battelle and vendor staff.
- Attend the verification test kick-off meeting.
- Support the test by providing space and needed utilities (e.g., instrument shelter, electricity, deionized (DI) water, compressed air) for the nutrient analyzers during testing.
- Provide on-site health and safety training to Battelle, EPA, and vendor staff prior to their conducting work at the test site.
- Assist Battelle and vendor staff in the installation, operation, testing, and removal of the nutrient analyzers at the test site.
- Perform sample collection and arrange for sample transport to and analysis by the commercial reference laboratory, Froehling and Robertson, Inc. (Richmond, Virginia), as detailed in this test/QA plan.
- As needed, perform testing activities on the nutrient analyzers specified in this test/QA plan.
- Provide daily oversight of the nutrient analyzers during periods of routine operation, checking diagnostic indicators according to vendor directions and contacting Battelle if faults in analyzer operation are observed.
- Record observations about the maintenance and operation of the nutrient analyzers during the on-line testing period.
- Obtain the nutrient sample results from the reference laboratory and provide a data package to Battelle that includes all sampling data sheets, analysis records, calibration data, and QA information, and that presents the nutrient sample analysis results, QA results, and calculated nutrient concentrations.
- Review the draft test/QA plan, verification reports, and verification statements.

A5 BACKGROUND

The ETV Program's AMS Center conducts third-party performance testing of commercially available technologies that detect or monitor natural species or contaminants in air, water, and soil. Stakeholder committees of buyers and users of such technologies recommend technology categories, and technologies within those categories, as priorities for testing. Nutrient analyzers for wastewater monitoring were identified as a priority technology category through the AMS Center stakeholder process.

A6 VERIFICATION TEST DESCRIPTION AND SCHEDULE

A6.1 Summary of Technology Category

The nutrient analyzers to be tested during this verification test consist of on-line instruments that can perform continuous monitoring of nutrient concentrations in industrial and/or municipal wastewater. Nutrient analyzers are typically capable of determining concentrations of at least one of the following nutrients: nitrate, nitrite, ammonia, total nitrogen (TN), phosphate, and/or total phosphorus (TP). The nutrient analyzers that will be tested in this verification test are capable of determining nitrate, TN, and/or TP concentrations; therefore, this test/QA plan is limited to the verification of nutrient analyzers for nitrate, TN,^a and TP.^b

High nutrient concentrations are a leading cause of impairment of lakes, rivers, and estuaries in the U.S. Over-enrichment of nitrogen and phosphorus can lead to reductions in water quality including harmful algal blooms, hypoxia, and declines in wildlife habitat, and may increase human pathogen levels. Because of the differing characteristics of various water bodies, region-specific nutrient criteria are being developed under the guidance of the U.S. EPA to reduce nutrient loading of the aquatic environment. On-line monitoring of nutrients in wastewater is a critical component in the reduction of nutrient loading. It is recognized that the composition of wastewater will vary considerably depending upon the sampling location, source, and/or treatment methods. Use of a wastewater from the DuPont Plant as a sample matrix in this

^a TN is defined analytically as the sum of total Kjeldahl nitrogen (TKN), nitrate, and nitrite analyses.

^bTP is defined analytically as the sum of measured organic, reactive, and acid hydrolyzable phosphorus.

test will provide a single example (not a comprehensive overview) of possible matrix effects associated with wastewater monitoring.

The analyzers that will be evaluated in this verification test are stand-alone, automated instruments that continuously or routinely determine nutrient concentrations in wastewater for extended periods without user intervention. However, a number of analytical approaches may be utilized to determine nutrient concentrations, ranging from direct spectroscopic detection to oxidation and chemical reaction with colorimetric detection of the product. In performing the verification test, Battelle will follow the technical and QA procedures specified in this test/QA plan and will comply with the data quality requirements in the AMS Center QMP.¹

A6.2 Verification Schedule

Table 1 shows the planned schedule of activities in field testing and data analysis/reporting in this verification test. As shown in Table 1, the field test of nutrient analyzers is planned to begin in May 2005 with installation of the analyzers at the DuPont wastewater treatment plant, and extend into June 2005. The period of operation of the analyzers at the facility will be approximately 6 weeks, during which time the analyzers will measure and record nutrient concentrations in the final combined plant effluent (on-line monitoring phase) or in prepared samples (off-line testing phase). At the beginning of the field period (Off-line Phase I), the analyzers will be challenged with prepared nutrient standards and other samples; at the end of the field period (Off-line Phase II), the analyzers will be challenged with various matrix samples, such as plant influent or process water. Aliquots of the prepared samples and plant effluent will also be collected throughout the field period and analyzed by a reference laboratory for the nutrients of interest. Subsequent to the field testing, a separate verification report for each analyzer will be drafted, reviewed, revised, and submitted to EPA for final signature.

Nutrient Analyzers Test/QA Plan Page 16 of 52 Version 1.0 April 22, 2005

Month	Test Activity			
(2005)	Analyzer Field Activities	Data Analysis and Reporting		
May	Set up/install nutrient analyzers Analyzer training by vendors Off-line Phase I testing On-line effluent monitoring Nutrient reference sampling	Begin preparation of report template Analysis of nutrient samples Compile data from off-line testing		
June	On-line effluent monitoring Nutrient reference sampling Off-line Phase II matrix challenges Remove analyzers from test site	Review and summarize operator observations Compile data from on-line monitoring Compile data from matrix challenges Analysis of nutrient samples Compile data from reference laboratory		
July		Complete summary of operator observations Finalize data from on-line and off-line testing Finalize results for reference analyses Complete common sections of reports Complete draft reports Internal review of draft reports Vendor review of draft reports		
August		Complete vendor review of draft reports Revision of draft reports Peer review of draft reports		
September		Revision of draft reports Submission of final reports for EPA approval		

Table 1. Planned Verification Schedule

Table 2 shows the activities to be conducted in each week of the test during the field period in May and June, 2005. The test procedures are described in Section B of this test/QA plan. Multi-point challenges with nutrient standards will be conducted during Off-line Phase I testing to address analyzer measurement accuracy, bias, and linearity. Repeated challenges with the same nutrient standard will be conducted to evaluate analyzer reproducibility. The nutrient analyzer responses to multiple challenges with DI water will be used to determine the analyzer limit of detection (LOD) with respect to each nutrient detected by that analyzer. Repeated challenges with DI water and nutrient standards will be conducted early in the field period to obtain the analyzer "baseline" responses. The same challenges with DI water and nutrient standards will be conducted once each week to address analyzer span and zero drift. Single

Nutrient Analyzers Test/QA Plan Page 17 of 52 Version 1.0 April 22, 2005

nutrient standard solutions and mixtures of nitrogen species and/or phosphorus species will be delivered to the nitrate analyzers to evaluate interference effects (specificity). Matrix effects will be evaluated from each analyzer's response to nutrients in the presence of chlorophyll *a*, at three pH levels, and in wastewater samples collected from the influent, treatment process, and effluent streams. Samples will be collected and analyzed by a reference method throughout the field period, for comparison of the analyzer response to the nutrient concentrations in prepared standards and wastewater samples. For approximately four weeks of the field period, the analyzers will routinely monitor nutrients in the effluent stream to allow assessment of operational factors and data completeness under continuous operation and to collect additional matrix effects samples.

Week of (Planned Month and Day, 2005)	5) Test Activities		
May 2	 Install nutrient analyzers Establish inlet connections Training of DuPont, OMI, and Battelle staff by vendor representatives Conduct trial operations Off-line Phase I testing Nutrient standard challenges for reproducibility, span and zero drift, and interference effects DI water challenges for LOD determination 		
May 9	 Off-line Phase I testing Nutrient standard and DI water challenges for linearity, accuracy, bias, span and zero drift pH-adjusted nutrient standard challenges for matrix effects Chlorophyll <i>a</i> challenges for matrix effects DI water challenges for LOD determination 		
May 16	 On-line effluent monitoring Nutrient standard and DI water challenges for span and zero drift Three nutrient samples collected, submitted for analysis 		
May 23	 On-line effluent monitoring Nutrient standard and DI water challenges for span and zero drift Three nutrient samples collected, submitted for analysis 		
May 30	 On-line effluent monitoring Nutrient standard and DI water challenges for span and zero drift Three nutrient samples collected, submitted for analysis 		
June 6	 On-line effluent monitoring Nutrient standard and DI water challenges for span and zero drift Three nutrient samples collected, submitted for analysis 		
June 13	 Off-line Testing Phase II Nutrient standard and DI water challenges for span and zero drift Plant influent water challenges for matrix effects Plant process water challenges for matrix effects Plant effluent water challenges for matrix effects Remove analyzers from test site 		

Table 2. Planned Weekly Test Activities During the Field Period

A6.3 Test Site

The host facility for the nutrient analyzer verification test will be the DuPont Spruance Plant in Richmond, Virginia. At this plant, DuPont engineers polymers/plastics and fibers (e.g. NOMEX® flame retardant and KEVLAR®). The waste treatment processes utilized at the plant include aerated lagoons and a polishing pond. Flow leaving the polishing pond joins a flow of non-contact cooling water from the plant in an open drainage ditch that empties into the James River. Table 3 summarizes the characteristics of this combined effluent stream (final outfall) at the Spruance plant. The generalized layout of the test site with respect to the treatment facility is shown in Figure 2 (not drawn to scale). The site for testing will be located along the combined final outfall stream. The nutrient analyzers will be installed inside a shelter that will provide protection from precipitation and direct sunlight; the shelter may be temperature-regulated if needed. The final effluent will be pumped out of the open drainage ditch into the analyzer shelter(s) and delivered to the nutrient analyzers at the required flow rate or pressure.

Parameter	Typical Range
Temperature (°C)	3 - 36
Total organic carbon (ppm)	10-30
Total suspended solids (ppm)	10 - 50
рН	6.3 - 9.0
Total Nitrogen (mg N/L) ^a	1 – 13
Nitrate-N (mg N/L) ^a	0.1 - 5
Nitrite-N (mg N/L)	< 0.1 - 0.5
Ammonia-N (mg N/L)	0.2 - 1
Total Phosphorus (mg P/L) ^a	0.1 - 0.4
Inorganic Phosphorus (mg P/L)	0.02 - 0.4

Table 3. Effluent Characteristics at the Test Site

^a Measurements of this nutrient will be verified for at least one submitted technology as a part of this verification test.



Figure 2. Generalized Test Site Layout

A6.4 Health and Safety

The verification test described in this test/QA plan will be performed at the DuPont Spruance Plant in Richmond, Virginia. All participants in this verification test (i.e., Battelle, DuPont, OMI, EPA, and vendor staff) will adhere to the health and safety requirements of the test facility. Those requirements include completion of a safety orientation before participation in any activities at the facility. DuPont may require a criminal background check and drug screening for verification test participants who will be working at the DuPont site. Vendor staff will only install and operate their nutrient analyzers during the verification test; they are not permitted to perform any other verification activities identified in this test/QA plan. Operation of the nutrient analyzers themselves does not pose any known fire, mechanical, electrical, noise, or other potential hazard. Material Safety Data Sheets (MSDS) will be provided by the vendor for any chemicals or reagents needed to operate their nutrient analyzer and all test participants will follow appropriate safety precautions during chemical and reagent handling. MSDSs for all chemicals used in this verification test will be present at the test site; all test participants will review the MSDSs.

All staff visiting the DuPont test site will be given a safety briefing prior to conducting work at the test site, including installation and operation of the nutrient analyzers. This briefing will include a description of emergency operating procedures (i.e., in case of fire, tornado, laboratory or plant accident) and identification, location, and operation of safety equipment (e.g., fire alarms, fire extinguishers, eye washes, exits). All staff working at the test site must follow DuPont Spruance Site Conditions, included as Appendix A of this test/QA plan. Non-compliance with DuPont Spruance Site Conditions or other DuPont regulations may warrant removal of personnel from the premises.

A7 QUALITY OBJECTIVES

This verification test will evaluate the performance of analyzers for determining nutrients in wastewater at an industrial treatment facility. This evaluation will include a comparison of the analyzer results to results of sample analyses by a reference laboratory. The standard QA/QC procedures for the reference laboratory will include analysis of the following quality control samples (QCS): initial calibration verifications (ICV), initial calibration blanks (ICB), method blanks (MB), lab control standards (LCS), matrix spikes (MS), matrix spike duplicates (MSD), continuing calibration verifications (CCV), and continuing calibration blanks (CCB). Results of all QCSs will be included in the verification test documentation and must fall within the laboratory's quality requirements. The quality of the reference measurements will also be monitored by inclusion of performance evaluation (PE) audit samples submitted as blind (i.e., unknown) samples to the reference laboratory. The PE audit samples will be prepared from certified aqueous nutrient standards that are independent of those used for reference method calibration and will include prepared standards and/or matrix spikes. These samples are meant to independently confirm that the reference measurements are being performed correctly and are producing accurate results. Control limits on the PE audit samples are given in Section C1. The Battelle Quality Manager or his designate will perform a technical systems audit (TSA) at least once during this verification test and will audit at least 10% of the verification data acquired in this verification test. The EPA Quality Manager also may conduct an independent TSA, at her discretion.

A8 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, field testing, data analysis, and reporting is maintained for all Battelle, DuPont, and OMI technical staff in training files at the respective locations. The Battelle Quality Manager may verify the presence of appropriate training records prior to the start of testing. If Battelle, DuPont, or OMI staff operate and/or maintain an analyzer during the verification test, the analyzer vendor will be required to train those staff prior to the start of testing. Battelle will document this training with a consent form, signed by the vendor, that states which specific Battelle, DuPont, and OMI staff have been trained on their analyzer. Battelle technical staff will have a minimum of a bachelor's degree in science/engineering or have equivalent work experience.

A9 DOCUMENTATION AND RECORDS

The records for this verification test will include the test/QA plan, chain-of-custody forms, laboratory record books (LRB), data collection forms, electronic files (both raw data and spreadsheets), and the final verification reports and verification statements. All of these records will be maintained in the Verification Test Coordinator's office or at the test site during the test and will be transferred to permanent storage at Battelle's Records Management Office at the conclusion of the verification test. The location (e.g., specific personal computer, server, or media type and storage location) of final versions of the electronic files will be noted in the test records. All Battelle LRBs are stored indefinitely, either by the Verification Test Coordinator or Battelle's Records Management Office. EPA will be notified before disposal of any files. The

QA/QC documentation and results of the nutrient reference measurements made by the commercial laboratory will be submitted to Battelle immediately upon completion of all sample analyses and maintained with the records for this test. Section B10 further details the data recording practices and responsibilities.

All written records must be in ink. Any corrections to notebook entries, or changes in recorded data, must be made with a single line through the original entry. The correction is then to be entered, initialed, and dated by the person making the correction. In all cases, strict confidentiality of data from each vendor's analyzer, and strict separation of data from different analyzers, will be maintained. Separate files (including manual records, printouts, and/or electronic data files) will be kept for each analyzer.

SECTION B MEASUREMENT AND DATA ACQUISITION

B1 EXPERIMENTAL DESIGN

This test will specifically address verification of nutrient analyzers under the conditions of an industrial wastewater treatment plant by evaluating the accuracy, bias, linearity, LOD, and selectivity of nutrient measurements made by each analyzer in wastewater and/or synthetic nutrient mixtures, the extent of baseline and calibration drift, as well as the ability of the analyzers to perform continuous monitoring with minimal intervention. The reproducibility of the analyzer measurement data while sampling synthetic nutrient standards will also be determined. Specifically, the nutrient analyzers will be evaluated for the performance parameters summarized in Table 4 and discussed in detail in the following section.

In addition to the testing activities specified in this test/QA plan, Battelle, DuPont, and/or OMI staff will perform regular maintenance and other routine procedures requested by the vendor for their analyzer. These activities will be summarized by the vendor in a "daily checklist" that will include specific instructions and frequency for each regular maintenance activity or routine procedure, any diagnostic values that should be recorded and typical or acceptable ranges for those values, vendor contact information, and a space for Battelle, DuPont, and/or OMI staff to sign and date the form daily upon completion of the items on the checklist.

Performance Parameter	ce Method of Evaluation		
Accuracy	Closeness of analyzer response to nutrient standards compared to reference method value		
Bias	Systematic error in analyzer response to nutrient standards compared to reference method value		
Linearity	Analyzer response to nutrient standards compared to reference value		
Limit of Detection	Calculated from analyzer response to repeated measurements of DI water		
Reproducibility	Percent relative standard deviation (%RSD) of repeated analyses of the same nutrient standard		
Span and Zero Drift	Stability of analyzer response to DI water and nutrient standards over time		
Interference Effects	Analyzer response to non-nitrate compounds containing nitrogen and phosphorus compared to reference value (only for nitrate analyzers)		
Matrix Effects	Analyzer response to nutrients in several sample types (chlorophyll <i>a</i> , varied pH, wastewater) compared to reference value		
Data Completeness	Percentage of maximum data return over field period		
Operational Factors	Operator observations, records of needed and performed maintenance, vendor activities, use of expendable supplies		

Table 4. Verification Test Performance Parameters

B1.1 Test Procedures

The following sections describe the test procedures that will be used to evaluate each of the nutrient analyzer performance parameters listed in Table 4. The test will have two components: off-line testing and on-line effluent monitoring. Procedures during the off-line testing phases will be conducted on each analyzer separately. During the on-line effluent monitoring phase, one or more manifolds will be used to simultaneously deliver the final outfall to each analyzer, e.g., by connecting each analyzer to a multi-spigot tap that will include ports for collection of grab samples and to monitor general water quality parameters. If available, water quality parameters measured at the same site by DuPont and OMI may be used in lieu of monitoring at the sample collection point.

Nutrient solutions will be prepared from certified standards or high-purity solids (e.g., potassium nitrate, potassium dihydrogenphosphate) in DI water or wastewater (influent, process, and effluent) samples. Depending on the number of nutrient analyzer measurements that will be conducted with each solution, the total volume prepared for each nutrient solution will vary from 5 liters (L) to more than 25 L. It is not required that the nutrient solutions be prepared quantitatively since all evaluations of analyzer performance specified in this test/QA plan will utilize the reference laboratory analysis result for each prepared solution, rather than the nominal concentration calculated from the sample preparation. However, the solutions will be prepared as close to the target concentrations outlined in this test/QA plan as is feasible.

B1.1.1 Accuracy, Bias, Linearity, and Limit of Detection

During Off-line Phase I testing, the analyzers will be supplied with nutrient standards and DI water. Each analyzer will be challenged only with the nutrient(s) for which it was submitted for verification. Samples will be supplied to the analyzers by immersing the sample inlets in the nutrient standard solution or gravity-feeding the solution into the calibration inlet.

For each nutrient, three non-consecutive measurements will be recorded at each of five different nominal concentrations (including zero); the three measurements at each concentration need not be conducted on the same day but should be conducted within a span of three days if possible. For continuous analyzers, each nutrient standard solution will be supplied for no less than five minutes. Non-continuous analyzers will be supplied with each nutrient standard solution for the duration of the sample intake period (~6 seconds). Table 5 shows the approximate concentration values for each nutrient to be supplied to the analyzers being tested and the suggested order in which the concentrations may be supplied. Upon the completion of testing each day, analyzers will sample DI water for at least five minutes to obtain an additional blank measurement. Analyzers submitted for TN will be challenged with additional standards containing nitrite, ammonia, organic nitrogen, and a mixture of several nitrogen-containing species. Analyzers submitted for TP will also be challenged with an organic phosphorus standard and a mixture of inorganic and organic phosphorus compounds. Nutrient species and

approximate concentrations for these samples are listed in Table 6. For these additional samples, only analyzer accuracy will be evaluated.

N f	Nutrient Concentration				
Sequence	TN (mg N/L) ^a	TP (mg P/L) ^b	$NO_3^{-}-N (mg N/L)^a$		
0	0	0	0		
1	0.5	0.5	0.1		
2	5	1	0.5		
3	10	3	2		
4	20	5	5		
5	0	0	0		
6	10	3	2		
7	5	1	0.5		
8	20	5	5		
9	0.5	0.5	0.1		
10	0	0	0		
11	20	5	5		
12	10	3	2		
13	5	1	0.5		
14	0.5	0.5	0.1		
15	0	0	0		

 Table 5. Approximate Nutrient Concentrations and Suggested Order for Multi-point

 Challenges

^a Nitrogen in the form of nitrate (e.g. from potassium nitrate).

^b Phosphorus in the form of phosphate (e.g., from potassium dihydrogenphosphate)

Nutrient	Nutrient Source	Nitrogen Concentration (mg N/L)	Phosphorus Concentration (mg P/L)
	Ammonia	5	
	Nitrite	5	
	Nitrate	5	
TN	Organic nitrogen	5	
	Approximately equally apportioned mixed nitrogen sample (ammonia, nitrite, nitrate, organic nitrogen)	10	
	Inorganic phosphorus		3
	Organic phosphorus		3
TP	Approximately equally apportioned mixed phosphorus sample (inorganic phosphorus and organic phosphorus)		5

Table 6. Additional Standards for Total Nitrogen and Total Phosphorus Analyzers

The analyzer response to the series of nutrient standards listed in Table 5 will be used to evaluate accuracy, bias, and linearity. Section B1.2 presents the statistical procedures that will be used. Accuracy will be calculated at each concentration and for each replicate relative to the reference laboratory value for the nutrient concentration. Bias will be calculated once for the full set of multi-point nutrient measurements. Linearity will be assessed by establishing a multi-point calibration curve from the analyzer response versus the reference laboratory value. For TN and TP, accuracy will also be calculated for the samples summarized in Table 6. The average and standard deviation of analyzer blank (DI water) measurements conducted throughout both off-line testing phases will be used to calculate the LOD for each nutrient measured by each analyzer.

B1.1.2 Reproducibility

The reproducibility of each analyzer for measurement of a mid-level nutrient standard will be evaluated during Off-line Phase I testing. Each analyzer will be challenged with a mixed standard containing nitrate and phosphate at approximately 5 mg N/L and 3 mg P/L, respectively, at least five successive times. For continuous analyzers, the nutrient standard will be delivered five times in succession for at least 5 minutes each time. Non-continuous analyzers will sample the appropriate nutrient standard five times sequentially.

B1.1.3 Span and Zero Drift

The "baseline" response of each analyzer to nutrient standard and DI water samples will be determined early in the verification test. The mean and standard deviation of the analyzer response to the mid-level nutrient standards supplied for the reproducibility checks (B1.1.2) will be calculated from the five replicates for each analyzer and each verified nutrient. Similarly, the mean and standard deviation of the analyzer response to DI water will be calculated from the first five DI water measurements obtained during the verification test.

Once each week during the verification test, DI water and the mid-level nutrient standard will again be supplied to each of the analyzers being tested for a total of six zero/span checks. Each response will be compared to the baseline response to determine whether or not drift has occurred in the analyzer response to DI water or the nutrient standards.

B1.1.4 Interference Effects

Interference effects will be evaluated only for the nitrate analyzers; interference effect evaluation will not be conducted on TN and TP analyzers since they are expected to detect all of the nitrogen- and phosphorus-containing species, respectively. During Off-line Phase I testing, nitrate analyzers will be challenged with standards containing non-nitrate nutrients. The additional TN standards listed in Table 6 will be sampled by the nitrate analyzers for at least 5 minutes each.

Nutrient Analyzers Test/QA Plan Page 30 of 52 Version 1.0 April 22, 2005

B1.1.5 Matrix effects

B1.1.5a Off-Line Testing Phase

During Off-line Phase I and Phase II testing, the analyzers will be challenged with a series of samples containing altered matrices that may affect the analyzer accuracy. In Phase I, synthetic samples containing nutrients at three pH levels and in the presence and absence of chlorophyll *a* (present in algal blooms) will be prepared in DI water and delivered to each analyzer. In Phase II, wastewater samples will be collected from three points in the treatment process streams – influent, process, and treated effluent – and delivered to each analyzer both unspiked and after spiking with nitrate and/or phosphate. Off-line analysis of the Phase II wastewater samples will be conducted after completion of the on-line final outfall monitoring to minimize the potential impact of analyzer fouling from potentially high total suspended solids levels in the influent samples. Table 7 summarizes the samples and approximate composition that will be delivered to each analyzer to assess matrix effects. Continuous analyzers will measure each sample for at least 5 minutes. Analyzer responses will be compared to reference laboratory analysis results for the same samples to determine the percent recovery (%R) for each sample. The dependence of the %R results on matrix type will indicate any matrix effects.

Matrix	Samela Danaiatian	Number of	Target Nutrient and Spike Concentration		
(Variable)	Sample Description	Replicates	TN and Nitrate Nitrogen (mg/L)	TP Phosphorus (mg/L)	
	pH = 5	1	5	3	
pH	pH = 7	1	5	3	
	pH = 9	1	5	3	
Chlorophall	120 microgram (μg)/L Chlorophyll <i>a</i>	1	0	0	
(pH 7)	0 μg/L Chlorophyll a	1	5	3	
	120 µg/L Chlorophyll a	1	5	3	
Influent comple	Influent	3	0	0	
influent sample	Influent spike	3	5	3	
	Process	3	0	0	
Process sample	Process spike	3	5	3	
Effluent comple	Effluent	3	0	0	
	Effluent spike	3	5	3	

Table 7. Approximate Sample Specifications for Off-line Evaluation of Matrix Effects

B1.1.5b On-Line Testing Phase

The on-line effluent monitoring phase will also be used to evaluate matrix effects for each of the analyzers. The nutrient analyzers will sample the combined plant effluent for a period of approximately four weeks. Three times per week, samples of the plant effluent will be collected for reference analysis. The nutrient analyzer response to the plant effluent at the time of the sample collection will be compared to the reference laboratory result to determine the %R for each sample. The dependence of the %R results on matrix type will indicate any matrix effects. A minimum of 12 samples will be collected and analyzed; reference results from at least 10 samples must meet QC requirements (see Section B5) and be used to determine matrix effects.

Nutrient Analyzers Test/QA Plan Page 32 of 52 Version 1.0 April 22, 2005

B1.1.6 Data Completeness

No additional test procedures will be carried out specifically to address data completeness. This parameter will be assessed based on the overall data return achieved by each analyzer as a percentage of the maximum possible data return.

B1.1.7 Operational Factors

Operational factors such as maintenance needs, calibration frequency, data output, consumables used, ease of use, repair requirements, waste production, etc., will be evaluated based on observations recorded by Battelle, DuPont, and OMI staff. A separate LRB will be maintained at the test site for each analyzer undergoing testing, and will be used to enter daily observations on these factors. Examples of information to be recorded in the record books or on data sheets include the daily status of diagnostic indicators for the analyzer; use or replacement of any consumables; the effort or cost associated with maintenance or repair; vendor effort (e.g., time on site) for repair or maintenance; the duration and causes of any analyzer down time or data acquisition failure; and operator observations about ease of use of the analyzer. These observations will be summarized to aid in describing analyzer performance in the verification report on each analyzer.

B1.1.8 Sampling Summary

Table 8 summarizes the nutrient samples that will be used to evaluate the performance parameters described in this section, including number of conditions and readings per condition.

Nutrient Analyzers Test/QA Plan Page 33 of 52 Version 1.0 April 22, 2005

Parameter	Test Phase	Number of Conditions or Samples	Number of Replicates	Total Number of Analyzer Readings for each Verified Nutrient
Accuracy ^a	Off-line	4	3	12
Accuracy ^{a,b} (TN)	Off-line	5	1	5
Accuracy ^{a,c} (TP)	Off-line	3	1	3
Bias ^a	Off-line	4	3	12
Linearity ^a	Off-line	5	3	15
Limit of Detection	Off-line	1	15	15
Reproducibility ^d	Off-line	1	5	5
Span and Zero Drift	Off-Line	2 2	5 ("baseline") ^d 2 (once each week)	14
	On-Line	2	4 (once each week)	8
Interference Effects ^e	Off-line	8	1	8
	Off-line	6	1	6
Matrix Effects	Off-line	6	3	18
	On-line	12	1	12

Table 8. Nutrient Sample Summary

^a The same samples will be used to evaluate accuracy, bias, and linearity.

^b Additional accuracy samples for verification of TN.

^e Additional accuracy samples for verification of TP.

^d The same samples will be used to evaluate reproducibility and span and zero drift "baseline" responses. DI water challenges will also be included.

^e Interference effects will be evaluated only for nitrate verification.

B1.2 Statistical Analysis

The statistical methods and calculations used for evaluation of the quantitative

performance parameters are described in the following sections.

B1.2.1 Accuracy

Accuracy of the nutrient analyzers with respect to the individual nutrient standards will

be assessed as the percent recovery (%R), using Equation 1:

Nutrient Analyzers Test/QA Plan Page 34 of 52 Version 1.0 April 22, 2005

$$\%R = \left[1 + \left(\frac{Y - X}{X}\right)\right] \times 100\tag{1}$$

where Y is the measured nutrient analyzer value and X is the reference laboratory nutrient concentration. For continuous analyzers, Y will be the average analyzer response for each sample. The average, minimum, and maximum %R values will be reported for each series of multi-level nutrient challenges.

B1.2.2 Bias

Bias of the nutrient analyzers is defined as a systematic error in measurement that results in measured error that is consistently positive or negative compared to the true value. The bias will be calculated as the average percent difference (%D) of the nutrient analyzer compared to the reference laboratory nutrient concentration and will be calculated for the entire series of multi-point challenges for each nutrient, using Equation 2:

$$\%$$
D = $\frac{1}{k} \sum_{j=1}^{k} \left(\frac{Y-X}{X}\right)_{j} \times 100$ (2)

where k is the number of valid comparisons, and Y and X are the same as stated in Section B1.2.1.

B1.2.3 Linearity

Linearity will be assessed by a linear regression analysis using the reference laboratorydetermined nutrient concentration as the independent variable and results from the nutrient analyzers being tested as the dependent variable. Linearity will be expressed in terms of slope, intercept, and coefficient of determination (r^2).

B1.2.4 Limit of Detection

The LOD is the minimum concentration of nutrient that is significantly different from the blank or background signal and is defined as the average blank signal (\overline{Y}_b) plus three standard deviations of the blank (s_b). The LOD will be calculated from at least 15 blank (DI water) measurements using Equation 3:

$$LOD = \overline{Y}_b + 3s_b \tag{3}$$

For each nutrient analyzer, the LOD will be calculated separately for each nutrient being verified.

B1.2.5 Reproducibility

The reproducibility of the nutrient analyzers will be evaluated from the five repeated measurements a mid-level nutrient standard as described in Section B1.1.2. Reproducibility will be defined as the %RSD of the five measurements, using Equation 4:

$$\% \text{RSD}_i = \frac{\text{s}}{\overline{\text{Y}}_i} \times 100 \tag{4}$$

where \overline{Y} is the average analyzer response at the mid-level nutrient concentration *i*, and s the standard deviation of the analyzer responses. One reproducibility value will be determined for each nutrient being verified.

B1.2.6 Span and Zero Drift

The "baseline" response of the nutrient analyzers to DI water and the mid-level nutrient standard will be established early in the verification test, as outlined in Section B1.1.3. The mean (\overline{Y}) and standard deviation (s) of the analyzer response to DI water and the mid-level nutrient standards will each be calculated from the five replicate measurements conducted for the reproducibility checks. From these values, a control chart will be constructed and the $\overline{Y} \pm 2s$ "warning limit" and the $\overline{Y} \pm 3s$ "action limit" will be calculated. Span drift will be defined as
having occurred if three consecutive span checks all fall outside of the warning limit. Zero drift will also be defined as having occurred if three consecutive zero checks all fall outside of the warning limit. However, if the mean and/or standard deviation from the baseline DI water challenges are equal to zero, the warning limits may not be meaningful. In this case, the absolute differences to the DI water baseline mean will be reported for each zero check.

B1.2.7 Interference Effects

The interference effects of the nitrate analyzers will be calculated in terms of the ratio of the response of the analyzer to the non-nitrate nutrient relative to the reference laboratory concentration of the non-nitrate nitrogen and/or phosphorus species. For example, if 5 mg N/L of ammonia results in a 0.5 mg N/L change in the response of the analyzer, the interference effect will be reported as 10% (i.e., 0.5 mg N/L \div 5 mg N/L \times 100). Interference effects will be reported separately for each non-nitrate nutrient individually, for a mixed nitrogen sample, and for a mixed phosphorus sample.

B1.2.8 Matrix Effects

Matrix effects on the nutrient analyzers with respect to each matrix sample will be assessed based on the dependence of the %R on matrix type. The identification of matrix effects will be conducted by comparing %R values determined on other matrix samples to those determined on the final outfall stream samples during the on-line testing. Specifically, the mean and standard deviation of the %R values for the final outfall samples collected three times a week will be determined, and the mean (± 2 s) range of those %R values will be calculated. As noted in Section B1.1.5b, at least 10 such samples meeting QA requirements will be obtained. %R values for other matrices falling outside the mean (± 2 s) range of the final outfall samples will be taken to indicate a matrix effect.

Nutrient Analyzers Test/QA Plan Page 37 of 52 Version 1.0 April 22, 2005

B1.2.9 Data Completeness

Data completeness will be calculated as the percentage of the total possible data return over the entire field period that is achieved by each analyzer. This calculation will use the total hours (h) of data recorded from each analyzer, divided by the total h of data in the entire field period. The field period is defined to begin at 8:00 a.m. on the first day of testing and to end at the completion of the last testing activity on the final day of the field period. No distinction will be made in this calculation between data recorded during a specific test activity (e.g., data recorded for off-line testing) and that recorded during the on-line effluent monitoring phase. The causes of any substantial loss of data return will be established from operator observations or vendor records, and noted in the discussion of data completeness results.

B1.3 Reporting

The statistical comparisons described above will be conducted separately for each of the analyzers being tested, and information on the operational parameters will be compiled and reported. The data for each analyzer will be kept separate from data for all other analyzers, and no intercomparison of the analyzer data will be performed at any time. A separate verification report will be prepared for each analyzer tested that presents the test procedures and test data, as well as the results of the statistical evaluation of those data.

Operational aspects of the analyzers will be recorded by testing staff at the time of observation during the field test, and summarized in the verification report. For example, descriptions of the data-acquisition procedures, use of vendor-supplied proprietary software, consumables used, repairs and maintenance needed, and the nature of any problems will be presented in the report. Each verification report will briefly describe the ETV program, the AMS Center, and the procedures used in verification testing. The results of the verification test will be stated quantitatively, without comparison to any other analyzer tested, or comment on the acceptability of the analyzer's performance. Each draft verification report will first be subjected to review by the respective analyzer vendor, then revised and subjected to a review by EPA and other peer reviewers. The peer review comments will be addressed in further revisions of the

report, and the peer review comments and responses will be tabulated to document the peer review process. The reporting and review process will be conducted according to the requirements of the AMS Center QMP.¹

B2 SAMPLING REQUIREMENTS

As described above, testing of nutrient analyzers will consist of two off-line testing phases and an extended on-line effluent monitoring phase. Battelle, DuPont, and/or OMI staff will collect samples throughout the verification test that will be submitted to a reference laboratory for analysis. The samples will be collected following guidelines set in each standard reference method listed in Section B4. The methods describe the appropriate sampling containers, preservation techniques, and maximum holding times. During the off-line testing phase, aliquots of the nutrient and other samples prepared for testing the analyzers will be transferred to appropriate sample containers, preserved if necessary, and submitted to the reference laboratory for analysis. During the on-line effluent monitoring phase, grab samples will be collected from the manifold spigot three time per week, in coordination with the sample collection times of the non-continuous nutrient analyzers; the samples will be collected at a location as close to the analyzer inlets as possible. Sufficient sample will be collected to split as needed for nitrate, TN, and TP analysis. Table 9 summarizes the samples to be collected during each phase of the verification test.

Nutrient Analyzers Test/QA Plan Page 39 of 52 Version 1.0 April 22, 2005

Test Phase	Parameter	Sample Description	Number of Samples
	Reproducibility	DI water blank	2
	Span and Zero Drift	Mixed N and P	2
		Nitrogen Mixture	1
		Ammonia	1
	TN and TP Accuracy Nitrate Interference Effects	Nitrite	1
		Nitrate	1
Off-line		Organic Nitrogen	1
Pliase I		Phosphorus Mixture	1
		Inorganic Phosphorus	1
		Organic Phosphorus	1
	Accuracy and Linearity	Nitrate	10
		Phosphate	10
	Motin Effects	pH adjusted series	7
	Matrix Effects	Chlorophyll a series	6
Off-line Phase I On-line Off-line Phase II Total	Matrix Effects	Combined effluent from manifold	12
		DI water blank	4
	Span and Zero Drift	Mixed N and P	4
Off-line Phase II	Matrix Effects	Plant influent	4
		Process sample	4
		Treated effluent	4
	Snon and Zara Drift	DI water blank	1
		Mixed N and P	1
Total			79

Table 9. Reference Method Sample Summary

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample custody will be documented throughout collection, transport, shipping (if necessary), and analysis of the samples, using standard forms used by the reference laboratory for this purpose or forms provided by Battelle. Each chain-of-custody (COC) form summarizes the samples collected and analyses requested. The COC form will track sample release from the sampling location to the reference laboratory. COC forms will be used regardless of whether the

samples are being transferred within the DuPont facility or to an external location. Each COC form will be signed by the person relinquishing samples once that person has verified that the COC form is accurate. The original sample COC forms will accompany the samples; the shipper will keep a copy. Upon receipt at the laboratory, COC forms will be signed by the person receiving the samples once that person has verified that all samples identified on the COC forms are present. Any discrepancies will be noted on the form and the sample receiver will immediately contact the DuPont sampling leader or the Verification Test Coordinator to report missing, broken, or compromised samples. Copies of all COC forms will be delivered to the Verification Test Coordinator, and maintained with the test records.

B4 LABORATORY REFERENCE METHODS

Table 10 provides the standard laboratory methods that will be used for the sample collection and reference analyses during this verification test. Also included in the table is each method's method of preservation and maximum holding time. The collection of the samples will be the responsibility of Battelle, DuPont, and OMI staff. DuPont is responsible for coordinating analysis of the samples by the reference laboratory. Results of the reference analyses will be provided to DuPont within 14 days of sample receipt. DuPont will be required to provide to Battelle all sample results within one week of receipt of the sample results from the reference laboratory.

Nutrient Analyzers Test/QA Plan Page 41 of 52 Version 1.0 April 22, 2005

Parameter	Method (Standard Methods 18 th Edition) ³	Method LOD mg/L	Preservation and Storage Conditions	Holding Time
Ammonia	4500-NH3 Parts B & C (Distillation & Nesslerization Method)	0.05	0.8 mL conc. H ₂ SO ₄ /L (1.5 <ph<2); 4°c<="" td=""><td></td></ph<2);>	
Nitrate	4500-(NO3F &NO2B)	0.02	4°C	Up to 24 h
	minus NO2B		$2mL \text{ conc } H_2SO_4/L; 4^{\circ}C$	48 h
Nitrite	4500-NO2B	0.02	Frozen at -20°C or stored at 4°C	48 h
Total Nitrogen	sum of total Kjeldahl nitrogen (TKN), nitrate, and nitrite	0.5		48 h
Organic Nitrogen	TKN minus ammonia	0.5	Add 40 mg HgCl ₂ /L; Frozen at or below -10°C	
Total Phosphorus	4500-Р В&Е	0.05	Add 40 mg HgCl ₂ /L; Frozen at or below -10°C	28 days
Dissolved Inorganic Phosphorus	4500-P B&F TP minus organic phosphorus	0.05	Add 40 mg HgCl ₂ /L; Frozen at or below -10°C	28 days
Organic Phosphorus	4500-P B&E TP minus (reactive and acid hydrolyzable P)	0.05	Add 40 mg HgCl ₂ /L; Frozen at or below -10°C	28 days

Table 10. Reference Methods

B5 QUALITY CONTROL

As described in Section B2, the reference laboratory will follow their standard QA/QC protocols for analysis of QCSs with each set of samples analyzed. Steps will be taken to maintain the quality of the data collected during this verification test. QCSs producing results not meeting the laboratory's standard requirements will be reanalyzed. If the results are still

outside the required tolerance, the reference instrument will be recalibrated and the samples reanalyzed. If the outlying results persist, the affected data will be flagged and a repeat of the affected parts of the verification test may be considered. Sample results not meeting these requirements will be flagged and excluded from comparison to the nutrient analyzer results. A minimum of 10 reference measurements from the on-line testing phase must meet these QC requirements for use in the matrix effects comparisons, as stated in section B1.1.4b.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The equipment used for the reference analyses will be tested, inspected, and maintained as per the standard operating procedures of the reference laboratory or the standard methods being used to make each measurement. The reference analyses require use of the following apparatuses and instrumentation: a distillation apparatus, pH meter, spectrophotometer, and continuous-flow analytical instrument. When Battelle, DuPont, or OMI staff operate and maintain the nutrient analyzers undergoing testing, those activities will be done as directed by the vendor. Otherwise, operation and maintenance of the analyzers will be the responsibility of the analyzer vendors.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

The instrumentation used by the commercial reference laboratory for the reference analyses (e.g., spectrophotometers and flow injection analyzers) will be calibrated per the standard reference methods being used to make each measurement or the standard operating procedures of the reference laboratory. Other instrumentation used in this verification test, such as a multi-parameter water probe, will have been calibrated within the 12 months prior to this verification test and, if possible, the calibration will be verified immediately prior to use in this verification test.

The nutrient analyzers undergoing testing will be calibrated initially by the respective analyzer vendors at the time of installation at the test site using nutrient standards independent of those used for testing activities. Automated or manual calibration checks will be performed only upon direction by the analyzer vendor. Additional analyzer calibrations may be performed during the verification test if they are part of the standard operating procedures for that analyzer. In the event that recalibration is necessary due to maintenance activities performed on the analyzer or other malfunction (e.g., power loss at the test site), recalibration will be carried out by the analyzer vendor, or by Battelle, DuPont, or OMI staff under the direction of the vendor. All calibrations performed will be documented by Battelle, DuPont, or OMI staff in the LRB dedicated to the respective analyzer.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All materials, supplies, and consumables will be ordered by the Verification Test Coordinator or designee. Where possible, Battelle will rely on sources of materials and consumables that have been used previously as part of ETV verification testing without problems. Battelle will also rely on previous experience or recommendations from EPA advisors, stakeholders, DuPont staff, or analyzer vendors. When possible, National Institute of Standards and Technology (NIST) traceable standards will be used.

B9 NON-DIRECT MEASUREMENTS

Data published previously in the scientific literature will not be used during this verification test.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by Battelle, vendor, DuPont, and OMI staff during this verification test. Table 11 summarizes the types of data to be recorded. All maintenance activities, repairs, calibrations, and operator observations relevant to the operation of the nutrient analyzers will be documented by Battelle, DuPont, or OMI staff in LRBs or on data sheets. A separate record book will be provided for each participating analyzer. Results from the reference methods, including raw data, analyses, and final results, will be compiled by DuPont staff, preferably in electronic format, and submitted to Battelle at the conclusion of reference nutrient analyses.

Nutrient Analyzers Test/QA Plan Page 45 of 52 Version 1.0 April 22, 2005

Data to Be Recorded	Where Recorded	How Often Recorded	By Whom	Disposition of Data
Dates, times, and details of test events, analyzer maintenance, down time, etc.	ETV LRBs or data recording forms	Start/end of test procedure, and at each change of a test parameter or change of analyzer status	Battelle if on-site; DuPont or OMI at other times	Used to organize and check test results; manually incorporated in data spreadsheets as necessary
Analyzer calibration information	ETV LRBs or electronically	At analyzer calibration or recalibration	Electronic data by vendor; Battelle if on-site; DuPont or OMI at other times	Incorporated in verification report as necessary
Analyzer nutrient readings	Either recorded electronically by the analyzer and downloaded to an independent computer at least weekly or hard copy data printed by the analyzer and taped into the ETV LRB.	Recorded continuously for electronic data and printed after each measurement for hard copy print- outs.	Analyzer vendor, for transfer to Battelle	Converted to or manually entered into spreadsheet for statistical analysis and comparisons
Sample collection and reference method analysis procedures, calibrations, QA, etc.	LRBs, or data recording forms	Throughout sampling and analysis processes	Battelle if on-site, DuPont or OMI at other times, and Reference laboratory	Retained as documentation of reference method performance
Reference method nutrient analysis results	Electronically from analytical method	Every sample analysis	Reference laboratory	Transferred to spreadsheets for calculation of ambient nutrient results, and statistical analysis and comparisons

Table 11. Summary of Data Recording Process

Records generated by any Battelle, DuPont, or OMI staff or the reference laboratory during the verification test will be reviewed by a Battelle staff member within two weeks of

generation (or receipt, if generated by DuPont or OMI staff or the reference laboratory), before the records are used to calculate, evaluate, or report verification results. If a Battelle staff member generated the record, this review will be performed by a Battelle technical staff member involved in the verification test, but not the staff member who originally generated the record. The review will be documented by the person performing the review by adding his/her initials and date to the hard copy of the record being reviewed. In addition, any calculations performed by Battelle or DuPont staff will be spot-checked by Battelle technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this test/QA plan. The data obtained from this verification test will be compiled and reported independently for each nutrient analyzer. Results for analyzers from different vendors will not be compared with each other.

Among the QA activities conducted by Battelle QA staff will be an audit of data quality. This audit will consist of a review by the Battelle Quality Manager of at least 10% of the test data. During the course of any such audit, the Battelle Quality Manager will inform the technical staff of any findings and any need for immediate corrective action. If serious data quality problems exist, the Battelle Quality Manager will request that Battelle's AMS Center Manager issue a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken.

Nutrient Analyzers Test/QA Plan Page 47 of 52 Version 1.0 April 22, 2005

SECTION C

ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in this verification test to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this test/QA plan is to establish mechanisms necessary to ensure this. Internal quality control measures described in this test/QA plan, which is peer reviewed by a panel of outside experts, implemented by the technical staff and monitored by the Verification Test Coordinator, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the Verification Test Coordinator. Technical staff have the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the Verification Test Coordinator, who will work to resolve any issues. Action will be taken to control the problem, identify a solution to the problem, and minimize losses and correct data, where possible. Independent of any EPA QA activities, Battelle will be responsible for ensuring that the following audits are conducted as part of this verification test.

C1.1 Performance Evaluation Audits

A Performance Evaluation (PE) audit will be conducted to assess the quality of the nutrient reference method measurements made in this verification test. The PE audit of the nutrient reference methods will be performed by supplying each reference method a blind, independent, NIST-traceable nutrient standard provided by Battelle. The PE audit samples will be analyzed in the same manner as for all other samples and the analytical results for the PE audit samples will be compared to the nominal concentration. The target criterion for this PE audit is agreement of the analytical result within 25% of the nominal nutrient concentration (by

%D). If the PE audit results do not meet the tolerances shown, they will be repeated. If the outlying results persist, a change in reference instrument and a repeat of the PE audit may be considered. This audit will be performed once prior to the start of the test and once during the verification test, and will be the responsibility of the Verification Test Coordinator or her designee.

C1.2 Technical Systems Audits

The Battelle Ouality Manager or his designee will perform a TSA at least once during this verification test. The purpose of this audit is to ensure that the verification test is being performed in accordance with the AMS Center QMP¹, this test/QA plan, published reference methods, and any Standard Operating Procedures (SOPs) used by DuPont or the reference laboratory. In the TSA, the Battelle Quality Manager, or a designee, may review the reference methods used, compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures. In the TSA, the Battelle Quality Manager will tour the test site, observe the nutrient reference method sampling and sample recovery, inspect documentation of nutrient sample chain of custody; and review analyzer-specific record books. He will also check nutrient standard certifications and analyzer data acquisition procedures, and may confer with the analyzer vendors and DuPont and OMI personnel. He may also visit the reference laboratory where the nutrient reference method analysis is conducted, to review procedures and adherence to this plan and applicable SOP's. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The EPA AMS Center Ouality Manager will receive a copy of Battelle's TSA report. At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

Nutrient Analyzers Test/QA Plan Page 49 of 52 Version 1.0 April 22, 2005

C1.3 Data Quality Audits

The Battelle Quality Manager or his designee will audit at least 10% of the verification data acquired in the verification test. The Battelle Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked.

C1.4 QA/QC Reporting

Each assessment and audit will be documented in accordance with Section 3.3.4 of the AMS Center QMP.¹ The results of the technical systems audit will be submitted to EPA. Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Response to adverse findings or potential problems
- Recommendations for resolving problems
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others.

C2 REPORTS TO MANAGEMENT

The Battelle Quality Manager, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the Battelle Quality Manager is authorized to request that Battelle's AMS Center Manager issue a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken. The test/QA plan and final report are reviewed by EPA AMS Center QA staff and EPA AMS Center program management staff. Upon final review and approval, both documents will then be posted on the ETV website (www.epa.gov/etv).

Nutrient Analyzers Test/QA Plan Page 51 of 52 Version 1.0 April 22, 2005

SECTION D

DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The key data review requirements for the verification test are the collection of QC samples according to the reference laboratory's standard procedures, a comparison of field data sheet comments against final data to flag any suspect data, and a review of final data to resolve any questions about apparent outliers. The QA audits described within Section C of this document, including the audit of data quality, are designed to assure the quality of the data.

D2 VALIDATION AND VERIFICATION METHODS

Section C of this test/QA plan provides a description of the validation safeguards employed for this verification test. Data validation and verification efforts include the collection of QC samples as required in this document, and the performance of TSA and PE audits as described in Section C.

D3 RECONCILIATION WITH USER REQUIREMENTS

This test/QA plan and the resulting ETV verification report(s) will be subjected to review by the nutrient analyzer vendors, DuPont, EPA, and external expert peer reviewers. These reviews will assure that this test/QA plan and the resulting report(s) meet the needs of potential users and permitters of nutrient analyzers. The final report(s) will be submitted to EPA in Word Perfect and Adobe pdf format and subsequently posted on the ETV website.

SECTION E

REFERENCES

- Quality Management Plan for the ETV Advanced Monitoring Systems Center, Version 5.0, U.S. EPA Environmental Technology Verification Program, Battelle, Columbus, Ohio, March 2004.
- Environmental Technology Verification Program Quality Management Plan, EPA/600/R-03/021, U.S. Environmental Protection Agency, Cincinnati, Ohio, December 2002.
- Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association, Washington D.C., 1992.

Nutrient Analyzers—Appendix A Test/QA Plan Page A-1 of 29 Version DRAFT March 23, 2005

APPENDIX A

SPRUANCE PLANT SITE CONDITIONS

Nutrient Analyzers—Appendix A Test/QA Plan Page A-2 of 29 Version DRAFT March 23, 2005

Spruance Plant Site Conditions

Table Of Contents

1.0 SAFETY	3
1.1 Safety Requirements	3
1.2 Reporting Incidents/ Injuries	3
1.3 Contractor Qualification and Selection	4
1.4 Contractor Supervision and Subcontractor	4
1.5 Owner Representative	4
1.6 Safety Program	4
1.7 Safety Orientation/ Reorientation	5
1.8 Safety Submittals	5
1.9 Safety Task Assignment/ Safety Meetings/ Safety Training	6
1.10 Safety Audits / Violations.	6
1.11 Personal Protective Equipment	6
1.12 Respiratory Equipment	7
1.13 Ergonomics	8
1.14 Permits	8
1.15 Barricades	8
1.16 Construction Equipment	9
1 17 Rigging	. 12
1.18 Access	. 12
1.19 Spray Painting	. 12
1.20 Fall Prevention	. 12
1.21 Ladders and Scaffolding	. 13
1.22 Ground Fault Protection and Power Cords	. 14
1.23 Gas Cylinders	. 14
1.24 Tool Inspection	. 15
1.25 Inspection Color Code System	. 15
1.26 Lead Containing Materials	. 15
1.27 Asbestos Containing Materials	. 16
1.28 Non-Asbestos Respirable Fibers	. 17
1.29 Projecting Materials	. 17
1.30 Pipe, Ductwork, Cable Trays	. 17
1.31 Tag, Lock, Clear, Try Procedure	. 17
1.32 Confined Space and Vessel Entry	. 17
1.33 Electrical Qualification	. 18
1.34 Dismantling and Rearranging	. 18
1.35 Hazard Communication	. 18
1.36 Electrical Welding and Portable Generators	. 19
1.37 Excavations and Wall Penetrations	. 19
1.38 High Pressure water Cleaning	. 19
1.39 Housekeeping	. 19
1.40 Pipe Jackstands	. 20
1.41 Pneumatic Testing	. 20
1.42 Powder Actuated Devices	. 20
1.43 Railroad Operations	. 20
1.44 Temporary Lighting	. 21
1.45 Throwing Material	. 21

Rev. Date 06/02/04

1.46 Vehicle Safety	. 21
1.47 Waste Material Control	. 21
2.0 FIRE PROTECTION	. 22
2.1 Procedures	. 22
2.2 Smoking	. 22
2.3 Flammable Materials	. 22
2.4 Fire Extinguishers.	. 22
2.5 Oily Rag Storage	. 22
2.6 Protective Structures	. 23
2.7 Burning and Welding	. 23
3.0 CONSTRUCTION FACILITIES	. 23
3.1 Compressed Air	. 23
3.2 Drinking Water and Sanitary Facilities	. 23
3.3 Electric Power	. 23
3.4 Identification	. 23
3.5 Plant Facilities	. 24
3.6 Site Access	. 24
3.7 Telephones and other Communication Equipment.	. 24
3.8 Temporary Construction (TC) Facilities and Trailers	. 24
3.9 Water	. 24
4.0 WORKING CONDITIONS	. 24
4.1 Cellular Telephones	. 25
4.2 Co-occupancy	. 25
4.3 Coordination with Others	. 25
4.4 Diversity	. 25
4.5 English Requirement	. 25
4.6 Material Shipment and Storage	. 26
4.7 Prohibited Items	. 26
4.8 Testing for Substance Abuse	. 26
4.9 Tool and Equipment Control	. 27
4.10 Visitor Control	27
4.11 Insignias	27
4.12 Parking/Gate Entry Site Procedures	. 27

Nutrient Analyzers—Appendix A Test/QA Plan Page A-4 of 29 Version DRAFT March 23, 2005

1.0 SAFETY

The safety requirements listed herein shall not relive Contractor from complying with State and Federal Occupational Safety and Health Act (OSHA) regulations or any other contractual agreement, and are only noted to highlight potential problem areas.

1.1 Safety Requirements

Contractor's work shall be performed in accordance with the instructions set forth in DuPont's "Green Safety Handbook" (Green Book, G770). Contractor shall also comply with the safety program in effect at the plant site. This program includes use of the above referenced book which will be furnished by DuPont.

Contractor shall employ a full time safety professional approved by DuPont when on-site workforce is equal to or greater than 25 persons, including subcontractors. Contractor shall provide DuPont with a resume for review and approval.

1.2 Reporting Incidents/ Injuries

Contractor shall immediately notify DuPont of any injury, first aid case, or potentially serious incident or hazard to personnel on the site. Each injury or incident will be jointly investigated by Contractor's supervisor, the affected employees, and DuPont's representatives. Contractor shall submit a detailed report to Dupont within 24 hours of the injury or incident.

Before beginning work, all contractors shall provide DuPont with the name and address of their Emergency Care Provider in case of injury to contractor personnel. A preferred local Occupational Health Medical provider is:

Concentra Medical Center 1603 Commerce Road Richmond, VA 23224 (804) 230-6600

DuPont will provide immediate medical care and attention in the event of a serious injury to contractor personnel. Care and attention will be limited to stabilizing the injured person until follow up care can be arranged. The contractor shall ensure that any employee who receives an electrical shock is provided a professional medical evaluation immediately following any such occurrence.

Contractor shall keep at least one person qualified in administering first aid treatment on site during all working hours. At a minimum, the person shall hold a Red Cross First Aid and CPR card (or equivalent) and be included in the contractor's Bloodborne Pathogens Exposure Control Program. In addition to qualified personnel, the contractor shall maintain first aid supplies and equipment adequate for the number of employees and approved, in writing, by the contractor's physician.

All contractor personnel shall be aware of the hazards associated with blood and body fluid contact.

Rev. Date 06/02/04

Nutrient Analyzers—Appendix A Test/QA Plan Page A-5 of 29 Version DRAFT March 23, 2005

1.3 Contractor Qualification and Selection

DuPont is committed to working with safe contractors and has established minimum acceptable criteria for contractors and tier subcontractors. Exceptions to the following criteria are granted only by written approval from the site Contracts group.

Successful bidders shall be limited to second-tier subcontractors. All subcontractors shall meet the same requirements as prime contractors.

Contractors and their subcontractors shall meet the following minimum criteria:

- Have a Workers Compensation Insurance Experience Modification Rate (EMR) of .99 or less for the last three (3) years. Proof shall include copies of the EMR rates from the workers compensation carrier or underwriter.
- Have a Total Recordable Injury Frequency Rate (TRIFR) of 7.0 or less for the last three (3) years. Proof shall include the previous 3-year's OSHA 200/300 and the company's formal documentation of employee exposure hours (total hours worked) for those years.

1.4 Contractor Supervision and Subcontractor

The contractor's (prime and all sub-contractor's) on-site project supervisor shall attend a supervisor safety communication meeting with DuPont's contract administrator (and others) prior to the initiation of project activities. The Contractor's site supervisor (prime contractor) shall be on-site at all times when contractor or their subcontractor employees are performing work activities.

Fluor Entrerprises presently performs work for DuPont on the Spruance plant and may NOT be used as a subcontractor on this contract.

The contractor supervisor (prime) will be held accountable for the safety and health of all employees working on the project (prime and subcontractor) employee.

1.5 Owner Representative

DuPont shall designate Area Contract Coordinator (ACC's) and Contract Administrators (CA's) through whom all work questions or interpretations must be cleared.

1.6 Safety Program

Before starting work, contractor shall submit, for DuPont review and acceptance, a written safety program that all contractors' employees must follow while on the job site. Minimum acceptable program shall meet OSHA requirements and include the Hazard Assessment per OSHA 1910.

Contractor's program shall meet the requirements of 29 CFR 1910.119 (Process Safety Management). Pursuant to 1910.119, contractor shall ensure that employees:

> Have the necessary job skill training and are qualified to safely perform the contracted work.

Rev. Date 06/02/04

- Are instructed in the known potential fire, explosion, or toxic release hazards related to their jobs and the process.
- Receive and understand training regarding site safety rules, the Emergency Response Plan, and safety work practices of the facility.
- > Follow all applicable work practices and safety rules of the facility.
- > Are fit for duty and are not compromised by external influences.
- Advise DuPont of any unique hazards presented by the contractor's work, or any hazards found by the contractor's work.
- Audited to ensure that they are working safely and that they follow all applicable work procedures, practices and safety rules of the facility.

Contractor shall submit documentation of above at DuPont's request.

High Hazard areas in the plant have been identified as the following:

- Nomex: RP, Chemical Process, Spinning (2nd floor and roof), Hot Flush Room, and Fibradations
- > Tyvek: Line 4, Line 7, and the Pilot Plant
- > Power: Sulfuric Acid, Caustic Lime Tower System, and Caustic Filter Plant
- Nomex Solvent Recovery
- > Kevlar Solvent Recovery (Includes TCL Dike and Polymer)
- Films: Coating Solution Preparation Area (Mix House), Coating Towers, R1 Coater, Solvent Recovery

Work in these areas require special procedures and will be covered in area specific orientation.

1.7 Safety Orientation/ Reorientation

Prior to beginning work on site, all contract employees must attend the site and area safety orientation. Performance testing to confirm employee knowledge is part of this process. Safety orientation is given in American English. Contractor shall provide a translator if needed. The supervisor shall escort the new employee (s) to the work area.

The cost of attending orientation shall be included in the Contractor's proposal.

Site safety orientations are given Monday through Thursday promptly at 8:00am in the Site Services building and are valid for one year.

1.8 Safety Submittals

At DuPont's request, contractor may be required to supply the following:

- > Written Safety program and training documentation
- OSHA 200 or 300 logs
- > Written fall protection plan/ scaffold procedure
- ➢ Written rigging plan
- Documentation on rigging/ lifting work equipment
- Documentation for equipment operators
- > Documentation of respiratory protection certification
- Listing of "Competent Persons" as per OSHA requirements

Rev. Date 06/02/04

Nutrient Analyzers—Appendix A Test/QA Plan Page A-7 of 29 Version DRAFT March 23, 2005

1.9 Safety Task Assignment/ Safety Meetings/ Safety Training

Contractor shall conduct a project-specific safety orientation, covering the scope of work, these Site Conditions and other safety procedures covered at the pre-construction meeting, for each contractor employee before he/she begins work on the site. Meeting attendance shall be documented and furnished to DuPont.

Contractor shall inform each employee of his/her responsibility to work safely and prevent occupational injury. Contractor shall hold a safety meeting for its personnel at the beginning of the first shift of each week. Meeting attendance will be documented and furnished to DuPont.

Contractor will train employees in safe work methods of their assignments and in some cases certification documents will be required before employees start work and will continue throughout all phases of the work. The contractor shall be responsible for maintaining records of all training in accordance with OSHA and ANSI requirements. Personnel training records shall be submitted upon request. DuPont will reserve the right to audit the training program and procedures that the contractor has established. Employees sent to work on site without the required training will be denied access until the records reflect the required training.

1.10 Safety Audits / Violations

Contractor's job supervisor shall conduct daily safety audits of contractor's work area. Contractor shall send a representative to participate in the weekly safety audit conducted by Contract Services. The audit team meets each Tuesday at 1:30 pm in the Site Services Building. Contractor shall also participate in the Weekly Contractor Safety Meeting held each Thursday at 2:00pm in the Site Services Building.

Employees who repeatedly violate requirements or commit acts that endanger themselves or others will be denied access to the job site.

In the event of OSHA violations or unsafe practices involving imminent danger to DuPont or contractor personnel, immediate action will be taken to stop work and correct the hazardous situation. If violations continue or corrective actions are not taken after a reasonable period of time, DuPont reserves the right to correct the hazardous situations and back-charge the contractor for the cost. DuPont may also exercise the option of terminating the contract in accordance with the General Conditions.

1.11 Personal Protective Equipment

Contractor's personnel and subcontractors performing work shall wear hardhats, steel-toed shoes, and safety glasses with side shields. Steel-toed shoes shall conform to ANSI Z41.1.

Hardhats shall conform to ANSI Z89.11 and are not permitted to be worn backwards. Bump caps are not permitted. Contractor employees shall have on their hardhats a company insignia showing the name of the contractor they are working for. The insignia shall be different from others in the plant. Contract employees shall have their names clearly displayed on the hardhat for identification purposes.

Safety glasses, including all components (frames, lenses, and side shields) shall conform to ANSI Z87.1. Rigid side shields will be required on safety glasses. They can be either a permanent part

of the glasses, snap on, or slip-on type shields (flimsy, heavy film slip-on side shields are not permitted). The lenses must be etched and the frames marked with Z87.1.

Tinted lenses are not permitted indoors.

Impact and chemical resistant mono-goggles, or coverall visitor spectacles conforming to ANSI Standard Z87.1, worn over prescription glasses will be permitted only on an interim bases until personnel are furnished safety glasses as specified above. Coverall spectacles or mono-goggles shall not be used as an ongoing substitute for safety glasses.

Contact lenses are NOT permitted to be worn while working on site in process areas. Monogoggles shall be readily available, when walking in/near process areas.

Leather gloves are required to be worn at all times while performing work unless gloves will not allow for the level of manual dexterity required to achieve the given task, gloves increase the hazard (i.e. rotating machinery or equipment), or another type of glove is more appropriate for the task. DuPont Contract Services Safety Personnel must be involved in the review process.

(Note: Latex gloves are not permitted to be worn in any Tyvek area for quality reasons.)

Cut resistant gloves (i.e. Kevlar or similar) are required during cutting operations involving a knife, box cutter, or like tool

Hearing protection shall be required where noise levels exceed 85 dBA or where posted. EAR, Com-fit and Pura-fit earplugs have been approved for use on site. Contractors performing work in PSM areas will be required to wear full-body Nomex Flame Retardant clothing or equivalent.

Other protective clothing will vary depending on the type of work being performed and the location within the plant site. However, all protective clothing and equipment will be worn as applicable to comply with plant area and regulatory requirements. All such clothing and equipment shall be provided by contractor.

All Personal Protective Equipment shall be provided by the Contractor at no cost to DuPont.

Non-compliance with the site requirements regarding the use of personal protective equipment shall warrant removal of personnel from the premises.

1.12 Respiratory Equipment

In the event respiratory equipment is required, employee must be clean shaven as defined by OSHA.

Before any employee starts work requiring the use of respiratory equipment, Contractor shall furnish DuPont proof of Compliance with OSHA requirements, including the following documentation:

- > Physician's approval for the employee to use respiratory protective equipment
- > Training records indicating each type of respirator the employee is trained to use
- > Fit test records for each type of respirator the employee is trained to use
- Proper certification and proof must be provided with each breathing air supply order to insure Grade D air is supplied.

1.13 Ergonomics

The goal of ergonomics is to fit individual jobs to the capabilities of the person, rather than make the person fit the job. The contractor shall comply with general philosophy that ergonomically related injuries and illnesses are preventable by employing sound ergonomic control measures.

At a minimum, the contractor shall use proper mechanical tools (such as hoists, forklifts and other pieces of equipment) for material handling tasks. Where manual material handling is performed, the contractor shall enforce a fifty-(50) pound maximum manual lift limit under optimal conditions (load is close to body) and thirty-(30) pound limit under less-than-optimal conditions (awkward lifting position.)

1.14 Permits

Contractor is responsible for obtaining all permits required by local, county and state laws that have not been obtained by DuPont for the performance of their work at this site.

- 1.14.1 Electrical permits will be required when working in or on energized electrical equipment, i.e. panels, MCC's, sub stations, etc. (Spruance safety manual item 122-3) DuPont will provide contractor with a copy of permit before contractor commences electrical work in area covered by permit.
- 1.14.2 Work permits will be required on a job duration basis. Contractor is responsible for obtaining such permit from the Contract Administrator before the start of work.
- 1.14.3 Close proximity permits will be required daily for use of construction equipment over, or within fifteen (15) feet of, any overhead electric power line, process pipe, or service pipe. Contractor shall fill out necessary permit forms covering the following days work and submit them daily for DuPont's approval prior to beginning tasks.
- 1.14.4 Vessel or confined space entry permits will be required for any work inside a vessel or confined space (i.e. area which must be entered through a restricted opening, such as tanks, vessels, ductwork, vessel skirts, manholes, etc.)

DuPont will provide contractor with the necessary permits before work in the vessel or confined space will be permitted to start. Contractor shall furnish all protective and emergency equipment and shall comply with all precautions stated in permit.

1.14.5 An excavation permit will be required for each area to be excavated. Excavation is penetration greater than (3 inches).

DuPont will provide contractor with a copy of permit before contractor commences excavation work in area covered by the permit. The contractor shall comply with the requirements in DuPont FC&S Safety Manual procedure B-5.1, Excavations.

1.14.6 A radiographic permit will be required prior to beginning any work associated with a radiation source on site. DuPont will provide contractor with a copy of permit before contractor commences work. All radioactive materials, radiation devices and installations must be reviewed and approved by the radiation safety officer before they are bought on the site.

1.15 Barricades

Rev. Date 06/02/04

Barricades are erected to protect areas where unusual activities or conditions in the area exist. Only authorized persons are allowed to enter these barricaded areas and only after learning of any additional precautions or protective equipment/ clothing that may be required.

There are many informational, caution and danger signs posted throughout the plant site. All personnel are to comply with the directions of posted signs.

Barricade tape and stands will be furnished by DuPont. Contractor shall furnish all other material. Each individual contractor will be responsible for maintaining and ensuring integrity of their own barricaded work areas.

Barricades are required:

≻

- > Around all work areas
- > Around crane swing areas; post overhead work signs.
- To define outer limits of high noise areas. Post appropriate signs.
- To define areas of overhead work. Post appropriate signs.
- Around excavations, post appropriate signs and follow OSHA guidelines.
- For road closures, barricades may be saw horse type. Post appropriate signs. Provide flashing yellow lights if barricades are left overnight. Notify DuPont 48 hours in advance before closing off any road.
- Red barricades are to be used for asbestos, crane usage, suspended material, certain types of electrical work, etc. when entry is strictly prohibited. This list is not all inclusive.

Warning (tape and stand) barricades - Tape shall be supported only by stands or posts acceptable to DuPont. Do not tie to pipes, valves, material drums, vehicles, etc. Stands or posts shall be spaced no more than 25 feet apart. Stands or posts subjected to wind shall be weighted or otherwise secured so they remain erect.

Protective (rigid) barricades – shall consist of a guardrail and midrail meeting OSHA definition of "standard railing" [OSHA Section 1926.500(f)], capable of supporting a 200 pound force in any direction with minimal deflection. This type of barricade is required:

- Around excavations in certain locations
- Around floor openings
- At floor and roof edges

All barricades shall be equipped with an entrance/exit "gate" (no more than 4 feet wide). All barricade gates shall be properly identified with the appropriate signage. Barricades shall be promptly removed when no longer required.

1.16 Construction Equipment

- 1.16.1 Contractors and all tier subcontractors shall comply with the provisions of OSHA and ANSI/ASME 30.5 in the operation, control and inspection of major equipment. All contractor owned or rental equipment shall be inspected prior to use by site Powered Equipment Operations (PEO) shop. This inspection conforms to industry standard and the referenced equipment procedure inspection forms (Ref. EN1088). The contractor shall provide Contract Administrator two (2) days advance notice for equipment inspection. Lifting charts shall be kept with all lifting equipment.
- 1.16.2 No vehicles shall be left unattended unless the engine is turned off, transmission is in park or low gear, and the parking brake is set to prevent unintentional movement. No mobile equipment shall

Rev. Date 06/02/04

be left unattended unless the previous criteria are met and the wheels are chocked in opposite directions or at least two (2) outriggers are down.

Semi-trailers to be loaded or unloaded with the tractor disconnected shall have wheels chocked in opposite directions and a support jack installed under the fifth wheel. The support jack shall be locked and tagged by all exposed personnel to prevent unintentional hook-up or movement.

Semi-trailers to be loaded or unloaded with the trailer connected must have approval of the Contract Administrator.

All vehicle occupants must wear seatbelts.

1.16.3 Truck-mounted, hydraulic, telescoping boom cranes shall not be moved until the boom is fully retracted and lowered to the boom cradle, if provided, and the hook is secured in a tie-down lug at some point on the truck bed. Anti two-blocking devices are required when using any crane or lift truck.

Modifications shall not be made to lifting equipment without manufacturer's written permission. Each custom designed, job made lifting accessory (i.e. grabs, hooks, clamps, etc.) shall be marked to indicate safe working load and shall be tested, prior to use, to 125 percent of their rated load. [Reference OSHA Section 1926.251(a)(4)].

Rigging from buckets or booms of backhoes, front end loaders, dozers, etc. to lay pipe, erect steel, pick and carry, etc. will not be permitted unless a properly installed and tested lifting eye or lug has been attached to the bucket or boom by the manufacturer, and lifting capacities and configurations are specified in the manufacturer's operating manual.

Access to, and egress from, crane cabs, material wagons, truck beds, crawler backhoes, etc. shall be achieved by built-in ladder and or hand holds provided by the equipment manufacturer. If such means are not provided, Contractor shall provide a properly positioned and secured ladder or other appropriate method to gain access/egress.

All mobile equipment, including personnel transportation vehicles having the rear view obstructed, shall be equipped with audible backup alarms or in the absence of such, a flag person shall be used. A flag person shall be used whenever the vehicle or piece of equipment is being operated or backing up in a congested area.

- 1.16.4 Use of a crane or derrick to hoist personnel on a personnel platform is prohibited except when erection or use of other conventional means of reaching the work location would be more hazardous or is not possible because of the work site. Contractor shall submit details of its proposed work platform and rigging method for DuPont approval before conducting any work from a platform suspended from a crane. Included in the details must be clear justification for using a crane-suspended work platform rather than another method (i.e. scaffolds, aerial lifts, etc.). Contractor shall comply with all provisions of OSHA section 1926.550(g).
- 1.16.5 Contractor shall comply with the following requirements for aerial work platforms, man lifts, scissor lifts or bucket trucks:
 - > Operator shall be trained at Contractor's expense
 - Occupancy by more than two people shall not be permitted
 - Personnel in manlifts and bucket trucks shall use full body harness with shock absorbing lanyards secured to the platform or personnel bar.
 - Personnel shall perform their work while standing on the platform floor. Standing on the top rail, mid rail, or toe board shall not be permitted.
 - Rigging from the platform of boom shall not be permitted.
 - Climbing out of the platform to an elevated work location shall not be permitted.

Rev. Date 06/02/04

- > Before equipment travel, the boom must be fully retracted and lowered to the horizontal position.
- Use to raise of lower materials will not be permitted, unless materials fit within the basket and weight requirements of equipment are adhered to.

Scissor lifts and other vertical tower devices used to elevate personnel above ground or floor level shall be operated and used in accordance with the manufacturer's operating manual which shall accompany all equipment on site.

No one shall be permitted to operate a lifting device (material or personnel) unless they have been properly trained. Documentation of the training shall be furnished to DuPont. Also, Contractor shall set up and demonstrate, to DuPont's satisfaction, that each potential operator of a lifting device proposed for use on the site can properly operate the device. The manufacturer's operating instructions shall accompany the device.

Crane operator qualification (per ANSI B-30.5) must be documented and signed by contractor management and furnished to DuPont prior to any crane work. This includes any crane work to be done by subcontractors.

Weight capacities shall be clearly posted on forklift jibs, concrete buckets, crane baskets, etc.

- 1.16.6 The contractor shall submit to DuPont for review and acceptance a critical lift plan at least seven (7) days prior to making the lift and a complex lift plan at least four (4) days prior to lift.
 - > A crane lift is considered critical when one or more of the following conditions exist:
 - The load exceeds seventy-five percent (75%) of the crane's capacity.
 - The load must be lifted by more than one crane (or other rigging).
 - o The lift requires a deviation from the manufacturer's recommendations.
 - A crane lift is considered complex when one or more of the following conditions, but not limited to, exist:
 - Multiple points of rigging suspension are required.
 - o Spreader bars are utilized.
 - Hand rigging is used to level or stabilize the load.
 - Crane operator is not in direct visual contact with the signal person (i.e. radio signaling).

The rigging plan shall consist of:

- Manufacturer, model, and capacity of crane(s)
- Capacity charts
- ➢ Working radius
- Boom length
- Weight of load including rigging
- > How the weight was determined
- Number of parts of line to be used
- Size and capacity of rigging hardware (clings, shackles, etc.)

If requested by DuPont, Contractor shall submit a rigging preplan as outlined above for each non-critical or non-complex lift.

Contractor shall not be permitted to lift a suspended load over personnel. Contractor shall not lift suspended loads over buildings, processes or electrical conductors without the written consent of DuPont.

Rev. Date 06/02/04

Spruance Site Conditions

11

1.17 Rigging

The contractor shall comply with the requirements in the Orange Rigging Handbook (EN-3964) this book will be provided by DuPont. All rigging equipment and hardware (chain falls, slings, etc.) shall be thoroughly inspected prior to the initiation of rigging activities and at least quarterly by a qualified person.

The contractor shall ensure that competent riggers are used for rigging tasks. The contractor shall document, in writing that the persons are competent and shall provide to DuPont, or designee, a copy of the written training material and test results.

Existing lifting lugs, eyebolts, etc. on structures and other equipment are NOT permitted to be used by the contractor for rigging. Overhead supports and I-beams are to be certified by the contractor prior to use for rigging. The contractor shall provide written plans on rigging methods to DuPont prior to the initiation of rigging activities for review and acceptance. Rigging methods shall not include field-modified tools or use of tools outside of tool manufacturers written consent. All structural steel, piping and equipment shall be rigged with at least two points of rigging - no single point picks will be permitted to be dropped from elevated locations.

1.18 Access

Access to exit doors, stairways, electric or elevator panels, and fire extinguishers or other emergency equipment must not be blocked at any time.

1.19 Spray Painting

During spray painting operations where a hazardous warning is posted on the paint can label and controls are inadequate to prevent harmful exposure to employees, Contractor shall provide, at no additional cost to DuPont, and require its employees to use, respirators approved for spray painting operations, and shall comply with OSHA Sections 1926.103 and 1910.134. Precautions stated on the MSDS shall be followed at all times.

1.20 Fall Prevention

Protection is required to prevent personnel or material from falling through floor openings, wall openings, or from roof edges, stairways, elevator shafts, and other elevated locations greater than four (4) feet above floor/ground level including, but not limited to ladders, scaffold erection and dismantlement. Employees must also be protected when working within six (6) feet of roof edges, floor openings, open pits, etc. This list is not all inclusive and work areas need to be evaluated for fall hazards.

Engineering methods such as a system of handrails, mid rails, and toes boards or hole covers are the preferred method of fall protection. Fall protection is required when working from an incomplete scaffold platform. If engineering methods are not feasible*, the protection shall comply with OSHA 1926.500, subpart M and the DuPont "Green Safety Handbook".

*The feasibility analysis must be reviewed and accepted by DuPont.

Rev. Date 06/02/04

Work shall be evaluated by a competent person and Contractor shall submit a fall prevention plan to DuPont for approval prior to starting elevated work. As a minimum, the plan shall include the following:

- > A list of all anticipated tasks to be performed at elevated locations
- > The proposed method(s) of fall protection for each task, using in order of preference:
 - Elimination of fall hazards (through engineered means)
 - Prevention of falls (by changing work procedures or the work environment)
 - Control falls (thought the use of fall arrest equipment)
- > The manufacturer and model number of each component of fall arrest equipment
- Name(s) and qualifications of Contractor's competent person(s) relative to fall protection
- Names of personnel trained in proper use of fall arrest equipment each has been trained to use
- Description of the training program

A fall protection system can include any or all of the following:

- ➢ Full body harness system
 - Anchorage connector
 - Lanyard
 - Energy shock absorber
 - Full body harness
 - Self-locking snap hook
- Retractable lifelines
 - Installation links
 - Energy shock absorber
 - Self –locking snap hook
- > Ladder safety system
- Safety net system
- ➢ Beam straps

Lanyards may not be wrapped and hooked back into themselves unless it has been designed by the manufacturer to do so.

Failing to follow fall prevention rules can result in employee removal from site.

1.21 Ladders and Scaffolding

All ladders (over 6ft) require tie off with a $\frac{1}{2}$ " or larger rope. Secure a $\frac{1}{2}$ " (1.3 centimeter) rope (Nylon, "Dacron", or polypropylene, depending on environmental exposure) to the top back rung of step ladder or to the third rung from the top of straight and extension ladders to provide a tie-off rope when the ladder is set up.

All ladders used on site must be type 1A(300lb. Rating). For information on appropriate ladders for use in these situations, see ANSI Standard A14.2.

Ladders shall be visually inspected by the user before each use and inspected every three months and color coded per site inspection procedures.

Defective ladders shall be promptly removed from the site. Ladders without inspection tags will not be used.

Metal ladders are not to be used for electrical work, near electrical lines or circuits.

Rev. Date 06/02/04

Nutrient Analyzers—Appendix A Test/QA Plan Page A-15 of 29 Version DRAFT March 23, 2005

All scaffolding shall be erected, used, and dismantled in accordance with OSHA 1926 Subpart L and the manufacturer's recommendations.

Contractor shall provide written proof of qualified and competent persons for erection, inspection and proof of training for scaffold users.

All scaffolds shall be inspected for structural integrity and deficiencies by the competent person prior to each shift use. The inspection shall be documented in writing and posted on the scaffold.

Scaffolds will be provided with an access gate at the work platform, be erected as complete as possible and shall be identified with a properly filled out tag prior to use. Tags will be provided by DuPont.

1.22 Ground Fault Protection and Power Cords

Contractor shall provide ground fault circuit interrupter protection for all cord sets, receptacles, electrical tools, and equipment connected by cord and plug which are used or available for use by employees. All GFCI receptacles shall be placed at the source end of electrical service and shall be tested prior to each use.

Drop cords are required to be at least #14 gauge SO or SJO minimum in size. All cords, including welding leads, must be run overhead at the seven (7) foot level whenever possible.

No D/C electrical current producing receptacle on equipment shall be allowed to be used on site. Equipment with existing D/C electrical current shall be disconnected or covered.

1.23 Gas Cylinders

Compressed gas cylinders shall be properly secured on two-wheel hand trucks designed for this use and brought into buildings (building is defined as curbed area) only as needed and removed as soon as work is completed or tanks are emptied.

Unless individual cylinder is equipped with regulating device, it shall have a safety cap secured in place. When transporting cylinders, they must be secured in an approved rack in the vertical position.

While secured and not in use, all gas cylinders must be at least fifty (50) feet from adjacent buildings if space permits. Cylinders shall be stored at least twenty (20) feet from any smoking, spark producing work and open flames.

Combination check valve and flash arrestors are required on BOTH the torch and regulator of any oxygen/fuel rig system. Employees engaged in welding or burning activities must be trained and documentation provided to DuPont.

The use of acetylene is discouraged. DuPont's management must approve, in writing, the use of acetylene for gas cutting, burning, and welding. Do not use acetylene at pressures over fifteen (15) psi (105 pa).

1.24 Tool Inspection

All contractor-furnished portable tools and equipment (including personal protective equipment) shall be maintained in safe working order and are subject to DuPont inspection at any time while on the plant site. DuPont retains the right to prohibit or restrict the use of tools and equipment determined to be in unsafe working condition.

All damaged electrical cords are to be removed from the site. New male or female plugs may be installed; however, taped repairs are not acceptable.

Damage to welding leads must be repaired to the original mechanical and insulating properties of the jacket. Use of heat shrink sleeves or similar is recommended. No repair may be made within ten (10) feet of either end.

All power equipment will be disconnected, locked, tagged, tried, and tested before work is performed on them.

Employees using tools and equipment shall be properly trained in their safe operation and may require documentation/ certification.

DuPont tools and equipment will not be used without written approval from DuPont through a hold harmless agreement.

Power tools shall be disconnected from the power source prior to making adjustments or changing bits and blades, and when left unattended.

Bits from drills and blades from jig and reciprocating saws must be removed when tool is in storage.

All electrical tools must have a ground plug unless tool states "double insulated" on handle or housing.

Contractor shall identify in writing to DuPont, the OSHA qualified person(s) that will be performing periodic inspections per applicable OSHA standards.

1.25 Inspection Color Code System

Contractor shall use the following inspection color code system while on site:

 Quarterly (Ladders, Harness and Lanyard, Rigging, Mag Drills)

 Jan. - March
 Yellow

 April - June
 Orange

 July - Aug.
 White

 Oct. - Dec.
 Red

 Annual (Pipe/Jack Stands)

Annual (Pipe/Jack Stands) Odd Years Green Even Years Blue

1.26 Lead Containing Materials

Nutrient Analyzers—Appendix A Test/QA Plan Page A-17 of 29 Version DRAFT March 23, 2005

The protective coatings applied to the outside of existing pipes, structures and associated hardware may contain lead. Unless the contractor has written verification from DuPont that the specific coatings do NOT contain lead, the coatings shall be treated as lead-containing OR the contractor shall sample the coatings and verify the presence or absence or lead by laboratory analytical methods. The contractor shall comply with OSHA, DuPont Corp. standards, and site safety procedures as outlined in:

- > 29 CFR 1926.62
- > DuPont Engineering Standard S5T
- Spruance Safety Procedure #70.

The landfill disposal of equipment and material, which has lead-containing coatings, will be the responsibility of DuPont. Salvage of the existing equipment and material must comply with federal, state and local regulations, including regulations for lead and lead-containing materials. Downstream receivers of the equipment must be informed that the coatings contain lead.

The contractor shall provide to DuPont, or designee, for review and acceptance, a copy of their written lead program, in accordance with OSHA 20 CFR 1926.62, before beginning any work activities that have the potential to expose personnel to lead and lead-containing materials. Furthermore, contractor shall comply with all other applicable federal, state and local regulations that may apply to lead and lead-containing materials.

All work involving lead or lead-based paint must be performed under the direction of a Lead Supervisor.

The Lead Supervisor must have completed a four day course based on the EPA LBP (lead based paint) abatement supervision curriculum. The course must be designed for professionals supervising work involving LBP abatement or possible exposure to lead.

The contractor shall provide training, medical surveillance, exposure control methods, and air sampling at no cost to DuPont. Contractor shall provide documentation of training, copies of lead hazard assessments, and air sample results to DuPont.

All lead-related activities must be coordinated through the Contract Administrator with the site Lead Coordinator.

1.27 Asbestos Containing Materials

Asbestos and asbestos-containing materials are present on site. Unless the contractor has written verification from DuPont that specific materials do NOT contain asbestos, the materials shall be treated as containing asbestos. Materials, which do or may contain asbestos, are insulating materials, floor tiles, fireproofing, mastics/adhesives, roofing materials, lab stone, transite/galbestos panels and gasket materials.

The contractor shall provide to DuPont, for review and acceptance, a copy of their written asbestos program according to OSHA 29 CFR 19 10.1001 and 20 CFR 1926.1101, before initiating any work activities that have the potential to expose personnel to asbestos and asbestos-containing materials. Furthermore, the contractor shall comply with all other applicable federal, state and local regulations that apply to asbestos and asbestos-containing materials. Contractor shall submit a copy of the company asbestos abatement license, individual asbestos worker certificates, proof of biological monitoring and proof of permit at least twenty-four (24) hours prior to the initiation of asbestos abatement activities.

The contractor shall provide training, medical surveillance, exposure control measures and air sampling at no cost to DuPont. Air sample results for asbestos-related activities are to be provided to DuPont.

All asbestos-related activities must be coordinated with the site asbestos coordinator.

No asbestos or asbestos containing material may leave the site without the consent of the site waste control coordinator. Asbestos containing material must be appropriately bagged and labeled and placed in Asbestos dumpsters or roll-off boxes. Contractor shall be responsible for the preparation of the required manifest and dispose of asbestos containing material.

1.28 Non-Asbestos Respirable Fibers

Contractor shall comply with the requirements of the DuPont Corporate SHE standard S15T, Management and Control of Non-Asbestos Respirable Fibers (NARFs), when respirable fibercontaining materials and used or disturbed. NARFs shall be managed in a way that controls the release of respirable fibers, reduces personnel exposure, and ensures that waste is disposed of safely and in an environmentally sound manner.

1.29 Projecting Materials

Material projecting above horizontal surfaces shall be capped or otherwise protected to prevent the possibility of impaling personnel. Rebar caps must meet manufacturer's specifications for impalement protection.

1.30 Pipe, Ductwork, Cable Trays

Walking on, crawling along, sitting on, or working from pipe, ductwork, or cable trays will not be permitted. Contractor shall provide mean of access and egress adjacent to the work area for personnel working in pipe bridges.

1.31 Tag, Lock, Clear, Try Procedure

Contractor shall follow the site Tag, Lock, Clear, Try procedure before starting work on any plant system and new systems which have been placed in service or turned over to operations. Contractor shall not operate valves or electrical switches, disconnects or starters without prior approval.

Contractor shall review, when applicable, appropriate drawings for complex lockouts regardless of intimate knowledge of the system.

1.32 Confined Space and Vessel Entry

Contractor shall comply with the requirements of Federal OSHA 1910.146, Permit Required Confined Spaces, and Spruance Site Safety procedure #116, Entry into Tanks, Vessels, and Confined Spaces. A confined space entry permit is required prior to beginning work. All entries must be coordinated with DuPont coordinator. (Reference: Permit 1.27.4), All contractors are

Nutrient Analyzers—Appendix A Test/QA Plan Page A-19 of 29 Version DRAFT March 23, 2005

responsible for providing their own rescue team. Contractor shall utilize personnel who have been trained to operate equipment.

1.33 Electrical Qualification

Contractor personnel shall be knowledgeable of and follow all FCSM Safety Procedures attached and made a part of this contract.

Contractors' electrical safety program shall comply with the requirements of NFPA 70E (Standard for Electrical Safety Requirements for Employee Work Places), Part II 2-3.

The contractor shall ensure that any and all Electrical Safety Training (OSHA 1910.331 through .335) is carried out before personnel start work at the site and continues throughout all phases of the work.

The contractor shall also ensure that only qualified and/or certified personnel perform particular tasks associated with particular equipment.

Personnel training records shall be submitted to DuPont and approved prior to beginning work. Annual re-training shall be provided to all personnel expected to perform electrical work. DuPont reserves the right to accept or reject the Contractor's training records or training procedures established for electrical training. Employees sent to work without required training will be denied access to the site.

1.34 Dismantling and Rearranging

Before permitting employees to start any dismantling and rearranging activities, the contractor must assure that the competent person has conducted an engineering survey to the exact scope of work and the method to safely execute it.

The engineering survey must be documented in writing and must be provided to DuPont for review and acceptance at least five (5) working days prior to initiation of D&R activities. The survey must include details on methods of removal, integrity of the structure (including surrounding structures that may be effected) and provisions to safeguard contractor and site personal from the hazards associated with D&R activities.

Contractor shall NOT be permitted to remove, handle, or repair any process system and/or piping unless the operating unit has finished decontaminating the system of piping. Decontamination will be considered complete after the operating unit has verified it in writing.

1.35 Hazard Communication

Contractor and all tier subcontractors shall submit, for DuPont's review and acceptance, Material Safety Data Sheets (MSDS) for all chemicals which will be used on site. A copy of all MSDS shall be submitted and kept on file in the Contract Administration building. The contractor is responsible fro complying with the Federal Hazard Communication Standard (OSHA 1910.1200)and all applicable state and local hazard communication requirements, which include:

Developing and implementing a written hazard communication program for the site, and a copy must be provided to DuPont.

Rev. Date 06/02/04
Nutrient Analyzers—Appendix A Test/QA Plan Page A-20 of 29 Version DRAFT March 23, 2005

- > Training its employees in handling all hazardous materials.
- Maintaining a list of all hazardous materials present in the work place and posting it in a place accessible to all employees. Material Safety Data Sheets must be on file for each material.

Contractor shall obtain the Owner's approval before introducing any hazardous material onto DuPont's property. Such materials shall be properly labeled and strictly controlled by contractor as the use and disposal. Storage and use of personal protection for handling such materials must comply with the instructions on the Material Safety Data Sheets.

DuPont will make available MSDSs for all hazardous chemicals used or manufactured by the site. Copies of the site's MSDSs are maintained and available on electronic database. Please see you Contract Administrator for copies. If electronic database is down, copies of MSDSs may be obtained from Site Medical 7:00am – 5:00pm Monday through Friday. Off hours and weekends, contact the guard office.

1.36 Electrical Welding and Portable Generators

All welding operations and training requirements shall be conducted in accordance with provisions set forth in OSHA 1910.251-255 and the FC&S safety manual.

Welding leads and rods must be inspected prior to use. Rods must be removed from the holder when left unattended.

All equipment must be properly grounded prior to use. This procedure does not apply to generators supplying voltages greater than 110/220.

Welding machines shall be inspected annually, with documentation of inspection made available to DuPont, upon request.

1.37 Excavations and Wall Penetrations

Excavation, including ground and/or concrete breaking/penetrating activities, shall be in compliance with FC&S Safety Manual procedure B-5.1, Excavations and Spruance Site Safety Manual Item 119 Excavation Concrete Breaking and Floor/Wall Cutting and the contractor shall provide a competent person for excavation tasks. The contractor will NOT be permitted to handle any known or suspected chemically impacted soil without written consent of DuPont. Permits are required when penetrating walls, floors, ceilings, etc. to prevent contract with unidentified (electrical sources energized) permits will be provided by DuPont.

1.38 High Pressure Water Cleaning

The contractor shall comply with the requirements of DuPont Standard PPI8, High Pressure Water Cleaning, for water cleaning activities.

1.39 Housekeeping

Rev. Date 06/02/04

Nutrient Analyzers—Appendix A Test/QA Plan Page A-21 of 29 Version DRAFT March 23, 2005

During the course of construction, alteration, or repairs, all debris shall be kept cleared from work areas, passageways and stairs in and around buildings or other structures.

Contractor shall promptly clean up and daily dispose of all scrap materials and waste materials that accumulate from its operations. Should contractor fail to keep its work area orderly, DuPont will perform this work on a time - and - material basis and back charge contractor.

1.40 Pipe Jackstands

Jack stands shall be positive locking (Pin Type) with large enough base to prevent tipping on a level surface. Friction type locking devices are not permitted.

Weight capacity for three legged jack stands shall be 2,000 pounds or manufacturer's recommended weight capacity, whichever is less. Weight capacity shall be plainly marked on the stand.

Three legged jack stands are not to be used to support material larger than 6" in diameter without site management approval. Diameter capacity shall be plainly marked on the stand.

Weight capacity for four legged jack stands shall be 3,000 pounds or manufacturer's recommended weight capacity, whichever is less. Weight capacity shall be plainly marked on the stand.

Four legged jack stands are not to be used to support material larger than 36" in diameter or manufacturer's maximum recommended diameter, whichever is less, without site management approval. Diameter capacity shall be plainly marked on the stand.

Jack stands with casters shall have a locking device. Locking device shall be in locked position when not being moved.

1.41 Pneumatic Testing

All gauges to be used in pneumatic testing and/or inflation type procedures should be calibrated before the first use and on a routine basis.

1.42 Powder Actuated Devices

Prior to the use of powder actuated device, such as a Hilti Nail gun, written permission must be granted by the Contracts organization.

1.43 Railroad Operations

The contractor shall not interfere with the normal operation of the plant rail system. Personnel and equipment shall not work or be operated within eight (8) feet of the rail centerline without the consent of DuPont. Work on or within eight (8) feet of the centerline requires that all potential affected individuals lock out the track at a point designated by the contract administrator and flag with blue ball sign.

1.44 Temporary Lighting

The contractor shall ensure that sufficient levels of illumination exist. Temporary lighting stringers shall be hard or extra hard usage cord construction with lap covers and be installed, supported, and maintained according to the requirements of the National Electric Code (NEC), Articles 230 through 503. Lighting stringers must be run at seven (7) foot elevations or higher.

Portable temporary lighting shall be 3-wire molded case type with hard or extra hard usage cords and shall be protected by ground fault circuit interrupters (GFCIs). The contractor shall protect personnel and equipment from the hazards associated with use of portable electrical lighting in damp or wet and potentially hazardous (explosive) environments.

1.45 Throwing Material

Material, equipment and tools shall not be thrown or dropped from roofs, buildings, platforms, etc. or from one individual to another or to another location.

1.46 Vehicle Safety

Vehicle operator shall obey all traffic regulations and signs on Plant property. All motor vehicles and material handling equipment will be required to be equipped with seat belts and worn by occupants. An exception to this requirement is for certain equipment which the manufacturer has intentionally not installed seat belts (i.e. equipment without rollover protections.)

Except for material delivery, only on contractor owned vehicle, meeting the minimum insurance requirements, in safe condition and used only by licensed supervisory personnel will be permitted on site. A vehicle pass authorized by the Contract Administrator is required for each vehicle entering the site.

Plant speed limit of 15 mph shall be observed.

1.47 Waste Material Control

Waste equipment oil shall be disposed off site.

Contractor shall ensure that its operations comply with the oil spill prevention provisions of 40 CFR 112. Further, the spillage of oil or any other foreign substance onto the ground or into plant sumps, trenches or ditches is strictly prohibited. All spills are to be reported to the contract administrator immediately after occurrence.

DuPont shall provide metal containers for the collection and separation of waste, trash and other refuse. Containers used for garbage, acids, caustics, harmful dust, etc. shall be equipped with covers. Contractor shall dispose of all trash and refuse in an on site disposal area at frequent and regular intervals.

The contractor shall not accumulate salvaged materials on site for periods in excess of ten (10) days. Asbestos material, lead debris and any other regulated material shall be placed in properly labeled dumpsters or receptacles as soon as possible after removal or at least daily.

Contractor shall not remove any excavated soils from the site. DuPont will dispose of all soils.

Nutrient Analyzers—Appendix A Test/QA Plan Page A-23 of 29 Version DRAFT March 23, 2005

All flammable and combustible liquids shall be stored in approved flammable storage cabinets. Open storage of flammables and combustibles will not be permitted without the written consent of DuPont. Secondary containment shall be provided capacity for 150% of the stored liquids and be protected against accumulations of rainwater or other debris.

2.0 FIRE PROTECTION

2.1 Procedures

Contractors shall observe DuPont's fire, fume release and disaster procedures. Contractor's job supervisor will be given a copy of and be oriented by DuPont on the alarm and evacuation procedures and shall ensure that all contractors' employees are thoroughly familiar with those procedures. Contractor will be reimbursed on a time-and-material basis for lost time resulting from evacuations due to fume release, or fire disaster drills.

2.2 Smoking

Smoking will be permitted only in areas designated by DuPont. Smoking is not allowed in any building, vehicle or on any roof.

Smoking by a contractor employee in any non-designated area shall be considered cause to require contractor to remove that employee from the plant site.

2.3 Flammable Materials

Storage and use of flammable materials will be subject to DuPont's approval. Safety cabinets and cans shall be used for storing and dispensing flammable liquids and must have prior Owner's approval. Refueling of gas powered tools or equipment is not permitted inside of building, without DuPont's approval.

2.4 Fire Extinguishers

Contractor shall furnish a sufficient number of fire extinguishers to protect its work area. Extinguishers shall be located, maintained and inspected according to NFPA 10. Fire extinguishers shall be visually inspected monthly. Contractor shall not consider Owner's extinguishers as fulfilling the requirement to furnish extinguishers.

2.5 Oily Rag Storage

Dirty and oily rags shall be kept in fireproof metal containers with self-closing lids and removed from premises daily to prevent spontaneous combustion.

Nutrient Analyzers—Appendix A Test/QA Plan Page A-24 of 29 Version DRAFT March 23, 2005

2.6 Protective Structures

Any plastic, tarpaulin, or other material used to construct a hut, tent, or similar protective structure, shall be constructed of flame retardant materials.

2.7 Burning and Welding

One portable fire extinguisher having a rating of not less than 2-A:20-B:C shall be kept at each location where welding or cutting is to be performed and one portable fire extinguisher of a rating of not less than 2-A:20-B:C shall be attached to or available on all portable welding machines and burning carts.

3.0 CONSTRUCTION FACILITIES

3.1 Compressed Air

Compressed air is not available within the work area. Contractor shall provide its own source of compressed air.

3.2 Drinking Water and Sanitary Facilities

Drinking water will NOT be available for contractor personnel consumption. Contractor shall furnish and maintain water coolers in a safe and sanitary condition for their employees. Drinking water coolers shall be cleaned and sanitized on a regular basis and shall be sealed and identified (dated) to prevent contamination from various potential sources. Drink cup dispensers (disposable cups) and waste receptacles shall be provided at each water cooler.

Sanitary Facilities will be designated by your Contract Administrator.

3.3 Electric Power

DuPont will furnish temporary power when available for the operation of small tools. Contractor shall make all connections (3-wire grounded/and run any extensions required) Ref. 1.22 of this document for ground fault circuit interrupter requirements.

3.4 Identification

Contractor shall post a sign, of a size and character consistent with the size, nature and duration of the contract, identifying its site headquarters, which may be an office, trailer, toolbox, or storage area.

3.5 Plant Facilities

Contractor's personnel will not be permitted to use plant facilities such as lunchroom and toilets unless directed by the Contract Administrator.

3.6 Site Access

Normal site working hours are 7:00 a.m. to 5:30 p.m., Monday through Friday. Entry and exit at all time shall be through the designated construction gate.

Designated routes will be established for access to the work area and for movement of material and equipment. Movement of material and equipment shall be scheduled with DuPont.

3.7 Telephones and other Communication Equipment

Contractor shall arrange telephone service, as needed, through a local telephone company. The contractor shall furnish DuPont and the Contracts Group with a means of contacting Job Foreman. No portable cell phone usage will be permitted while driving on site.

All portable electronic devices such as radios, pagers and telephones shall be electrically rated for classified areas. The classification shall be clearly identified by the manufacturer on the cast of the device. Only "Class V" rated devices shall be freely carried across the plant.

3.8 Temporary Construction (TC) Facilities and Trailers

Contractor shall comply with DuPont Standard SI8G, Office Trailers, for the use of trailers and other TC facilities provided by the contractor.

3.9 Water

Water for construction is not available; however, the contractor will be responsible for providing methods (hoses) to provide water to the work site where the source is made available to the contractor.

NOTE: Contractor shall NOT be permitted to OPERATE any valves, including water, fire water, or process systems. Where water is provided to the contractor, the contractor will be responsible for installing their own valve, downstream of the facility valve. ONLY THE OPERATING UNIT IS PERMITTED TO OPERATE FACILITY VALVES.

Fire hydrants shall not be used as a source of water without DuPonts written approval.

4.0 WORKING CONDITIONS

Rev. Date 06/02/04

Nutrient Analyzers—Appendix A Test/QA Plan Page A-26 of 29 Version DRAFT March 23, 2005

4.1 Cellular Telephones

Non-intrinsically safe cellular phones and two-way radios are not allowed in electrically classified areas. These devices have the potential to create sparks and ignite flammable chemicals that might be present in classified areas. Cellular telephones are not to be used in process control rooms, electrical control rooms or within 25 feet of operating equipment or E & I control panels. Cellular phones are not to be used by the driver of any moving vehicles on site property. Cellular phones with picture taking capabilities are not permitted on site.

4.2 Co-occupancy

Contractor shall provide all measures required to protect existing facilities and work performed by others from damage due to Contractor's (including its tier subcontractors') operations or negligence. DuPont may suspend Contractor's operation until such protective measures are provided and the cost for stand by of contractors, its tier subcontractors, or other contractor's men and equipment shall be for contractor's account.

4.3 Coordination with Others

Where contractor's work must be performed in conjunction with work by others, contractor shall cooperate with DuPont in scheduling, coordinating and sequencing its work with that of others so all work may proceed with minimal interference or delay.

4.4 Diversity

Creating the proper environment is a significant business issue with very high stakes for the contractor and for DuPont. Individuals uniquely contribute to make each company what it is. Recognizing we are a diverse people and will become more diverse, it is essential that we have a environment that value diversity. One of DuPont's key pursuits is to create this environment, where the vale of diversity is shared and all people have a equal opportunity to make their maximum contribution.

DuPont does not condone nor will it tolerate any activities, whether they be physical, verbal or written, which an individual and/or group would perceive as offensive, demeaning or exploitative, based on religion, gender, nationality, race, etc.

The contractor is responsible for informing personnel in his/her employ of this policy. Any persons found in violation of the above policy may be subject to dismissal from the site.

4.5 English Requirement

Anyone working on site shall be required to speak, read and understand English sufficiently enough to be able to properly work or respond to hazard warnings, safety signs and emergency signals. If the above criteria cannot be met, one translator for every 5 non-English speaking persons shall be present and shall remain in visual/vocal contact with these persons at all times. If visual or vocal contact is broken, the contractor agrees to immediate removal from the site and/or contract termination.

4.6 Material Shipment and Storage

Truck shipments to the plant are acceptable. Contractor shall coordinate shipment with Owner to avoid interfering with plant operations. Access for truck shipment to the storage and work areas is by construction road with sixteen-foot overhead obstructions. Contractors shall unload and handle its material. Motor control centers and fan rooms cannot be used as storage areas.

Reasonable ground space, but DuPont will make no structures, for storage of materials, office, etc., available to Contractor

Material being furnished by DuPont will be made available to Contractor at a designated location on site.

4.7 Prohibited Items

In addition to those items noted in DuPont's "Green Safety Handbook, cameras, no cameraenabled devices, hand warmers; (use of lighters and matches except in designated smoking areas), radios, tape recorders, contact lenses (in process areas) and glass containers are strictly prohibited on plant property.

4.8 Testing for Substance Abuse

4.8.1 Contractor shall not assign any employee to this site unless proper documentation is presented stating that such employee has taken a "7 panel" drug screen and the screen has proved negative.

Proof and/or confirmation may be by a "Letter of Certification", signed by an officer of the company, listing all employees who will be assigned to the DuPont site, their social security numbers, the date of the drug screen, and confirmation that the results were negative. Test shall be performed within 90 days of initial assignment to the site.

- 4.8.2 Contractor shall provide annual random testing for employees performing work in any PSM covered area as designated by DuPont. Documentation shall be provided to Contract Administration Safety Office stating that the employee has been randomly tested and the screen has proved negative.
- 4.8.3 Contractor shall develop and implement procedures to test its employees for drug (and alcohol) use when Contractor suspects that a performance deviation, an incident, or unusual behavior of one of its employees on DuPont's property is related to drug (or alcohol) use.
- 4.8.4 As a matter of policy, DuPont does not utilize blood test in connection with substance abuse control. To be consistent with said policy, contractor and all tier subcontractors shall not be permitted to utilize blood tests for substance abuse control related to work performed for DuPont.
- 4.8.5 All testing facilities and services utilized by the contractor must meet or exceed the requirements as set forth by DuPont.
- 4.8.6 Contractor shall provide for sample collection, drug screening and confirmation laboratory testing services at no cost to DuPont.

Rev. Date 06/02/04

4.9 Tool and Equipment Control

DuPont will not be responsible for the loss or theft, either on the plant or in the parking lot, of contractor's tools or equipment, or contractor employee's personal belongings.

No tools or equipment shall be borrowed from DuPont except with Hold Harmless agreement from DuPont Representative with the appropriate signatures.

4.10 Visitor Control

All visitors shall be escorted by Contractor's site supervision and must have and be wearing the appropriate personal protective equipment.

4.11 Insignias

Employees should be aware that when in the workplace, employees are expected to behave in a way that ensures all employees are treated with dignity and respect. Displaying disrespectful insignias, signs, pictures, T-shirts and the use of profane or offensive language is not appropriate in the workplace today. While it is impossible to establish guidelines for everything, it is appropriate to clarify expectations regarding the Confederate flag.

The Confederate flag continues to create controversy and concern in our workplace and society in general. It has different meanings to different individuals; and for many, represents a symbol of disrespect and hostility. It is considered offensive and disruptive in the workplace and should not be displayed in any form on Plant property including parking lots.

4.12 Parking/Gate Entry Site Procedures

Gates: Main Gate - Jefferson Davis Highway Construction Gate - South End Commerce Road Hours: Both Gates - 24 hours a day - 7 days a week

Gate Entry: Main Gate - All professional and site services contractors such as cafeteria, janitorial, visitors and consultant services will enter through the main gate. Construction Gate - All vendors and other contractors (general, electrical, environmental, etc.) will enter through the construction gate.

Emergency Entry - All contractor personnel responding to an emergency situation will enter through the construction gate under abnormal circumstance.

Parking: Closed Shop (union) - Contractors will park in West part of the lower Green lot "C" located adjacent to the pallet storage area.

Open Shop (non-union) - Contractors will park in the East part of the lower Purple lot "D" located adjacent to Commerce Road.

Parking Areas Described above will be designated with color coded signs: color coded permits will be issued by the Contractor Gate Pass Administrator to all eligible employees and contractor employees as described above. Permits are designed to hang from a vehicle's rear view mirror and must be displayed when the vehicle is parked in on site parking lot. A log system containing

Rev. Date 06/02/04

Nutrient Analyzers—Appendix A Test/QA Plan Page A-29 of 29 Version DRAFT March 23, 2005

employee name, contractor name, color code, permit number and vehicle license number will be maintained. Employees may provide up to two-license numbers.

Follow posted speed limit signs. The maximum speed limit in all parking lots is five- (5) mph.