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Southern Wood Piedmont Company

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SUPERFUND SECTION

October 7, 1999

Mr. Stuart F. Parker, Jr., Hydrogeologist
North Carolina Superfund Section
401 Oberlin Road, Suite 150
Raleigh, NC 27605-1350

Re: September 3, 1999 Review and Comment on Submittal: Schnabel Engineering Report on Remedial Investigation, SWP – Wilmington Site NCD 058 517 467

Dear Mr. Parker:

Please find enclosed a draft Supplemental Remedial Investigation Workplan for the Southern Wood Piedmont Company and North Carolina State Ports Authority Site at Wilmington, New Hanover County (USEPA ID# NCD 058 517 467). This supplemental plan is provided in regard to the subject September 3, 1999 letter from your office to mine.

The supplemental work plan is designed to address additional sampling required to complete the remedial investigation. The plan also addresses additional technical comments in the letter.

Please contact me at 864-599-1070, extension 103 if you have questions or comments.

Sincerely,

W. P. Arrants
Manager of Environmental Affairs/
Regulatory Compliance

CC: Greg Kuntz – Schnabel Engineering
L. Bedsole – NC Ports Authority
M. Maritato – Ogden Environmental & Energy Services

4327bw

Supplemental Remedial Investigation Workplan

Southern Wood Piedmont Company and North Carolina State Ports Authority Site Wilmington, New Hanover County, North Carolina

Prepared For

Southern Wood Piedmont Company
Spartanburg, South Carolina

Prepared By

Schnabel Engineering

October

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LIST OF ATTACHMENTS

Attachment A	NCDENR September 3, 1999 Draft RI Comment Letter
Attachment B	EISOPQAM Sample Control, Field Records, and Document Control
Attachment C	EISOPQAM Sediment Sampling
Attachment D	Triangle Laboratories Quality Assurance Manual

DRAFT

SUPPLEMENTAL REMEDIAL INVESTIGATION WORKPLAN
SOUTHERN WOOD PIEDMONT AND
NORTH CAROLINA STATE PORTS AUTHORITY SITE
WILMINGTON, NEW HANOVER COUNTY, NORTH CAROLINA
NCD 058 517 467
October 8, 1999

1.0 INTRODUCTION

Schnabel Engineering Associates, Inc. submitted a Draft Remedial Investigation Report (RI) on June 24, 1999 for the Southern Wood Piedmont (SWP) and North Carolina State Ports Authority (NCSPA) site to the North Carolina Department and Environment and Natural Resources (NCDENR). Stuart Parker of NCDENR reviewed the Draft RI report and provided comments in his letter dated September 3, 1999 (Attachment A). Southern Wood Piedmont has been directed to submit a Supplemental RI Workplan to address additional sampling requirements to complete the remedial investigation. The requirements and presentation order for the Supplemental RI Workplan are listed in the May 24, 1999 Administrative Order on Consent (AOC). Presented below is the proposed workplan.

2.0 SCOPE OF WORK

The scope of this investigation includes the following activities:

⇒ Dibenzodioxins/dibenzofurans sediment sampling in drainage ditch, Greenfield Creek, and the Cape Fear River.
⇒ High resolution (closer spacing) sediment sampling for detected constituents in drainage ditch and Greenfield Creek.
⇒ Fish tissue sampling in drainage ditch and Greenfield Creek.
⇒ Update the Human Health and Ecological Risk Assessment to 1997 guidance.
⇒ Ecotoxicity Testing and Chronic Exposure Assessment of drainage ditch and Greenfield Creek.
⇒ Initial evaluation of technical feasibility of partial DNAPL recovery.
⇒ Initial efforts in obtaining Perpetual Land Use Restrictions in accordance with 1999 guidelines.
⇒ Modifications to the Draft RI as presented in the Technical Comments section of the September 3, 1999 NCDENR review and comment letter. The modifications will be presented as an addendum to the Draft RI included in the Supplemental Remedial Investigation (SRI) report.
⇒ Verification and reference documentation of the absence of environmentally sensitive areas.

3.0 SUPPLEMENTAL REMEDIAL INVESTIGATION WORKPLAN

Section VI. C. of the AOC lists the requirements and contents for the Supplemental Remedial Investigation workplan. The requirements for the content of the workplan are detailed under Sections VI. D., E., F., G. and H. At the Divisions (NCDENR) sole discretion, requirements that duplicate previous submittals, may be omitted from future plans and reports.

3.1 Section VI. D., 1 - 13 - Previously Submitted Requirements

Section VI. D., 1 Site location information was previously submitted in the Draft RI Section 1.0.

Section VI. D., 2 A summary of hazardous waste management practices was previously submitted in the Draft RI Section 2.0.

Section VI. D., 3 A USGS topography map displaying a one-mile radius of the site was previously submitted in the Draft RI Section 3.0.

Section VI. D., 4 A site survey plat was previously submitted in the Draft RI Section 4.0.

Section VI. D., 5 A description of local geologic and hydrogeologic conditions was previously submitted in the Draft RI Section 5.0.

The Draft RI will be updated by an addendum presented in the SRI noting that the slug tests were not completed on wells that fully penetrate the respective aquifer. A statement will be included that the respective hydraulic conductivities are consistent with the composition of the aquifer materials.

An addendum to the Draft RI will be made stating that the tidal gate will not necessarily prevent sediment transport from Greenfield Creek to the Cape Fear River, especially during high creek discharge events at low river tide. Nor would the gate exclude all swimming organisms in the Cape Fear River from entering Greenfield Creek. Immature fish characteristically use tributaries to avoid predation and food competition in larger water bodies. It will also be noted that mature game fish were observed in Greenfield Creek, both during the 1997 Expanded Site Inspection and during an off-site reconnaissance by NCDENR on April 20, 1999.

Section VI. D., 6 An inventory of all well, springs, and surface water intakes was previously submitted in the Draft RI Section 6.0.

An addendum to the Draft RI report will be prepared noting that the emergency surface-water intakes on Smith and Toomers Creeks have been unused for several decades due to salt-water encroachment.

Section VI. D., 7 Identification of environmentally sensitive areas on and adjacent to the site was previously submitted in the Draft RI Section 7.0.

The appropriate sensitive environment contacts will be made to provide documentation in the SRI of the absence of sensitive environments not previously identified on or adjacent to the site.

Section VI. D., 8 A copy of the owner's deed was previously submitted in the Draft RI Section 8.0.

Section VI. D., 9 A listing of previous owners and period of ownership was previously submitted in the Draft RI Section 9.0.

Section VI. D., 10 An operational history was previously provided in the Draft RI Section 10.0.

An addendum to the Draft RI will be prepared noting that Greenfield Creek was channelized between 1938 and 1949, suggesting that contaminant migration to the ditch and creek occurred subsequent to that time.

Section VI. D., 11 A list of hazardous substances used or stored at the site was previously submitted in the Draft RI report Section 11.0.

Section VI. D., 12 The environmental permit history for the site was previously submitted in the Draft RI Report Section 12.0.

The position of the new ditch in relation to the covered ditch will be described as an addendum to the Draft RI in the SRI.

Section VI. D. 13 A summary of all environmental investigations, reports and laboratory data was previously provided in the Draft RI report Section 13.0.

Table 2-5 will be updated in the SRI to list the State Soil Remediation Goals for each dioxin and furan species. In addition, possible semi-volatile sources > 0.5 miles upstream will be identified and the source cited. The source of the statement was from the Site Investigation Prioritization. This report will be reviewed and the location of the potential sources will be determined.

The samples used in the Geraghty and Miller, Inc. 1996 report will be listed in the SRI that indicate the described degradation in total wood-preserving constituent concentrations in the landfarm.

Cape Fear River sediment sample SS-14 will not be used as background because of potential impact from the former Wilmington Coal Gasification Plant. The range of values in sediment samples SS-16, SS-20, SS-22, and SS-24 will be used to demonstrate background concentrations.

A statement will be added as an addendum to the Draft RI indicating that copper was detected in the surface water, but at concentrations less than the Class SC water quality standard.

3.2 Section VI. D., 14 - Proposed Procedures for Characterizing and Delineating Contamination Sources

3.2.1 Sediment Sampling

The sediment samples in the waterways have not been tested for dibenzodioxins/dibenzofurans. Select sediment samples from previously sampled locations will be resampled for dioxins/furans to determine if a release has occurred to the drainage ditch, Greenfield Creek and the Cape Fear River. A background and two downgradient samples are proposed for each waterway (Figure 1). If dioxins/furans are detected above background, then all previously collected sediment samples and proposed sediment and fish tissue samples will be analyzed for dioxins/furans.

A total of fifteen (15) additional sediment samples will be collected from the drainage ditch and Greenfield Creek to provide for a higher resolution sediment characterization to delineate "hot" segments of the ditch and creek. This will provide a sample spacing of 200 feet between samples (Figure 1).

In order to generate data in support of the ecotoxicity assessment, the physico-chemical characteristics of all proposed sediment samples will be determined. These will include particle size distribution, total organic carbon, salinity, pH, and ammonia.

Bulk composite sediment samples will also be collected for the sediment toxicity assessment. For each composite sample, a total of 5 subsamples will be homogenized following Environmental Response Team guidelines (EPA, 1994). The 5 locations will be selected to represent the different conditions within the drainage ditch and creek system. It is anticipated that 2 composite samples will be collected from the drainage ditch and 3 composite samples will be collected from the creek. In addition, 1 composite sample will be collected from the Reference Area. The Reference Area will be the same as that used for prior investigations (i.e., on Greenfield Creek between the site and Greenfield Lake dam).

3.2.2 Fish Tissue Sampling

Up to four fish tissue samples will be collected in Greenfield Creek downstream of the confluence with the drainage ditch. Target species will be recreational species including bass, catfish, crappie and bream that are potentially used for human consumption. Fillets may be composited from several game fish of the same species due to the likely small overall number of fish of edible size that can be harvested from this system. If successful, up to four composite samples of fish fillets will be collected within Greenfield Creek, and 1 composite sample of fish fillets will be collected from the drainage ditch and Reference Area, respectively.

Up to three composites of small fish will be collected from 3 representative areas within Greenfield Creek, 1 composite of small fish will be collected from the drainage ditch, and 1 composite will be collected from the Reference Area. The sampling areas will be determined based upon the potential for suitable avian feeding habitat.

3.2.3 Human Health and Ecological Risk Assessment

A baseline Human Health Risk Assessment (HHRA) and screening-level Ecological Risk Assessment (ERA) were prepared for the SWP Wilmington site in 1996. These assessments preceded the collection of supplemental sampling data during USEPA's Expanded Site Inspection, which occurred in 1997. The HHRA and ERA were submitted by SWP as part of the Draft Remedial Investigation Report, which was issued to NCDENR on June 24, 1999. The following HHRA and ERA supplemental work plans were developed to address comments raised by NCDENR in their September 3, 1999 letter to SWP.

3.2.3.1 Human Health Risk Assessment Workplan

Fish Tissue Sampling/Fish Ingestion Exposure Pathway

The 1996 HHRA relied upon modeled concentrations in fish using a predictive food web model to evaluate the fish ingestion exposure pathway since fish tissue sampling data were unavailable. This conservative modeling approach resulted in estimated risks that were within the range of risks deemed acceptable by USEPA at CERCLA sites. For the supplemental HHRA, SWP will use actual fish tissue (fillet only) sampling data to the extent that fish desirable for human consumption are caught using proposed fish sampling techniques. As discussed below in the ERA supplemental work plan, NCDENR staff has observed the presence of bass and other game fish in Greenfield Creek.

Once fillet sampling data are available, potential carcinogenic and noncarcinogenic risks associated with consumption of fish from the Greenfield Creek drainage system will be calculated. Results will be summarized in tables and text that clearly lay out all assumptions and calculations. It is SWP's understanding that, depending upon the fish sampling results, the Department may post a fish consumption advisory.

Specific Comments on HHRA

Since completion of the 1996 HHRA, supplemental sampling data are available which were collected as part of USEPA's Expanded Site Inspection. Still more data is proposed in support of addressing the Division's comments on the Draft RI. Thus, exposure point concentrations (EPCs) for chemicals of potential concern (COPC) for all scenarios will be revisited to the extent that new sampling data will be included in the various exposure assessment evaluations.

Where the Department's HHRA reviewer (in a letter to Jack Butler dated June 17, 1996) has noted potential errors or discrepancies, any errors will be corrected or additional clarification will be provided. To the extent practicable, the supplemental HHRA and ERA will build upon the baseline reports and submit HHRA and ERA supplemental reports in addendum format in the SRI.

The supplemental HHRA will provide the following:

- An update of physical attributes of the site, to the extent any may have changed since 1996.
- A re-analysis of all data, and selection of chemicals of potential concern (COPC), based on the expanded dataset.
- Streamlined toxicity profile summaries for any chemicals that are not included in the 1996 report.
- A table of all current carcinogenic and noncarcinogenic toxicity values.
- A review of exposure setting/pathway analysis to ensure that prior assumptions are representative of site conditions today. Exposure pathways previously discussed in the HHRA include:
  - incidental ingestion of soils and sediments
  - dermal contact with soil/sediment
  - dermal contact with groundwater
  - inhalation of soil particles (dust)
  - inhalation of soil vapors
  - inhalation of groundwater vapors, and
  - fish ingestion.
- An expanded fish ingestion exposure analysis will be provided, to address the potential of anglers consuming fish from the drainage ditch and Greenfield Creek.

3.2.3.2 Ecological Risk Assessment Workplan

A Screening-Level Ecological Risk Assessment (SLERA) was prepared for the site in 1996 (ChemRisk, 1996). This document was developed using analytical data collected over the period from 1985 through 1995, and was prepared in accordance with guidance that was available at the time. This document will be updated to reflect the guidance set forth in *Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments* (herein referred to as "ERA Guidance"; EPA, 1997). The ERA Guidance specifies that an eight step process be used to perform ecological risk assessments within the Superfund Process. These steps include the following:

- Step 1 – Preliminary Screening Level, which includes a site visit, preliminary Problem Formulation, and preliminary Toxicity Evaluation.
- Step 2 – Screening Level, which includes development of Exposure Estimates and Preliminary Risk Calculations.
- Step 3 – Problem Formulation, which includes toxicity evaluation, development of a preliminary site conceptual model and exposure pathways, and development of assessment endpoints.
- Step 4 – Study Design and DQO Development. This includes development of the Work Plan and Sampling and Analysis Plan based upon results of the previous three steps.
- Step 5 – Verification of Field Sampling Design. This step includes a determination of the feasibility of the field program as outlined in Step 4.
- Step 6 – Site Investigation and Data Analysis.
- Step 7 – Risk Characterization. This step includes more refined and detailed quantification of potential site risks, and is generally a more realistic evaluation of risks than was performed in Step 2.
- Step 8 – Risk Management.

Scientific/Management Decision Points (SMDPs) are made as part of Steps 2 through 8 in this process. SMDPs are "checkpoints" in the ERA process to verify that the work that was completed at each step was complete and to determine the need, if any, for proceeding to the next step. SMDPs are critical as they provide the opportunity to exit the process, since all eight steps may not be required for all site evaluations.

The completed SMDPs and their results are discussed in the next section. This Work Plan outlines the elements of ERA Step 4. The subsequent steps in the ERA process will occur following initiation of the field activities.

Summary of Screening-Level Ecological Risk Assessment

Table 2 summarizes the results of the SLERA and compares the results to each of the appropriate steps specified in the ERA Guidance and their appropriate context. The key conclusions of the SLERA are shown below:

- Metal concentrations in sediments of the drainage ditch and Greenfield Creek system are generally below levels that would be of concern based on the sediment toxicity benchmarks that were used. Cape Fear River sediments have concentrations of metals that were slightly higher than the benchmarks; however, AVS/SEM results indicate that most locations have sufficient chelating properties (including TOC) to reduce the bioavailability of these metals to aquatic organisms.

- Potential risks in the ditch/Creek sediments appear to be currently confined to benthic macroinvertebrates. However, despite the elevated HQs derived for benthic organisms, observations made on the community composition indicate that they are still well represented by taxa that are suited for freshwater habitats having shallow surface waters, low flow, and silt/sand substrate.

- Risks associated with the direct contact and ingestion of sediments by benthic invertebrates, and to a lesser extent, the locally abundant spot, have been shown to present a potential hazard to these species at some riverine locations. These risks have been conservatively identified based on hazard quotients that do not incorporate the use of physicochemical factors that control bioavailability (e.g. AVS and TOC), and indicate the need for further investigation.

- The potential risks from PAH exposure in the Cape Fear River are also predominately attributed to direct contact and ingestion by benthic organisms. Potential hazards of PAH exposure for the spot appear to be limited to only a few locations in the study area (e.g. the old slip, and the north shoreline near Pactank Bulk Chemical Storage Facility which are of limited size relative to the overall habitat range of this species. Finally, as indicated by background concentrations of PAH in the Cape Fear River, the potential hazard of PAH exposure to these fish is more than likely not restricted to sediments or areas adjacent to the site.

The SLERA addressed the conservative screening presented in Steps 1 through 3 of the ERA Guidance. The results from the SLERA indicated that potential adverse effects were estimated for benthic invertebrates, based upon sediment benchmarks, and fish, based on estimated body burdens calculated from a conservative uptake model. No potential adverse effects were identified for the piscivorous avian receptor (great blue heron). The current Work Plan describes the elements of ERA Step 4, which represents the baseline ecological risk assessment (BERA) for this project.

BERA Scope of Work

The relevant activities for ERA Step 4 for the SWP project have been outlined in Table 3. Based upon the results of the SLERA, the principal focus of the current Work Plan will be the assessment of potential benthic toxicity due to exposure to COPCs. However, since additional sediment and surface water samples were collected after the SLERA was produced, and additional samples (and analytical parameters) are planned for the current SRI work scope, the same receptors and pathways presented in the SLERA will be re-evaluated using the new analytical results.

Problem Formulation

Elements of the problem formulation step and the site conceptual model that were developed for the drainage ditch, Greenfield Creek, and adjacent areas of Cape Fear River in ERA Step 1 still apply to the current activity. However, it will be extended to evaluate the potential sediment toxicity to benthic organisms by performing standardized sediment toxicity bioassays. The Site Conceptual Model is shown in Figure 2.

Analytical Database

The SLERA was prepared based upon analytical data collected from 1985 through 1995. Additional samples were collected following completion of this 1996 report. These include 21 soil samples, 21 subsurface soil samples, 20 sediment samples, 9 surface water samples, and 42 groundwater samples, and were collected as part of the Expanded Site Inspection (Black and Veatch, 1997). Relevant portions of these results will be incorporated into the BERA.

Supplemental Field Sampling

Additional field sampling will be performed in support of the current project. This will include the following:

- Collection of sediment samples from 24 locations for chemical analysis. These will include collection from areas with known spatial data gaps, as well as the collection of samples for additional chemical analyses (e.g., ammonia, select dioxin/furan congeners).

- Collection of 5 composite samples for toxicity testing, as well as 2 composite samples from within a reference area. These samples will also be analyzed for chemical parameters.

- Collection of small fish for chemical body burden analyses for the BERA.

- Collection of game-size fish for the HHRA.

Composite Samples for Toxicity Testing

Bulk composite samples will be collected for the sediment toxicity assessment, using a similar collection method as specified above for the individual sampling stations. For each composite sample, a total of 5 subsamples will be homogenized. The 5 locations will be selected to represent the different conditions within the drainage ditch and creek system.

Whole-body Fish Composites –Small Fish

Small fish may be preyed upon by piscivorous avian species. In the SLERA, the PAH concentrations were estimated using an equilibrium partitioning model. Since most aquatic organisms can readily metabolize PAHs, collections of small, whole body fish composites will be performed to provide empirical data for the quantification of risks to the piscivorous avian receptor.

Fillet Fish Composites – Game Fish

NCDENR staff has observed the presence of bass and other similar game fish within the drainage ditch-Greenfield Creek system. In order to estimate the potential human health risks associated with the consumption of these fish, fillets will be composited from several game fish collected within the system.

Physico-Chemical Characterization

In order to generate data in support of the ecotoxicity assessment, the physico-chemical characteristics of the composited sediments will be determined. These will include particle size distribution, total organic carbon, salinity, pH, and ammonia.

- *Particle size distribution:* Sediment particle size distribution determines the types of benthic invertebrate species that are expected to be found in a given sediment (See Table 4).

- *Total organic carbon (TOC):* TOC is a critical variable that regulates the bioavailability of non-ionic organic contaminants in sediments (Di Toro et al., 1991).

- *Salinity*: Salinity must be measured to match this variable to the known salinity tolerances of the candidate benthic test species (See Table 4).

- *pH*: pH affects the behavior of many chemicals (e.g., metals, ammonia) and is tolerated differently by benthic invertebrates.

- *Ammonia concentration:* Ammonia can be present in sediments, irrespective of their origins or anthropogenic contaminant loads. It is produced when nitrogenous compounds in sediment pore water are reduced to ammonia by bacterial metabolism. This compound

could be present in sediment samples from the creek and ditches due to the suburban setting of Greenfield Lake, located upstream of the site. Ammonia is highly toxic to aquatic organisms, including benthic invertebrates, and can lead to unexpected responses (Kohn et al., 1994). Ammonia toxicity depends partly on the pH of the test system (temperature also plays a role): toxicity increases at higher pHs where the unionized form (NH_3) predominates over the ionized form (NH^+_4) (Bower and Bidwell, 1978).

Identification of Receptors

No further refinement will be required. The receptors selected in the SLERA will be carried forward in the BERA. These receptors include the following:

- Benthic Invertebrates - direct contact/ingestion of sediments.
- Fish: Spot - indirect food-chain exposure.
- Avian: Great Blue Heron - indirect food-chain exposure.

Assessment and Measurement Endpoints

According to USEPA (1998b), assessment endpoints are explicit statements of the characteristics of the ecological system that are to be protected. Assessment endpoints either are measured directly or are evaluated through indirect measures. Measurement endpoints represent quantifiable ecological characteristics that can be measured, interpreted, and related to the valued ecological component(s) chosen as the assessment endpoints. Assessment endpoints, and the associated measurement endpoints, provide information to support or refute the risk hypotheses generated from the conceptual assessment model.

The assessment and measurement endpoints will be used to interpret data concerning ecological risks within the study area and include the following:

Assessment Endpoint No. 1 - Adverse effects on benthic macroinvertebrates as a potential prey base to higher tropic levels resulting from exposure to PAHs in sediments.

Corresponding Measurement Endpoint:

- Comparison of the distribution of chemical concentrations in sediment with the distribution of concentrations associated (in field and laboratory studies) with adverse effects on macroinvertebrates. The proportion of the chemical concentration distribution that exceeds the toxicity distribution is the measure of potential impact on the community.
- Reductions in survival and growth of *Hyalella azteca* and *Leptocheirus plumulosus* exposed to Study Area sediments. Sediments producing responses that are statistically

significant relative to responses in control sediments are assumed to be toxic.

- Reductions in survival and growth, and anomalies in head capsule width of the midge *Chironomous tentans* exposed to Study Area sediments. Sediments producing responses that are statistically significant relative to responses in control sediments are assumed to be toxic.

- Comparison of the distribution of chemical concentrations in sediment with thresholds derived from site-specific toxicity test data. Thresholds are measured concentrations above which toxicity is observed. The proportion of the chemical concentration distribution that exceeds the threshold is a measure of potential impact on the community.

Assessment Endpoint No. 2. - Adverse effects on invertivorous bird populations resulting from exposures to chemicals in sediments and/or prey.

Corresponding Measurement Endpoint:

- Comparison of predicted average daily doses of chemicals to avian receptor to toxicity reference values for the species. If the average daily dose is greater than the toxicity reference value, this indicates the potential for adverse effects to some portion of the population.

Ecotoxicological Testing

All ecotoxicity tests will closely follow standard test protocols published by EPA and/or ASTM in terms of test organism husbandry, experimental design considerations, and data interpretation. The ecotoxicity testing laboratory will maintain up-to-date control charts based on periodic reference toxicant testing to ensure that the sensitivity of the test organisms fall within expected ranges and do not deviate significantly over time.

Up to three test species will be used for sediment toxicity tests (Table 4), after the surficial sediments have been sampled and characterized as described earlier. Based upon the results of the chemical analyses, a "range finder" evaluation may be performed to determine the potential acute toxicity of the sediments. The sediment composite samples will be screened using a standard dilution series (i.e., 100%, 50%, 25%, 12%, 5%, 1%, plus control).

Based upon the results of the acute toxicity test, the chronic toxicity tests will be performed at an appropriate dilution. *Hyalella* and a *Chironomus* species will be the test organisms used in the freshwater drainage ditch/Greenfield Creek system.

To properly interpret ecotoxicity test data, test species sensitivity will also be evaluated using control and reference sediments.

- *Control Sediments* - Control sediments are used to evaluate the health and condition of test organisms to the laboratory environment, and are tested concurrently with the site samples. If survival falls below a minimum threshold (e.g., 80%) at the end of a test (consistent with ASTM protocols), then the entire data set from that test is considered invalid.

- *Reference Sediments* - Reference sediments are collected in the field and are comparable to the test sediments in physical and chemical characteristics, but without site-specific contaminants. They assess the presence of "background" toxicity in sediments due to unidentified off-site sources of contamination or the presence of naturally occurring toxicants such as ammonia. These off-site sources may include petroleum hydrocarbons from road runoff, pesticides from agricultural or lawn care activities, or heavy metals from atmospheric depositions.

COPC Fate and Transport

The SLERA included discussion of the potential fate of PAHs due to biodegradation in the environment. This discussion will be expanded for this phase of the project to include other elements relevant to ecological risk assessment.

Input Assumptions for Risk Calculations

ERA Steps 6 and 7 allow the use of more representative risk assumptions (e.g., area use factors) for the estimation of site ecological risks. Some of the key elements for each of the receptors include the following:

- Benthic organisms: Risks will be evaluated by using sediment toxicity testing. These results will also be used in conjunction with the expanded analytical database to fill data gaps in the ecological risk assessment to this receptor group in the drainage ditch-creek system.

- Fish: Empirical data on the body burdens of the COPCs will be collected as part of the proposed field program. These data will be available only for small fish (due to fillet-specific sampling being conducted for game fish). Results will be evaluated in conjunction with studies that evaluate toxic effects and body burdens.

- Bird: Exposure assumptions used in the SLERA will be refined to reflect site- or region-specific conditions. In addition, empirical fish COPC concentrations will be used to better define the risks associated with dietary exposure to COPCs.

Toxicity Evaluation

Further assessment of critical body burden values in fish and the avian TRVs used in the SLERA will be performed as part of the toxicity evaluation. Relevant enhancements of the toxicological database of the COPCs for the key receptors will be evaluated. Both freshwater and saltwater

benchmarks will be reviewed, depending upon whether the samples have been collected in the freshwater drainage ditch-Greenfield Creek system or in the more saline Cape Fear River.

Reporting and Analysis

The BERA report will be prepared in accordance with the relevant components of Steps 6 and 7 of the ERA Guidance, and will consist of the following components:

- Compilation and summary of analytical and toxicity laboratory results;
- Complete copy of field collection logs and chain-of-custody forms;
- Analysis of spatial trends (e.g., drainage ditch versus Greenfield Creek, upstream versus downstream sections of Greenfield Creek).
- Data on sediment PAH and TCDD/TCDF concentrations at the locations where fish were captured will be qualitatively inspected for correlation with fish body burdens. Spatial trends in PAH and TCDD/TCDF sediment concentrations will also be evaluated.
- Risk characterization.

3.3 Section VI. D., 15 - Proposed Methods, Locations, Depths, and Justification for all Proposed Samples

The Division (NCDENR) or its representatives may take split or duplicate samples pursuant to this workplan and the AOC. The Division will be notified not less than 10 days in advance of any sampling activities.

The proposed sample codes, locations, depths, methodology and justification for all proposed sampling points are listed on Table 1. Sample locations are also illustrated on Figure 1.

3.3.1 Sediment Sampling Methodology

Sediment samples will be collected using a stainless steel spoon and a 2-quart or equivalent glass or stainless steel bowl. Some sediment samples may be collected using stainless steel open or closed bucket hand augers, and/or Ponar dredge samplers. Samples being collected for volatile organic compounds (VOC) will be collected first and placed directly into the appropriate containers. The remainder of the sample will be placed into the bowl, mixed thoroughly, then distributed to the appropriate containers. The sediment samples will be collected from a depth of surface to 3-inches.

3.3.2 Fish Tissue Sampling Methodology

Fish tissue samples will be collected from Greenfield Creek by use of a small boat. Up to four samples will be collected to determine the human health exposure from the site. The preferred fish collection methodology will be by electro-shock with the assistance of a local pond consultant. Otherwise fish collection will be by rod and reel or seine. Upon collection of the desired fish species, the specimen will be rinsed with surface water from the collection point, identified, weighed, measured, catalogued, and visually inspected for any lesions or other physical abnormalities. After recording this information on field data sheets, the fish species will be filleted and collection will continue until the sample weight is obtained. The recommended weight for one sample is 100 grams, which may require the collection of more than one individual per sample. One sample will be identified when the weight of the combined target species is achieved. The combined fish sample will then be prepared for shipment to the analytical laboratory. The fish samples will be wrapped in aluminum foil, placed in a waterproof freezer bag, labeled, and placed immediately on either dry or wet ice. The fish sample will be frozen at the end of the sampling day and shipped to the laboratory following laboratory protocols. A chain-of-custody record will be completed by the sampler and included in the shipment of the samples to the laboratory.

Small fish whole body composites will not be filleted. These samples will be collected to provide empirical data for the quantification of risks to the piscivorous avian receptor.

3.4 Section VI. D., 16 - Proposed Field and Laboratory Procedures for Quality Assurance/Quality Control

3.4.1 Field Quality Assurance/Quality Control Procedures

All sample collection, sample preservation, and chain-of-custody procedures used during this investigation will be in accordance with approved health and safety plan and the current U.S. Environmental Protection Agency (EPA) Region IV, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), May 1996. In particular, the following sections of the EISOPQAM will be followed during completion of the activities in this workplan:

- Section 3 Sample Control, Field Records, and Document Control (Attachment B)
- Section 11 Sediment Sampling (Attachment C)

Field quality assurance/quality control (QA/QC) will include:

- One duplicate sample per medium per container type per field day.
- An equipment rinsate blank for each set of equipment that has been decontaminated per sample set.

- A VOA trip blank for each sampling group.

Sediment samples for volatile analysis will be collected directly into sample containers without mixing.

All sediment sample locations will be staked or flagged until surveyed.

3.4.2 Laboratory Quality Assurance/Quality Control Procedures

The laboratory reports will include at a minimum the items listed below:

A statement certifying that the laboratory is either certified for applicable parameters under 15A NCAC Subchapter 2H .0800, or that it is a contract laboratory under EPA's Contract Laboratory Program.

A signed statement that the samples were received in good condition and at the required temperature and that analysis of the samples complied with all procedures outlined in USEPA methodology, unless otherwise specified. Any deviations from the methods, additional sample preparation, sample dilution and analytical problems not rectified, will be justified in a narrative with the laboratory report.

Laboratory sheets for all analytical results, including sample identification, sampling dates, date samples were received, extraction dates, analysis dates, analytical methods used, dilution factors and sample quantitation limits.

Laboratory sheets for all laboratory quality control samples, including results for bias and precision and control limits used. The following minimum laboratory quality control sample reporting is required: (a) at least one matrix spike and one matrix spike duplicate per sample delivery group or 14-day period, whichever is more frequent (control limits must be specified); (b) at least one method blank per sample delivery group or 12-hour period, whichever is less; and (c) system monitoring compounds, surrogate recovery required by the method and laboratory control sample analysis (acceptance criteria must be specified). All samples that exceed control limits/acceptance criteria must be flagged in the laboratory report.

Completed chain-of-custody with associated air bill (if applicable) attached.

The laboratory report will include the names and qualifications of the individuals performing each analysis, the quality assurance officer reviewing the data, and the laboratory manager.

Review toxicity testing methods to determine the potential need to modify the methods due to known site contaminant concentrations and analyte "suite."

Verify that any analytical methods that will be performed will be capable of achieving detection limits specified in the Work Plan.

3.5 Section VI. D., 17 - Proposed Analytical Parameters and Analytical Methods for all Samples

The sampling investigation will include the collection of sediment and biological tissue samples. All sediment samples will be analyzed for previously detected extractable and purgeable organic compounds, and chromium, copper, and arsenic. Dioxin and furan analysis will occur only on select sediment samples as previously described. Sediment samples will not be analyzed for pesticides, PCBs, and cyanide. Fish tissue samples will be analyzed for previously detected extractable organic compounds and dioxins/furans if detected in proposed sediment samples.

Please refer to Table 5 for the sediment analyses and Table 6 for the fish tissue analyses.

Sediment analytical methods include:

Purgeable (volatile) compounds	Method 8260B
Extractable (semi-volatile) compounds	Method 8270C
Metals (CCA)	SW-846 Methods
Dioxins/Furans	Method 8290
Ammonia	Method 350.1
Particle Size	ASTM D422
Salinity	ASTM D4542
pH	ASTM D4972
Total Organic Carbon	Method 9060

Fish tissue analytical method includes:

Extractable (semi-volatile) compounds	Method 8270C
Dioxins/Furans	Method 8290
Lipid Content	Method OB\1090

Sample containers, holding times, and preservation will be as recommended in EISOPQAM Appendix A. The following is a description of the analyses and types of containers required:

Analyses	Containers	Preservative	Holding Time (days)
Purgeable Organics, Sediment	2 oz. glass jar[1]	Ice (4°C)	14
Extractable Organics, Sediment	8 oz glass jar[2]	Ice (4°C)	54[3]

Metals, Sediment	8 oz glass jar[2]	Ice (4°C)	360[4]
Dioxins/Furans, Sediment	500 ml amber glass	Ice (4°C)	75[5]
Fish Tissue	Aluminum foil	Ice (4°C)	2[6]

[1]Teflon Septum Lid
[2]Teflon Lid
[3]54 days: 14 days to extraction, 40 days to analysis
[4]360 days: 180 days to extraction, 180 days to analysis
[5]75 days: 30 days to extraction, 45 days to analysis
[6]If holding time will exceed 2 days then freeze sample

3.6 Section VI. D., 18 - Contact Name, Address, Telephone Number and Qualifications for Principal Consultant and Laboratory

Principal Consultant:

Schnabel Engineering Associates, Inc.
Contact - Gregory B. Kuntz, P.G., Project Manager
104 Corporate Blvd., Suite 420
West Columbia, SC 29169
Telephone: 803-796-6240
Fax: 803-796-6250

Schnabel Engineering Associates, Inc. is a North Carolina Corporation, a Registered Engineering and Geology Firm (F-0678) with the North Carolina State Board of Registration for Professional Engineers and Land Surveyors, and a Registered Environmental Consultant (#00041) under the North Carolina Inactive Hazardous Waste Site Program.

Gregory B. Kuntz is a North Carolina Registered Geologist (#1203) and a Registered Site Manager under the North Carolina Inactive Hazardous Waste Site Program.

Rich Wargo is a North Carolina Registered Engineer (#23435) and the office manager.

Principal Laboratories:

Savannah Laboratories and Environmental Services, Inc.
Contact - James W. Andrews, Ph. D., Project Manager
5102 LaRoche Avenue
Savannah, GA 31404

Telephone: 912-354-7858
Fax: 912-352-0165

Savannah Laboratories and Environmental Services, Inc. in accordance with the provisions of N.C.G.S. 143-215.3 (a) (1), 143-215.3 (a) (10), and NCAC 2H.0800 is certified to perform environmental analysis and report monitoring data to the Division of Water Quality, North Carolina Department of Environment and Natural Resources.

Savannah Laboratories and Environmental Services, Inc. will perform extractable and purgeable organics, metals and fish tissue analysis. A quality assurance project plan was previously submitted in the June 24, 1999 Draft RI.

Triangle Laboratories, Inc.
Contact - Rose West
801 Capitola Drive
Durham, NC 27713
Telephone: 919-544-5729
Fax: 919-544-5491

Triangle Laboratories, Inc. in accordance with the provisions of N.C.G.S. 143-215.3 (a) (1), 143-215.3 (a) (10), and NCAC 2H.0800 is certified to perform environmental analysis and report monitoring data to the Division of Water Quality, North Carolina Department of Environment and Natural Resources.

Triangle Laboratories, Inc. will perform high-resolution dioxin/furan analysis. Triangle laboratories quality assurance manual is presented in Attachment (D).

3.7 Section VI. D., 19 - Equipment and Personnel Decontamination Procedures

3.7.1 Equipment Decontamination Procedures

Decontamination procedures will be implemented to avoid cross-contamination of samples. Sampling equipment will be thoroughly cleaned and decontaminated before initial use and between sample locations.

A designated cleaning/decontamination station will be established prior to beginning remedial assessment activities. This decontamination area will be located downgradient and down wind from the clean equipment drying and storage area. The decontamination area will consist of a polyethylene lined waste pit to contain the rinse water and waste materials until they can be collected. At the completion of sampling activities all waste materials and polyethylene will be removed from the decontamination pit and disposed in appropriate investigation derived waste (IDW) containers.

All sampling equipment involved in the assessment activities will be cleaned and decontaminated before entering designated activity areas, between samples and prior to leaving the site using the following procedure:

1) Wash equipment thoroughly with laboratory grade, phosphate-free detergent and potable water using a brush to remove any particulate matter or surface film.
2) Rinse thoroughly with potable water.
3) Rinse thoroughly with deionized water.
4) Rinse twice with pesticide-grade isopropanol.
5) Rinse with organic-free water and allow to air dry.
6) Wrap with aluminum foil to prevent contamination, if storing or transporting the equipment prior to use.

Organic-free water should contain no pesticides, herbicides, extractable organic compounds, and less than 50 ug/l of purgeable organic compounds. In addition, no metals or other organic compounds should be detected at routine detection limits. Deionized water, organic-free water, and isopropanol must be applied using non-interfering containers made of glass, Teflon, or stainless steel.

Following completion of decontamination, if decontaminated equipment touches the ground, it will be considered contaminated and require decontamination before use.

3.7.2 Personnel Decontamination Procedures

Pre-work and weekly health and safety tailgate meetings will be conducted by the Health and Safety Officer assigned for each phase of the investigations. Personnel will be instructed on the use of personnel protective equipment (PE). Level D protection will be used in all investigations at the site unless conditions warrant an upgrade in personal protection. Personnel will be instructed to wear rubber boots, Tyvek suits, and gloves appropriate for the tasks being completed. Instruction will be given on how to provide protection against dermal, inhalation, and ingestion of potential contaminated materials. No smoking, eating or drinking will be allowed when a potential for exposure is present. At task completion, breaks, or at the end of each day, or between individual samples, as appropriate, the PPE will be removed and placed in appropriate IDW containers. Decontamination will consist of a boot, hand, and face wash using detergent and potable water. A decontamination station including non-phosphate detergent, potable water, eye wash, fire extinguisher, first aid kit, emergency first aid guide book, and the task-specific health and safety plan will be established adjacent to the decontamination area. Mobile phones will be maintained in most of the work trucks.

The Health and Safety Officer will be responsible for upgrading the level of protection required based on field observations and measurements. If an increase in PE is required, a decontamination program will be established that includes the necessary stations, barrier tapes, and decontamination and observation personnel.

3.8 Section VI. D., 20 - Health and Safety Plan

A health and safety plan that conforms to OSHA 1910.120 requirements and assures that the health and safety of nearby residential and business communities will not be adversely affected by activities related to remedial investigation activities was previously submitted in Attachment K of the June 24, 1999 Draft RI.

3.9 Section VI. D., 21 - Proposed Schedule for Site Activities and Reporting

The proposed schedule for the Supplemental RI is as follows:

⇒ Submit proposed Supplemental RI workplan	October 8, 1999
⇒ NCDENR review of workplan	November 8, 1999
⇒ Correct deficiencies in workplan	December 8, 1999
⇒ Begin Supplemental RI workplan fieldwork	January 7, 1999
⇒ Submit Supplemental Workplan report	July 7, 1999

The Supplemental Remedial Investigation will begin no sooner than receiving written approval of the Investigation Plan from the Division, nor later than thirty (30) days thereafter.

The AOC requires that the Supplemental Remedial Investigation be submitted within 120 days of notice to proceed. An extension is requested for a total of 180 days from notice to proceed. This extension request is based on the time required to complete two phases of sediment sampling (60 days), ecotoxicity testing by the laboratory (90 days) and reporting (30 days).

3.10 Section VI. D., 22 - Additional Information Considered Relevant

3.10.1 Additional Technical Comments Specific to Draft RI Report

The following additional comments will be addressed in an addendum to the Draft RI and presented in the SRI:

Section 31 Pp. 49-51

The summary table of detected constituents will be updated to include the absence of sample analytical data for any medium.

Section 32.3 P. 54, Parag. 3

See Section 3.10.4 below on Land Use restrictions

Section 32.4 P.56 Parag. 1

An addendum to the Draft RI will be prepared indicating As < 50 ug/l, Cr < 50 ug/l, and Cu > 3 ug/l (but also detected in background sample).

Section 32.5 Pp. 57-58

Please see Section 3.10.4 below.

3.10.2 Investigation Derived Waste Management

Investigation derived waste (IDW) materials will be generated during the proposed field activities that must be properly managed. Potentially contaminated materials generated will include sediments, decontamination solution, and disposable equipment. Since some of these materials may be hazardous wastes, they will be handled, stored, treated, and disposed of properly.

Because only a small volume of waste will be generated during the field investigation all waste will be combined and placed in a DOT-approved 55-gallon drum and staged on site. It is anticipated that only one drum will be required to contain the waste from this investigation. A hazardous waste label will be placed on the drum on the first day in which any waste is added to the container. An in-house waste generation form will be completed at this time and updated as necessary. Weekly inspections of the container(s) will occur until the waste is picked up for disposal. The waste type will be identified and the containers labeled with the proper D.O.T. placards, manifested, and shipped off-site to an approved waste disposal facility.

All non-hazardous IDW will be disposed at the local sanitary landfill at the end of the remedial investigation activities.

3.10.3 DNAPL Recovery

Initial efforts to evaluate the technological feasibility of partial DNAPL recovery will be evaluated during the remedial investigation. Product accumulation has occurred in the vicinity of the covered ditch (MW-14 @ 1.91' and MW-26 @ 4.1') and beneath the wood treatment facility at MW-11B (0.23') in the intermediate aquifer.

Interim actions are proposed to evaluate recovery of DNAPL from existing well MW-26. Product recovery will occur utilizing a pump or a belt lift system and a product recovery tank. The product recovery system and product disposal will comply with applicable regulations.

3.10.4 Land Use Restrictions

Initial efforts towards submitting a Declaration of Perpetual Land Use Restrictions for both parcels of land at the site will occur. The land use restrictions will be accordance with the

August 1999 Guidelines for Assessment and Cleanup. The land use restrictions are recommended so that alternate site-specific remediation goals may be obtained. This submittal will occur with the Remedial Action Plan.

3.11 Section VI. D., 23 - Signature and Seal of Licensed Professional

In Southern Wood Piedmont Company's and Schnabel Engineering Associates, Inc. opinion and to the best of our knowledge and belief all comments and requirements as listed in the September 3, 1999 NCDENR Draft RI Comment Letter and the AOC are addressed in this workplan.

This workplan has been prepared solely and exclusively for Southern Wood Piedmont Company's and NCDENR's benefit and use for specific application to this project. The use of this report by a third party or parties will be at such party's sole risk and Schnabel Engineering Associates disclaims liability for any such use or reliance by third parties.

William Arrants
Southern Wood Piedmont Company

Gregory B. Kuntz, P.G., NC No. 1203
Schnabel Engineering Associates, Inc.

4.0 SUPPLEMENTAL REMEDIAL INVESTIGATION WORKPLAN DEFICIENCIES

Within 30 days of receiving notice from the Division (NCDENR) of any deficiency in the Supplemental Remedial Investigation Workplan, information or materials sufficient to correct such deficiency will be submitted.

5.0 SUPPLEMENTAL REMEDIAL INVESTIGATION REPORT

Four copies of the Supplemental Remedial Investigation Report will be submitted within 180 days of receiving written approval from the Division. The report will be organized in sections corresponding to Section VI. G. of the AOC.

6.0 REFERENCES

ASTM. 1997a. Annual Book of ASTM Standards, volume 11.05. E 1367-92: Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods.

ASTM. 1997b. Annual Book of ASTM Standards, volume 11.05. E 1525-94a: Standard Guide for Designing Biological Tests with Sediments.

ASTM. 1997c. Annual Book of ASTM Standards, volume 11.05. E 1706-95b: Standard Test Methods for Measuring the Toxicity of Sediment-Associated Contaminants with Fresh Water Invertebrates.

Black and Veatch. 1997. Expanded Site Inspection of Southern Wood Piedmont. Wilmington, New Hanover County, North Carolina. July

Bower, C.E., and J.P. Bidwell. 1978. Ionization of ammonia in seawater: effects of temperature, pH, and salinity. *Journal of the Fisheries Research Board of Canada*. 35:1012-1016.

Burton, A.G. 1991. Assessing the toxicity of freshwater sediments. *Environmental Toxicology and Chemisty*. 10: 1585-1627.

ChemRisk. 1996. Human Health and Ecological Risk Assessment for the Southern Wood Piedmont Site, Wilmington, North Carolina. Prepared for Southern Wood Piedmont. 29 May 1996.

Di Toro, D.M., C.S. Zarba, D.J. Hansen, W.J. Berry, R.C. Shwartz, C.E. Cowan, S.P. Pavlou, H.E. Allen, N.A. Thomas, and P.R. Paquin. 1991. Technical basis for establishing sediment quality criteria for nonionic organic chemicals using equilibrium partitioning. *Environmetal Toxicology and Chemistry*. 10:1541-1583.

EPA. 1993. Wildlife Exposure Factors Handbook. Office of Research and Development. EPA/600-R-93/187a,b.

EPA. 1994. EPA Environmental Response Team Standard Operating Procedure No. 2016: Sediment Sampling.

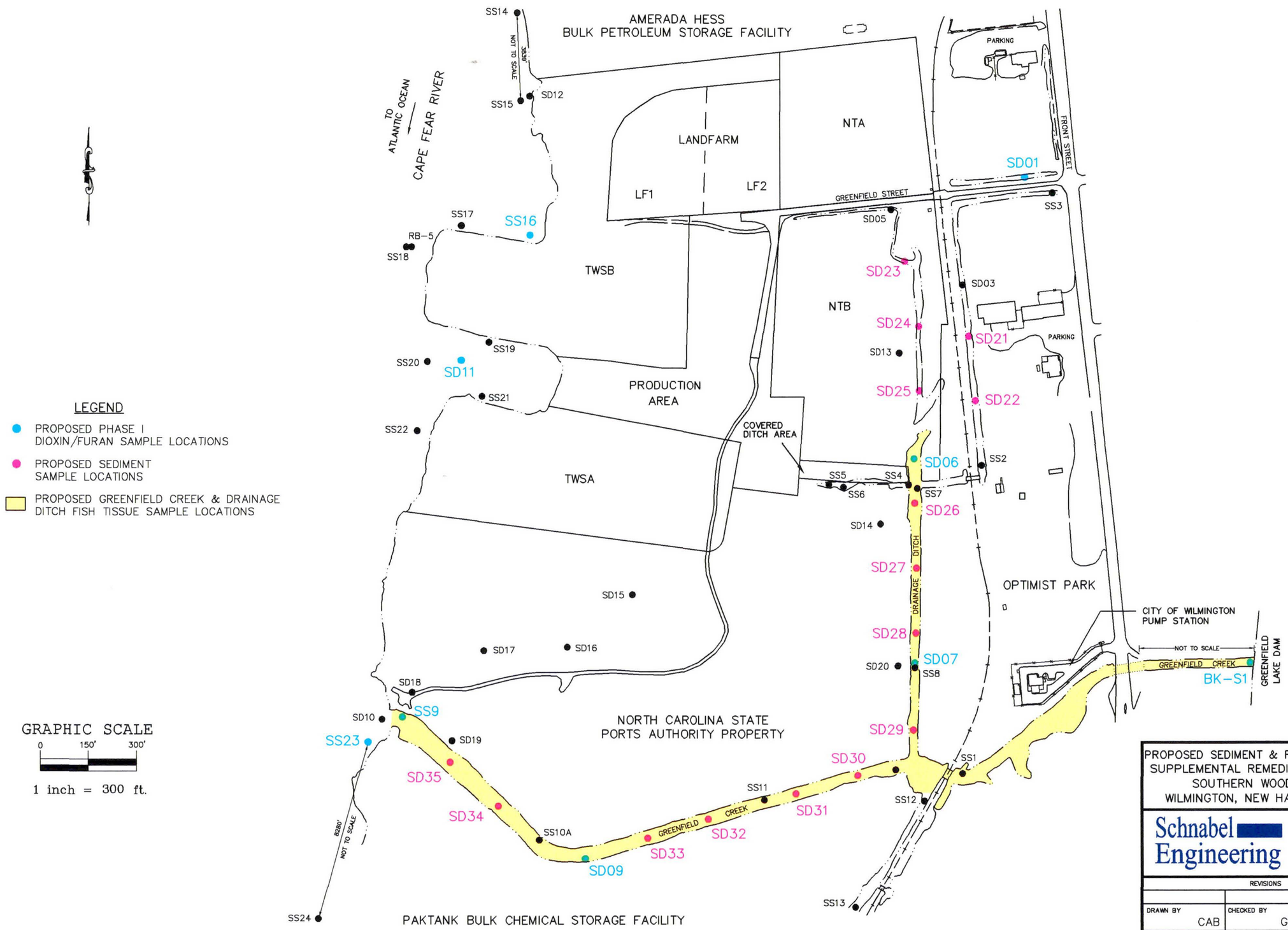
EPA. 1997. Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments. Office of Solid Waste and Emergency Response. EPA/540-R-97/006. Interim Final. June.

EPA. 1998a. Evaluation of Dredged Material Proposed for Discharge inWaters of the U.S. - Testing Manual.. US Army Corps of Engineers, USEPA. EPA/823-B-98/004. February 1998.

EPA. 1998b. Guidelines for Ecological Risk Assessment. 63FR26845. May.

Kohn, N.P., J.Q. Word, D.K. Niyogi, L.T. Ross, T. Dillon, and D.W. Moore. 1994. Acute toxicity of ammonia to four species of marine amphipod. *Marine Environmental Research.* 38:1-15.

NCDENR 1999. Review and Comment on Submittal: Schnabel Engineering Report on Remedial Investigation, Southern Wood Piedmont – Wilmington Site. 3 September 1999.



LEGEND

- PROPOSED PHASE I DIOXIN/FURAN SAMPLE LOCATIONS
- PROPOSED SEDIMENT SAMPLE LOCATIONS
- PROPOSED GREENFIELD CREEK & DRAINAGE DITCH FISH TISSUE SAMPLE LOCATIONS

GRAPHIC SCALE

0 150' 300'

1 inch = 300 ft.

PROPOSED SEDIMENT & FISH TISSUE SAMPLE LOCATIONS
SUPPLEMENTAL REMEDIAL INVESTIGATION WORKPLAN
SOUTHERN WOOD PIEDMONT COMPANY
WILMINGTON, NEW HANOVER CO., N.C. FACILITY

Schnabel Engineering
Schnabel Engineering Associates, Inc.
104 Corporate Blvd., Suite 410
West Columbia, South Carolina 29169
Phone: 803-796-6240
Fax: 803-796-6250

REVISIONS			CADD NO.
			SEDIMENTFG1
DRAWN BY CAB	CHECKED BY GBK	SCALE 1"=300'	FIGURE NO. 1
DESIGN BY GBK	DATE 10/08/99	PROJECT NO. 979007.A	SHEET NO.

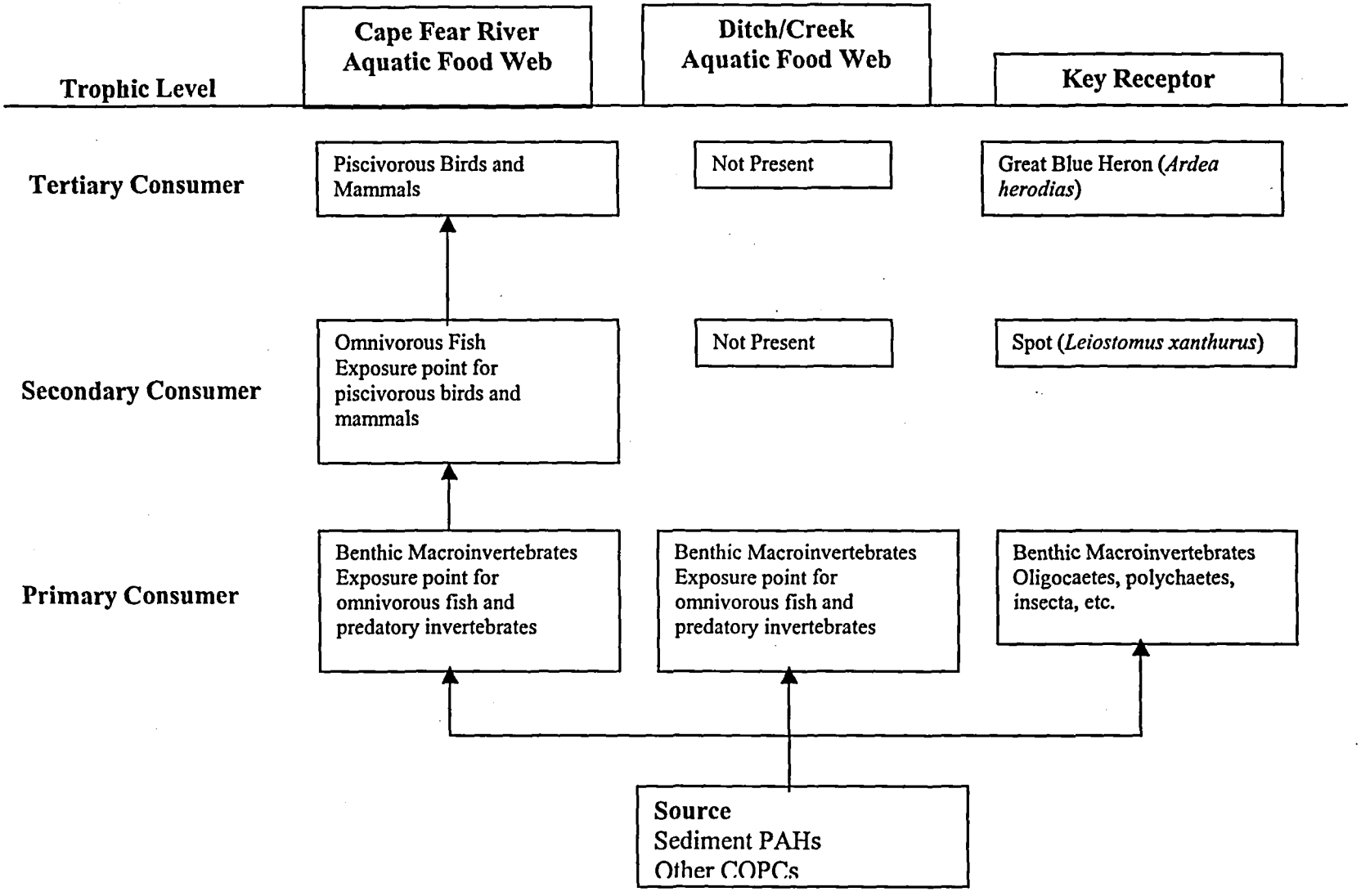


Figure 2. Site Conceptual Model for Ecological Risk Assessment at the SWP-Wilmington Project

TABLES

TABLE 1

TABLE 1 - SAMPLE IDENTIFICATION, LOCATIONS, DEPTHS, METHODOLOGY, AND JUSTIFICATION
SOUTHERN WOOD PIEDMONT AND NORTH CAROLINA STATE PORTS AUTHORITY
WILMINGTON, NEW HANOVER COUNTY, NORTH CAROLINA

Sample Code	Sample Media	Sample Location	Depth	Sample Method	Justification
SD-01DF	Sediment	Ditch east of the site near the entrance gates	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To establish background levels for Dioxins/Furans
SD-01DF-Dup	Sediment	Ditch east of the site near the entrance gates	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To establish background levels for Dioxins/Furans
SD-06DF	Sediment	Ditch southeast of Non-Treated Wood Storage Area B (NTB)	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of Dioxins/Furans
SD-07DF	Sediment	Ditch west of Optimist Park	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of Dioxins/Furans
BK-S1DF	Sediment	Greenfield Creek below dam at Greenfield Lake	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To establish background levels for Dioxins/Furans
SD-09DF	Sediment	Greenfield Creek halfway ditch and tidal gate	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of Dioxins/Furans
SS-9DF	Sediment	Greenfield Creek at tidal gate	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of Dioxins/Furans
SS-16DF	Sediment	North Slip in Cape Fear River	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To establish background levels for Dioxins/Furans
SD-11DF	Sediment	South Slip in Cape Fear River	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of Dioxins/Furans
SS-23DF	Sediment	Cape Fear River downstream of Greenfield Creek tidal gate	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of Dioxins/Furans
SD-21	Sediment	East of site in offsite ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-22	Sediment	East of site in offsite ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-23	Sediment	Ditch east of Non-Treated Wood Storage Area B (NTB)	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-24	Sediment	Ditch east of Non-Treated Wood Storage Area B (NTB)	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-25	Sediment	Ditch east of Non-Treated Wood Storage Area B (NTB)	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-26	Sediment	Ditch West of Optimist Park	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-27	Sediment	Ditch West of Optimist Park	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-28	Sediment	Ditch West of Optimist Park	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-29	Sediment	Ditch West of Optimist Park	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-30	Sediment	Greenfield Creek downstream of confluence with ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-30-Dup	Sediment	Greenfield Creek downstream of confluence with ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-31	Sediment	Greenfield Creek downstream of confluence with ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-32	Sediment	Greenfield Creek downstream of confluence with ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-33	Sediment	Greenfield Creek downstream of confluence with ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-34	Sediment	Greenfield Creek near outfall to Cape Fear River	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination
SD-35	Sediment	Greenfield Creek near outfall to Cape Fear River	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	To determine presence or absence of contamination

TABLE 1 - SAMPLE IDENTIFICATION, LOCATIONS, DEPTHS, METHODOLOGY, AND JUSTIFICATION
SOUTHERN WOOD PIEDMONT AND NORTH CAROLINA STATE PORTS AUTHORITY
WILMINGTON, NEW HANOVER COUNTY, NORTH CAROLINA

Sample Code	Sample Media	Sample Location	Depth	Sample Method	Justification
SD-36-Comp	Sediment	Drainage Ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	Sediment toxicity testing
SD-37-Comp	Sediment	Drainage Ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	Sediment toxicity testing
SD-38- Comp	Sediment	Greenfield Creek between drainage ditch and tidal gate	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	Sediment toxicity testing
SD-39-Comp	Sediment	Greenfield Creek between drainage ditch and tidal gate	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	Sediment toxicity testing
SD-40-Comp	Sediment	Greenfield Creek between drainage ditch and tidal gate	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	Sediment toxicity testing
SD-41-Comp	Sediment	Greenfield Creek upstream of drainage ditch	Surface	Stainless Steel Spoon Hand Auger or Ponar Dredge	Sediment toxicity testing
BIO-10	Biological, Fish Tissue	In Greenfield Creek downstream of confluence with ditch		Electro-shock	To determine presence or absence of contamination
BIO-10-Dup	Biological, Fish Tissue	In Greenfield Creek downstream of confluence with ditch		Electro-shock	To determine presence or absence of contamination
BIO-11	Biological, Fish Tissue	In Greenfield Creek downstream of confluence with ditch		Electro-shock	To determine presence or absence of contamination
BIO-12	Biological, Fish Tissue	In Greenfield Creek downstream of confluence with ditch		Electro-shock	To determine presence or absence of contamination
BIO-14	Biological, Fish Tissue	In Greenfield Creek downstream of confluence with ditch		Electro-shock	To determine presence or absence of contamination
BIO -15	Biological, Fish Tissue	In drainage ditch		Electro-shock	To determine presence or absence of contamination
BIO-16	Biological, Fish Tissue	In Greenfield Creek upstream of drainage ditch		Electro-shock	To determine reference concentration
BIO-17-Comp	Biological Whole Fish	Greenfield Creek downstream of drainage ditch		Electro-shock	To determine presence or absence of contamination
BIO-18-Comp	Biological Whole Fish	Greenfield Creek downstream of drainage ditch		Electro-shock	To determine presence or absence of contamination
BIO-19-Comp	Biological Whole Fish	Greenfield Creek downstream of drainage ditch		Electro-shock	To determine presence or absence of contamination
BIO-20-Comp	Biological Whole Fish	Drainage ditch		Electro-shock	To determine presence or absence of contamination
BIO-21-Comp	Biological Whole Fish	Greenfield Creek upstream of drainage ditch		Electro-shock	To determine reference concentration

TABLE 2

Table 2. Summary of Screening-Level Ecological Risk Assessment (SLERA)[1,2]

ERA Steps	Status	Outcome
Step 1: Screening-Level		
Site Visit	Completed	Site visits were completed in 1995 and 1996. Habitat structure and ecological community characterizations were performed for the drainage ditch, Greenfield Creek, and adjacent areas of Cape Fear River (including Eagle Island). A qualitative benthic invertebrate assessment was also performed.
Problem Formulation	Completed	Problem formulation performed and site conceptual model developed for drainage ditch, Greenfield Creek, and adjacent areas of Cape Fear River.
Analytical Database	Ongoing	SLERA prepared based upon analytical data collected from 1985 through 1995[4]. These included surface water, sediment, and AVS/SEM data.
Identification of Potential Receptors	Completed	Evaluated (1) aquatic plants, (2) benthic invertebrates, (3) fish, (4) amphibians and reptiles, (5) piscivorous wildlife (birds and mammals), and (6) threatened, endangered and rare species, as part of the SLERA. Key receptors that were evaluated quantitatively were the following: • Benthic Invertebrates – direct contact/ingestion of sediments • Fish: Spot – indirect food-chain exposure • Avian: Great Blue Heron – indirect food-chain exposure
Toxicity Evaluation	Completed	Toxicity profiles were provided for COPCs. <u>Metals</u>: ER-L values were used. <u>PAHs</u>: For the fish, the critical body burden for PAHs was used. For the avian receptor, a TRV derived from the literature was used.
Step 2: Screening Level		
Exposure Estimate	Completed	<u>Metals</u>: Screen was performed using conservative ER-L values. The bioavailability of metals was evaluated using AVS/SEM data. Metals were not found to be labile in drainage ditch, Greenfield

Table 2. Summary of Screening-Level Ecological Risk Assessment (SLERA)[1,2]

ERA Steps	Status	Outcome
		Creek, and adjacent areas of Cape Fear River. PAHs: Comparison made to site-specific sediment quality criteria based on Equilibrium Partitioning Theory. Most observed PAH concentrations were below these values. PAH fingerprinting indicated a potential pyrogenic source for the PAHs.
COPC Selection	On-going	Metals: Screen was performed using conservative ER-L values. Metals that were analyzed were not retained as COPCs since levels were below conservative ER-L values. PAHs: Comparison made to site-specific sediment quality criteria based on Equilibrium Partitioning Theory. PAHs were principal COPC.
Input Assumptions	Completed	Benthic: Conservatively assumed that the organisms were in direct contact with the sediments throughout their lives. Fish: Conservative biconcentration factors and no biodegradation were assumed for the fish. No empirical data were collected. Bird: Conservative assumptions for Area Use Factor, bioavailability, and dietary consumption were used. Inputs were derived from published studies and the Wildlife Exposure Factors Handbook (EPA, 1993). Exposure via diet was based upon estimated concentrations of PAHs in fish.
Risk Calculations	Completed	Potential benthic effects evaluated based upon comparison to ER-L values for COPCs Potential effects to fish receptors were evaluated using hazard quotients that were calculated using critical body burdens for the COPCs. Potential effects to avian receptors were evaluated using hazard quotient calculated using TRVs for the COPCs
Uncertainty Assessment	Completed	A non-quantitative uncertainty assessment was performed. Sources of

Table 2. Summary of Screening-Level Ecological Risk Assessment (SLERA)[1,2]

ERA Steps	Status	Outcome
		uncertainty were identified that included (1) selection of ecological COPC, (2) selection of key receptors, (3) exposure assessment, and (4) the ecological effects assessment.
Step 3: Baseline Risk Assessment Problem Formulation		
Refine Problem Formulation	Ongoing	Problem formulation as described in Step 1 still applies to this Step, except for the proposed effort related to the determination of potential sediment toxicity to benthic organisms.
Refine COPC list	Not Required	COPC list developed in Step 2 would still apply to this step and the subsequent steps.
Assessment Endpoints		Assessment Endpoints as described in Step 1 still applies to this Step
Conceptual Model Exposure Pathways	Ongoing	Conceptual models developed for drainage ditch, Greenfield Creek, and adjacent areas of Cape Fear River. Avian receptor relevant only to Cape Fear River.
COPC Fate and Transport	Ongoing	SLERA included discussion of the potential fate of PAHs due to biodegradation in the environment. This discussion will be expanded in the subsequent steps in the ERA.
Toxicity Evaluation	Ongoing	PAH toxicity in fish receptor based upon critical body burden. For the avian toxicity, a TRV based upon a single PAH toxicity study was used. Further assessment of critical body burden value in fish and the avian TRVs used in the SLERA will be performed as part of the new project. HQs indicated potential toxicity in sediments to benthic invertebrates. Sediment toxicity testing is proposed as part of ERA Step 4-8

Notes:

1. Based on report prepared by ChemRisk (1996).
2. Steps 4 through 8 of the EPA guidance (EPA, 1997) is the subject of this Work Plan.
3. Evaluation of potential risks based upon hazard quotients suggested that toxicity may be present. Sediment toxicity testing is proposed as part of Step 4 of the ERA.

TABLE 3

Table 3. Summary of Planned Ecological Risk Assessment Activities (ERA Steps 4 and 5)

ERA Steps	Outcome
Problem Formulation	Problem formulation and site conceptual model developed for drainage ditch, Greenfield Creek, and adjacent areas of Cape Fear River. Problem formulation as described in Step 1 still applies, except for the effort related to the determination of potential sediment toxicity to benthic organisms.
Analytical Database	SLERA prepared based upon analytical data collected from 1985 through 1995. Relevant data collected since that period and as part of the 1999-2000 field effort will be included in the updated ERA.
Identification of Receptors	No further refinement will be required. Receptors will include the following: • Benthic invertebrates – direct contact/ingestion of sediments • Fish: Spot – indirect food-chain exposure • Avian: Great Blue Heron – indirect food-chain exposure
Assessment and Measurement Endpoints	The assessment and measurement endpoints are updated to reflect the use of toxicity testing to evaluate the risks to benthic organisms.
Toxicity Testing	Toxicity testing of the biologically active zone of sediments will be performed with two amphipod species to determine whether the risks calculated in the SLERA represent actual effects at the site.
Develop DQOs	Review toxicity testing methods to determine the potential need to modify the methods due to known site contaminant concentrations and analyte "suite". Verify that any analytical methods that will be performed will be capable of achieving detection limits specified in the Work Plan.
COPC Fate and Transport	SLERA included discussion of the potential fate of PAHs due to biodegradation in the environment. This discussion will be expanded as part of the Work Scope for this phase of the project.
Input Assumptions for Risk Calculations	Benthic: Risks will be evaluated by using sediment toxicity testing. Fish: Empirical data will be collected as part of the proposed field program to evaluate potential body burden effects. Bird: Exposure assumptions used in the SLERA will be refined to reflect site- or region-specific conditions, as well as empirical fish concentrations.
Toxicity Evaluation	Further assessment of critical body burden value in fish and the avian TRVs used in the SLERA will be performed.

TABLE 4

Table 4. Summary of Test Organisms for Sediment Ecotoxicity Testing

Test Species	Geographic Range	Habitat	Salinity Preferences	Sediment Type	Age of Test Organisms	Test Duration	Endpoint Measured	Organism Source
Hyallela azteca (amphipod)	Lakes, ponds, streams, ditches, marshes	Free burrowing sediment dweller	0-15 ppt (can tolerate to 29 ppt)	From 100% sand to 90% silt/clay	7-14 d specimens	10 d	Mortality and growth	Lab cultures
					7-14 d specimens	28 d	Mortality and growth	Lab cultures
Leptocheirus plumulosus (amphipod)	East coast	U-shaped burrows in sediments	2-32 ppt	Fine sand to silty clay	As uniform as possible in age and size (adults)	10 d	Mortality, ability to rebuy in clean seds after exposure	Wild population; lab cultures
					Neonates (<24h)	28 d	Mortality, growth, reproduction	Lab cultures
Chironomus riparius (midge)	Eutrophic lakes and streams	Sediment tube dweller	0 - 5 ppt	Wide tolerance of grain size (>90% silt/clay to 100% sand)	First instar (<3 d old)	10-14 d	Mortality, growth	Lab cultures
						Up to 30 d	Emergence	Lab cultures
Chironomus tentans (midge)	Eutrophic lakes and streams	Sediment tube dweller	0 - 5 ppt	Wide tolerance of grain size (>90% silt/clay to 100% sand)	Second or third instar	10 d	Mortality, growth, head capsule width	Lab cultures

Note:
Compiled from Burton (1991), EPA (1998a) and ASTM (1997a-c)

TABLE 5

Table 5. Sediment Sample Locations, Estimated Number of Samples, and Analyses.

Location/ Samples	Matrix	SVOCs	PCDD/F	TOC	Physico-Chemical Parameters[1]	Benthic Invertebrate Toxicity
Cape Fear River						
- Samples	solid	0	3	3	0	0
- Field Duplicate	solid	0	0	0	0	0
- MS/MSD	solid	0	0	0	0	0
- Rinsate Blank	aqueous	0	0	0	0	0
Greenfield Creek						
- Samples	solid	6	3	9	12	3
- Field Duplicate	solid	1	0	1	1	1
- MS/MSD	solid	1	0	0	0	0
- Rinsate Blank	aqueous	1	0	0	0	0
Drainage Ditch						
- Samples	solid	9	3	12	14	2
- Field Duplicate	solid	0	1	0	0	0
- MS/MSD	solid	0	0	0	0	0
- Rinsate Blank	aqueous	0	0	0	0	0
Reference Area						
- Samples	solid	1	1	1	1	1
- Field Duplicate	solid	0	0	0	0	0

Notes:

1. Geochemical Parameters include sediment particle size distribution, salinity, pH, and ammonia concentration.

TABLE 6

Table 6. Fish Sample Locations, Estimated Number of Samples, and Analyses.

Location/ Samples	Matrix	SVOCs	PCDD/F[2]	Lipid Content
GreenField Creek				
- Fillet Samples[3]	tissue	4	4	4
- Whole Samples[3]	tissue	3	3	3
- Field Duplicate	tissue	1	1	1
- MS/MSD	tissue	1	1	0
- Rinsate Blank[4]	aqueous	1	1	0
Drainage Ditch				
- Fillet Samples	tissue	1	1	1
- Whole Samples	tissue	1	1	1
- Rinsate Blank	aqueous	0	0	0
Reference Area				
- Fillet Samples	tissue	1	1	1
- Whole Samples	tissue	1	1	1
- Field Duplicate	tissue	0	0	0

Notes:

1. Number of samples are estimated and depend upon actual abundance and sampling success.
2. Dioxins/Furans will only be analyzed if detected in proposed sediment samples.
3. Composite samples will be prepared for fillets from gamefish, and whole-body composites of small prey fish.
4. Rinsate blanks will be required only if the fillet samples are prepared in the field.

ATTACHMENT A

NORTH CAROLINA DEPARTMENT OF
ENVIRONMENT AND NATURAL RESOURCES
DIVISION OF WASTE MANAGEMENT

NCDENR

JAMES B. HUNT JR.
GOVERNOR

WAYNE MCDEVITT
SECRETARY

WILLIAM L. MEYER
DIRECTOR

September 3, 1999

Mr. William Arrants,
Manager of Environmental
Affairs / Regulatory Compliance
Southern Wood Piedmont Co.
P.O. 5447
Spartanburg, South Carolina 29304

Re: Review and Comment on Submittal:
Schnabel Engineering Report
on Remedial Investigation,
Southern Wood Piedmont - Wilmington Site
NCD 058 517 467

Dear Mr. Arrants:

Thank you for your timely submittal of the above draft RI report. I have reviewed the report for compliance with the terms of the State Deferral Administrative Order on Consent, and for factual consistency with the attached references and other sources of information. Schnabel Engineering has performed a thorough collation of the existing analytical data and site investigations completed to date. Attached are general comments on the status of remedial investigation of the site, as well as specific comments on the contents and findings of the draft RI report.

SWP is directed to submit a Proposed RI Workplan addressing additional sampling requirements within 30 days of receipt of these comments. Following our review and comment on the Proposed Workplan, SWP will have 30 days to revise the Draft RI Workplan as needed. If you have any questions or scheduling concerns, please contact me at (919) 733-2801, Ext. 277.

Sincerely,

Stuart F. Parker, Jr.
Hydrogeologist
NC Superfund Section

Attachments

cc: Mr. Gregory Kuntz, Schnabel Engineering
Pat DeRosa
File

401 OBERLIN ROAD, SUITE 150, RALEIGH, NC 27605
PHONE 919-733-4996 FAX 919-715-3605
AN EQUAL OPPORTUNITY / AFFIRMATIVE ACTION EMPLOYER - 50% RECYCLED/10% POST-CONSUMER PAPER

Review and Comment on
June 1999 Draft Remedial Investigation Report
Southern Wood Piedmont Site
Wilmington, New Hanover County, NC
NCD 058 517 467

Stuart F. Parker
NC Superfund Section
August 1999

PART I: GENERAL COMMENTS:

1) Sampling completed at the site has identified creosote contamination in sediments along the site's drainage ditch and lower Greenfield Creek, plus localized creosote contamination at the Cape Fear waterfront. Sampling results to date do not indicate that creosote contamination has migrated from Greenfield Creek to sediments on the adjacent Cape Fear River bottom. However, creosote-contaminated sediment was evident directly upstream of the tidal gate within the mouth of the creek.

2) Arsenic concentrations in the above sediment samples exceeded the State Soil Remediation Goal, however, the concentrations were generally in the same range as background levels. Possible exceptions were drainage ditch ESI samples SD-03, SD-06 and SD-08, for which arsenic results were qualified as estimated values. The Superfund Section concurs that the arsenic concentrations in Greenfield Creek and the Cape Fear River appear to represent ambient conditions.

3) None of the sediment samples has been tested for chlorinated dibenzodioxins/dibenzofurans. These soil contaminants were introduced to the site with the use of pentachlorophenol, and may have migrated to the waterways. As part of the RI, selected sediment locations previously sampled during site assessment should be resampled specifically for these contaminants, to determine whether release has occurred to the waterway. Results will indicate whether further evaluation for dioxins/furans is needed in the waterways.

4) Creosote-contaminated sediment locations identified thus far within the drainage ditch and Greenfield Creek are separated by intervals ranging up to several hundred feet. This resolution was adequate for site assessment purposes. However, higher-resolution sediment characterization will be required to delineate "hot" segments of the ditch and creek bed during Remedial Investigation. If dioxins/furans are detected above background at the site assessment sediment sample locations, the RI will require higher-resolution sampling for these contaminants as well.

5) Access routes to lower Greenfield Creek have been posted against trespassing by the State Ports Authority, in order to discourage continued fishing there. However the State of NC requires analytical documentation of fish tissue contamination before posting a fish consumption advisory. Results of ESI fish tissue sampling were inconclusive. Therefore, fish tissue sampling will be required as part of the RI.

6) The 1996 Human Health and Ecological Risk Assessment report for Southern Wood Piedmont is based on incomplete characterization of the site (see above), and on the presumed historical non-use of Greenfield Creek as a fishery, which remains a point of controversy. Human health risk scenarios should include fish consumption from the drainage ditch/Greenfield Creek.

The ecological risk assessment pre-dates the EPA's 1997 *Ecological Risk Assessment Guidance for Superfund, Process for Designing and Conducting Ecological Risk Assessments* (EPA 540-R-97-006). RI risk assessment must be in compliance with this guidance, and incorporate the results of future RI sampling.

NC Superfund Section Industrial Hygiene Consultant David Lilley reviewed the Chemrisk risk assessment report in June 1996. His technical comments are attached.

7) US EPA Region IV and State Inactive Hazardous Sites Program (IHSP) personnel agree that ecotoxicity testing of drainage ditch and Greenfield Creek sediments is an appropriate approach to evaluating ecological risk at the site. However, they maintain that ***chronic*** exposure scenarios will be required to complete a satisfactory evaluation. RI ecotoxicity studies and risk determinations will be reviewed by the NC Superfund Section and the NC Division of Water Quality.

8) Recent groundwater data and observations indicate that additional vertical migration of creosote DNAPL may be occurring beneath the site. Although groundwater is not the medium of primary concern at this site, the technical feasibility of partial recovery of creosote DNAPL will be investigated during site remediation.

9) At present, only the deed for the northern (former City of Wilmington) site parcel contains a restriction clause limiting future site use. This clause alone does not satisfy state requirements, as outlined in the August 1999 IHSP *Guidelines for Assessment and Cleanup*, Appendix D. In the event that alternate site-specific soil cleanup goals are to be sought, based on restricted future land use at the site, a request for Declaration of Perpetual Land Use Restrictions may be submitted to DENR from State Ports Authority as part of the Remedial Action Plan.

PART II: TECHNICAL COMMENTS SPECIFIC TO DRAFT RI REPORT:

<u>Section 5.5</u>

P. 11, Parag. 4-5: Several of the slug test wells were not screened across the entire thickness of their respective aquifer(s). However, the hydraulic conductivity results are consistent with the composition of the aquifer materials.

Section 5.9

P. 14, Parag. 5: The tidal gate would not necessarily prevent sediment transport from Greenfield Creek to the Cape Fear River, especially during high creek discharge events at low river tide. Nor would the gate exclude all swimming organisms in the Cape Fear River from entering Greenfield Creek. Immature fish characteristically use tributaries to avoid predation and food competition in larger water bodies. Note that mature game fish were observed in Greenfield Creek, both during the 1997 Expanded Site Inspection and during an off-site reconnaissance by the NC Superfund Section on 4/20/99.

Section 6.2

P. 15, Parag. 6: Emergency surface-water intakes on Smith and Toomers Creeks have been unused for several decades due to salt water encroachment.

Section 7.0

P. 16: The references document those environmentally sensitive areas present within the study area, but not the specific absence of the other environmentally sensitive areas.

Section 10.0

P. 20: Greenfield Creek was channelized between 1938 and 1949. The on-site drainage ditch is not evident in the 1938 photograph, suggesting that contaminant migration to the ditch and creek occurred subsequent to that time

Section 12.0

P. 23, Item 3: Position of new ditch in relation to covered ditch is unclear from description, but appears to be to the south.

Section 13.16

Ref. 35, Parag. 5: Table 2-5 does not list State Soil Remediation Goals for each dioxin and furan species.

P. 36, Parag. 2: Possible semi-volatile contaminant sources > 0.5 mile upstream of site are not identified, nor are they specified as being on Greenfield Creek or the Cape Fear River. Cite source.

Section 13.17

P. 37, Parag. 5: The indicated changes in total wood-preserving constituent concentrations within the landfarm are not evident from examination of Tables 10-1 through 10-6. Cite samples used in the determination.

P. 37, Parag. 7;
P. 38, Parag. 2: SS-14 is invalid as a background sample due to likely PAH contamination from the Wilmington Coal Gas Plant Site, NCD 986 188 910. SS-16, SS-20, and SS-22 through SS-24 demonstrate that PAH is not ubiquitous in the river system. Contribution of site contribution to Greenfield Creek is demonstrable from sediment samples.

Section 13.18
P. 39, Bullet 1: Copper was detected in surface water, but at concentrations less than the Class SC water quality standard.

P. 39, Bullet 3: See SS-14 comment above.

P. 39, Bullet 5: Greenfield Creek Tidal Gate is not a barrier against exposure via potential sediment migration to the Cape Fear River.

P. 39, Bullet 6: See game fish comment above.

Section 31
Pp. 49-51: Summary table does not indicate the absence of sample analytical data for any medium, e. g., Dioxin in Sediment. Instances where sampling has not occurred should be indicated "NA"

Section 32.3
P. 54, Parag. 3: Deed restrictions for site use do not meet requirements outlined in IHSP Guidance, Appendix D. If alternate cleanup goals are to be sought based on restricted land use at site, request for Declaration of Perpetual Land Use Restrictions by State Ports Authority may be submitted to DENR as part of the Remedial Action Plan.

Parag. 5: Same as P. 37, Paragraph 7.

Section 32.4
P. 56, Parag. 1: As < 50 ug/l;
Cr < 50 ug/l;
Cu > 3 ug/l, but also detected in background sample.

Section 32.5
Pp. 57-58: Creosote DNAPL exists beneath both southern and northern parcels of the site. The DNAPL apparently has already fully penetrated the peat layer and has begun pooling at the base of the intermediate (sandy) aquifer. Coarseness of the sandy aquifer materials and continued product mobility indicate the potential for some product recovery in areas of significant DNAPL thickness.

June 17, 1996

TO: Jack Butler

FROM: David Lilley DBL

RE: Comments prepared on the Human Health Risk Assessment for the Southern Wood Piedmont Site, Wilmington, NC May 29, 1996

After reviewing the above mentioned document, I offer the following comments:

1. Page ES-1, second paragraph, next to the last sentence: It is stated that true risks may be zero. There is no such thing as zero risk, risk is either above or below acceptable levels.

2. Table 3-3: The unites for the Inhalation Unit Risk Value should be $(ug/m^3)^{-1}$.

3. Table 3-3: The Inhalation Slope Factor for Benzo(b)fluoranthene should be 6.10E-01, not 6.10E+00 as written.

4. Tables 3-3 and 3-4: Was EPA-ORD consulted before extrapolating from the oral SF to inhalation SF (Table 3-3) and inhalation RfD to oral RfD (Table 3-4)? Such a consultation (and the appropriate documentation) will be necessary before these extrapolations can be accepted.

5. Tables 4-1 and 4-2, Lung Deposition Fraction (LDF): It is unclear to the reader where this factor used. According to EPA, this factor is to be used when extrapolating from an oral toxicity value to an inhalation value (under the guidance of EPA-ORD). See comment #4.

6. Table 4-9: The units for the dermal permeability coefficients are cm/hr, not cm^2/hr as written.

7. Table 4-5: Benzo(k)fluoranthene is listed on Table 2-3 as a COPC for Surface soil, but there is no Exposure Point Concentration (EPC) listed in Table 4-5. Please explain this inconsistency.

8. Tables 4-4 and 4-7: There is an EPC for groundwater listed for phenanthrene in these tables, but phenanthrene is not listed as a COPC in groundwater in Table 2-6. Please explain this inconsistency.

9. Appendix B, Industrial Scenario (typical) page 11: The EPC for benzene (according to Table 4-7) is 0.006, not - as listed on this page. Please make the appropriate correction.

10. Appendix B, Industrial Scenario (high end) page 1: The EPC for benzene (according to Table 4-7) is 0.009, not - as listed on this page. Please make the appropriate correction.

11. Appendix B, Trespasser Scenario (typical), pages 8, 10, and 11: The concentration of 1.5E+00 mg/kg for benzo(k)fluoranthene does not appear on Table 4-5 (Exposure Point Concentrations). Please explain this inconstancy.

12. Appendix B, Trespasser Scenario (high end), pages 2, 4, and 5: The concentration of 1.9E+00 mg/kg for benzo(k)fluoranthene does not appear on Table 4-5 (Exposure Point Concentrations). Please explain this inconstancy.

13. It is recommended that an Exposure Point Concentration summary table be added to Chapter 4 for the landfarm area.

dl/DL/ra.com/69,70

ATTACHMENT B

SECTION 3
SAMPLE CONTROL, FIELD RECORDS, AND DOCUMENT CONTROL

SECTION OBJECTIVES:

- Present standard procedures for sample identification.
- Present standard procedures for sample control.
- Present standard procedures for chain-of-custody.
- Present standard procedures for maintenance of field records and document control.

3.1 Introduction

All sample identification, chain-of-custody records, receipt for sample forms, and field records should be recorded with waterproof, non-erasable ink. If errors are made in any of these documents, corrections should be made by crossing a single line through the error and entering the correct information. All corrections should be initialed and dated. If possible, all corrections should be made by the individual making the error.

If information is entered onto sample tags, logbooks, or sample containers using stick-on labels, the labels should not be capable of being removed without leaving obvious indications of the attempt. Labels should never be placed over previously recorded information. Corrections to information recorded on stick-on labels should be made as stated above.

Following are definitions of terms used in this section:

Project Leader: The individual with overall responsibility for conducting a specific field investigation in accordance with this SOP.

Field Sample Custodian: Individual responsible for maintaining custody of the samples and completing the sample tags, Chain-of-Custody Record, and Receipt for Sample form.

Sample Team Leader: An individual designated by the project leader to be present during and responsible for all activities related to the collection of samples by a specific sampling team.

Sampler: The individual responsible for the actual collection of a sample.

Transferee: Any individual who receives custody of samples subsequent to release by the field sample custodian.

Laboratory Sample Custodian: Individual or their designee(s) responsible for accepting custody of samples from the field sample custodian or a transferee.

One individual may fulfill more than one of the roles described above while in the field.

3.2 Sample and Evidence Identification

PERFORMANCE OBJECTIVES:

- To accurately identify samples and evidence collected.
- To adequately insure that chain-of-custody was maintained.

3.2.1 Sample Identification

The method of sample identification used depends on the type of sample collected. Samples collected for specific field analyses or measurement data are recorded directly in bound field logbooks or recorded directly on the Chain-of-Custody Record, with identifying information, while in the custody of the samplers. Examples include pH, temperature, and conductivity. Samples collected for laboratory analyses are identified by using standard sample tags (Figure 3-1) which are attached to the sample containers. In some cases, particularly with biological samples, the sample tags may have to be included with or wrapped around the samples. The sample tags are sequentially numbered and are accountable documents after they are completed and attached to a sample or other physical evidence. The following information shall be included on the sample tag using waterproof, non-erasable ink:

- project number;
- field identification or sample station number;
- date and time of sample collection;
- designation of the sample as a grab or composite;
- type of sample (water, wastewater, leachate, soil, sediment, etc.) and a very brief description of the sampling location;
- the signature of either the sampler(s) or the designated sampling team leader and the field sample custodian (if appropriate);
- whether the sample is preserved or unpreserved;
- the general types of analyses to be performed (checked on front of tag); and
- any relevant comments (such as readily detectable or identifiable odor, color, or known toxic properties).

Samples or other physical evidence collected during criminal investigations are to be identified by using the "criminal sample tag." This tag is similar to the standard sample tag shown in Figure 3-1, except that it has a red border around the front and a red background on the back of the tag. If a criminal sample tag is not available, the white sample tag may be used and should be marked "Criminal" in bold letters on

the tag.

If a sample is split with a facility, state regulatory agency, or other party representative, the recipient should be provided (if enough sample is available) with an equal weight or volume of sample (see Section 2.3.6). The split sample should be clearly marked or identified with a stick-on label.

Tags for blank or duplicate samples will be marked "blank" or "duplicate," respectively. This requirement does not apply to blind-spiked or blank samples which are to be submitted for laboratory quality control purposes. Blind-spiked or blank samples shall not be identified as such. This identifying information shall also be recorded in the bound field logbooks and on the Chain-Of-Custody Record as outlined in Sections 3.3 and 3.5.

3.2.2 Photograph Identification

Photographs used in investigative reports or placed in the official files shall be identified on the back of the print with the following information:

- A brief, but accurate description of what the photograph shows, including the name of the facility or site and the location.
- The date and time that the photograph was taken.
- The name of the photographer.

When photographs are taken, a record of each frame exposed shall be kept in the bound field logbook along with the information required for each photograph. The film shall be developed with the negatives supplied uncut. The field investigator shall then enter the required information on the prints, using the photographic record from the bound field logbook, to identify each photograph. For criminal investigations, the negatives must be maintained with the bound field logbook in the project file and stored in a secured file cabinet.

3.2.3 Identification of Physical Evidence

Physical evidence, other than samples, shall be identified by utilizing a sample tag or recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (drums for example), mark the vessel or container with the field identification or sample station number for future identification, when necessary. The vessel or container may be labeled with an indelible marker (e.g., paint stick or spray paint). The vessel or container need not be marked if it already has a unique marking or serial number; however, these numbers shall be recorded in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. Mark the charts and write the following information on these charts while they are still in the instrument or recorder :

- Starting and ending time(s) and date(s) for the chart.
- Take an instantaneous measurement of the media being measured by the recorder. The instantaneous measurement shall be entered at the appropriate location on the chart along with the date and time of the measurement.
- A description of the location being monitored and any other information required to interpret the data such as type of flow device, chart units, factors, etc.

If a sample is split with a facility, state regulatory agency, or other party representative, the recipient should be provided (if enough sample is available) with an equal weight or volume of sample (see Section 2.3.6). The split sample should be clearly marked or identified with a stick-on label.

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Physical evidence, other than samples, shall be identified by utilizing a sample tag or recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (drums for example), mark the vessel or container with the field identification or sample station number for future identification, when necessary. The vessel or container may be labeled with an indelible marker (e.g., paint stick or spray paint). The vessel or container need not be marked if it already has a unique marking or serial number; however, these numbers shall be recorded in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. Mark the charts and write the following information on these charts while they are still in the instrument or recorder :

- Starting and ending time(s) and date(s) for the chart.
- Take an instantaneous measurement of the media being measured by the recorder. The instantaneous measurement shall be entered at the appropriate location on the chart along with the date and time of the measurement.
- A description of the location being monitored and any other information required to interpret

the data such as type of flow device, chart units, factors, etc.

All of the above information should be initialed by the field investigator. After the chart has been removed, the field investigator shall indicate on the chart who the chart (or copy of the chart) was received from and enter the date and time, as well as the investigator's initials.

Documents such as technical reports, laboratory reports, etc., should be marked with the field investigator's signature, the date, the number of pages, and from whom they were received. Confidential documents should not be accepted, except in special circumstances such as process audits, hazardous waste site investigations, etc.

3.3 Chain-of-Custody Procedures

PERFORMANCE OBJECTIVE:

- To maintain and document the possession of samples or other evidence from the time of collection until they or the data derived from the samples are introduced as evidence.

3.3.1 Introduction

Chain-of-custody procedures are comprised of the following elements; 1) maintaining sample custody and 2) documentation of samples for evidence. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample from the moment of collection to its introduction into evidence.

3.3.2 Sample Custody

A sample or other physical evidence is in custody if:

- it is in the actual possession of an investigator;
- it is in the view of an investigator, after being in their physical possession;
- it was in the physical possession of an investigator and then they secured it to prevent tampering; and/or
- it is placed in a designated secure area.

3.3.3 Documentation of Chain-of-Custody

<u>Sample Tag</u>

A sample tag (Figure 3-1) should be completed for each sample using waterproof, non-erasable ink as specified in Section 3.2.

Sample Seals

Samples should be sealed as soon as possible following collection utilizing the EPA custody seal (EPA Form 7500-2(R7-75)) shown in Figure 3-2. A similar seal is used for samples collected during criminal investigations, however, the seal is red. Though not required, red custody seal is preferred for sealing samples collected during criminal investigations. The sample custodian should write the date and their signature or initials on the seal. The use of custody seals may be waived if field investigators keep the samples in their custody as defined in Section 3.3.2 from the time of collection until the samples are delivered to the laboratory analyzing the samples.

Chain-of-Custody Record

The field Chain-Of-Custody Record (Figure 3-3) is used to record the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence or sample sets shall be accompanied by a Chain-Of-Custody Record. This Chain-Of-Custody Record documents transfer of custody of samples from the sample custodian to another person, to the laboratory, or other organizational elements. To simplify the Chain-of-Custody Record and eliminate potential litigation problems, as few people as possible should have custody of the samples or physical evidence during the investigation. This form shall not be used to document the collection of split samples where there is a legal requirement to provide a receipt for samples (see Section 3.4). The Chain-Of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A Chain-of-Custody Record will be completed for all samples or physical evidence collected. A separate Chain-of-Custody Record should be used for each final destination or laboratory utilized during the investigation.

The following information must be supplied in the indicated spaces (Figure 3-3) to complete the field Chain-Of-Custody Record.

- The project number.
- The project name.
- All samplers and sampling team leaders (if applicable) must sign in the designated signature block.
- The sampling station number, date, and time of sample collection, grab or composite sample designation, and a brief description of the type of sample and/or the sampling location must be included on each line. One sample should be entered on each line and a sample should not be split among multiple lines.
- If multiple sampling teams are collecting samples, the sampling team leader's name should be indicated in the "Tag No./Remarks" column.
- If the individual serving as the field sample custodian is different from the individual serving as the project leader, the field sample custodian's name and the title of the sample custodian (e.g., Jane Doe, Sample Custodian) should be recorded in the "Remarks" section in the top right corner of the Chain-of-Custody Record. The Remarks section may also be used to record airbill numbers, registered or certified mail serial numbers, or other pertinent information.

- The total number of sample containers must be listed in the "Total Containers" column for each sample. The number of individual containers for each analysis must also be listed. There should not be more than one sample type per sample. Required analyses should be circled or entered in the appropriate location as indicated on the Chain-of-Custody Record.

- The tag numbers for each sample and any needed remarks are to be supplied in the "Tag No./Remarks" column.

- The sample custodian and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. The person who originally relinquishes custody should be the sample custodian. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurred should be documented in the proper space on the Chain-of-Custody Record.

- Usually, the last person receiving the samples or evidence should be the laboratory sample custodian or their designee(s).

The Chain-of-Custody Record is a serialized document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

If chain-of-custody is required for documents received during investigations, the documents should be placed in large envelopes, and the contents should be noted on the envelope. The envelope shall be sealed and an EPA custody seal placed on the envelope such that it cannot be opened without breaking the seal. A Chain-Of-Custody Record shall be maintained for the envelope. Any time the EPA seal is broken, that fact shall be noted on the Chain-Of-Custody Record and a new seal affixed. The information on the seal should include the sample custodian's signature or initials, as well as the date.

Physical evidence such as video tapes or other small items shall be placed in Zip-Loc® type bags or envelopes and an EPA custody seal should be affixed so that they cannot be opened without breaking the seal. A Chain-Of-Custody Record shall be maintained for these items. Any time the EPA seal is broken, that fact shall be noted on the Chain-of-Custody Record and a new seal affixed. The information on the seal should include the sample field custodian's signature or initials, as well as the date.

EPA custody seals can be used to maintain custody of other items when necessary by using similar procedures as those previously outlined in this section.

Samples should not be accepted from other sources unless the sample collection procedures used are known to be acceptable, can be documented, and the sample chain-of-custody can be established. If such samples are accepted, a standard sample tag containing all relevant information and the Chain-Of-Custody Record shall be completed for each set of samples.

3.3.4 Transfer of Custody with Shipment

- Samples shall be properly packaged for shipment in accordance with the procedures outlined in Appendix D.

- All samples shall be accompanied by the Chain-Of-Custody Record. The original and one copy of the Record will be placed in a plastic bag inside the secured shipping container if samples are shipped. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-Custody Record. One copy of the Record will be retained by the project leader. The original Chain-of-Custody Record will be transmitted to the project leader after the samples are accepted by the laboratory. This copy will become a part of the project file.

- If sent by mail, the package shall be registered with return receipt requested. If sent by common carrier, a Government Bill of Lading (GBL) or Air Bill should be used. Receipts from post offices, copies of GBL's, and Air Bills shall be retained as part of the documentation of the chain-of-custody. The Air Bill number, GBL number, or registered mail serial number shall be recorded in the remarks section of the Chain-Of-Custody Record or in another designated area if using a form other than that shown in Figure 3-2.

3.4 Receipt for Samples Form (CERCLA/RCRA/TSCA)

3.4.1 Introduction

Section 3007 of the Resource Conservation and Recovery Act (RCRA) of 1976 and Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980 require that a "receipt" for all facility samples collected during inspections and investigations be given to the owner/operator of each facility before the field investigator departs the premises. The Toxic Substances Control Act (TSCA) contains similar provisions.

3.4.2 Receipt for Samples Form

The Receipt for Samples form (Figure 3-4) is to be used to satisfy the receipt for samples provisions of RCRA, CERCLA, and TSCA. The form also documents that split samples were offered and either "Received" or "Declined" by the owner/operator of the facility or site being investigated. The following information must be supplied and entered on the Receipt for Samples form.

- The project number, project name, name of facility or site, and location of the facility or site must be entered at the top of the form in the indicated locations.

- The sampler(s) must sign the form in the indicated location. If multiple sample teams are collecting samples, the sample team leader's name should be indicated in the "EPA Sample Tag No.'s/Remarks" column.

- Each sample collected from the facility or site must be documented in the sample record portion of the form. The sample station number, date and time of sample collection, composite or grab sample designation, whether or not split samples were collected (yes or no should be entered under the split sample column), the tag numbers of samples collected which will be removed from the site, a brief description of each sampling location, and the total number of sample containers for each sample must be entered.

- The bottom of the form is used to document the site operator's acceptance or rejection of split samples. The project leader must sign and complete the information in the "Split Samples Transferred By" section (date and time must be entered). If split samples were not collected, the project leader should initial and place a single line through "Split Samples Transferred By" in this section. The operator of the site must indicate whether split samples were received or declined and sign the form. The operator must give their title, telephone number, and the date and time they signed the form. If the operator refuses to sign the form, the sampler(s) should note this fact in the operator's signature block and initial this entry.

The Receipt for Samples form is serialized and becomes an accountable document after it is completed. A copy of the form is to be given to the facility or site owner/operator. The original copy of the form must be maintained in the project files.

3.5 Field Records

PERFORMANCE OBJECTIVE:

- To accurately and completely document all field activities.

Each project should have a dedicated logbook. The project leader's name, the sample team leader's name (if appropriate), the project name and location, and the project number should be entered on the inside of the front cover of the logbook. It is recommended that each page in the logbook be numbered and dated. The entries should be legible and contain accurate and inclusive documentation of an individual's project activities. At the end of all entries for each day, or at the end of a particular event if appropriate, the investigator should draw a diagonal line and initial indicating the conclusion of the entry. Since field records are the basis for later written reports, language should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate. Once completed, these field logbooks become accountable documents and must be maintained as part of the official project files. All aspects of sample collection and handling, as well as visual observations, shall be documented in the field logbooks. The following is a list of information that should be included in the logbook:

- sample collection equipment (where appropriate);

- field analytical equipment, and equipment utilized to make physical measurements shall be identified;

- calculations, results, and calibration data for field sampling, field analytical, and field physical measurement equipment;

- property numbers of any sampling equipment used, if available;
- sampling station identification;
- time of sample collection;
- description of the sample location;
- description of the sample;
- who collected the sample;
- how the sample was collected;
- diagrams of processes;
- maps/sketches of sampling locations; and
- weather conditions that may affect the sample (e.g., rain, extreme heat or cold, wind, etc.)

3.6 Document Control

The term document control refers to the maintenance of inspection and investigation project files. All project files shall be maintained in accordance with Divisional guidelines. All documents as outlined below shall be kept in project files. Investigators may keep copies of reports in their personal files, however, all official and original documents relating to inspections and investigations shall be placed in the official project files. The following documents shall be placed in the project file, if applicable:

- request memo from the program office;
- copy of the study plan;
- original Chain-Of-Custody Records and bound field logbooks;
- copy of the Receipt for Sample forms;
- records obtained during the investigation;
- complete copy of the analytical data and memorandums transmitting analytical data;
- official correspondence received by or issued by the Branch relating to the investigation including records of telephone calls;
- photographs and negatives associated with the project;
- one copy of the final report and transmittal memorandum(s); and
- relevant documents related to the original investigation/inspection or follow-up activities related to the investigation/inspection.

Under no circumstances are any inappropriate personal observations or irrelevant information to

be filed in the official project files. The project leader shall review the file at the conclusion of the project to insure that it is complete.

3.7 Disposal of Samples or Other Physical Evidence

Disposal of samples or other physical evidence obtained during investigations is conducted on a case-by-case basis. Before samples which have been analyzed are disposed, laboratory personnel shall contact the project leader or his/her supervisor in writing, requesting permission to dispose of the samples. The samples will not be disposed of until the project leader or his/her supervisor completes the appropriate portions of the memorandum, signs, and returns the memorandum to the laboratory, specifically giving them permission to dispose of the samples. Personnel should check with the EPA Program Office requesting the inspection or investigation before granting permission to dispose of samples or other physical evidence. The following general guidance is offered for the disposal of samples or other physical evidence:

- No samples, physical evidence, or any other document associated with a criminal investigation shall be disposed without written permission from EPA's Criminal Investigations Division.

- Internal quality assurance samples are routinely disposed after the analytical results are reported. The laboratory does not advise the Quality Assurance Officer of the disposal of these samples.

- Samples associated with routine inspections may be disposed following approval from the project leader.

After samples are disposed, the laboratory shall send the sample tags to the Field Equipment Center (FEC) coordinator. These sample tags are accountable and must be placed and maintained in official files at the FEC.

3.8 Field Operations Records Management System (FORMS)

FORMS is a computer program designed to streamline the documentation required by ESD and/or the Contract Laboratory Program (CLP) for sample identification and chain-of-custody. Once the appropriate information is entered into the computer, FORMS will generate stick-on labels for the sample tags, sample containers (CLP), and field logbooks, and will generate the sample receipt and chain-of-custody reports for the appropriate laboratory. The advantages to this system include faster processing of samples and increased accuracy. Accuracy is increased because the information is entered only once, and consequently, consistent from the log book to the tags, bottle labels, and chain-of-custody forms. Operating instructions are available for use with the FORMS program.

FIGURE 3-1
SAMPLE TAG

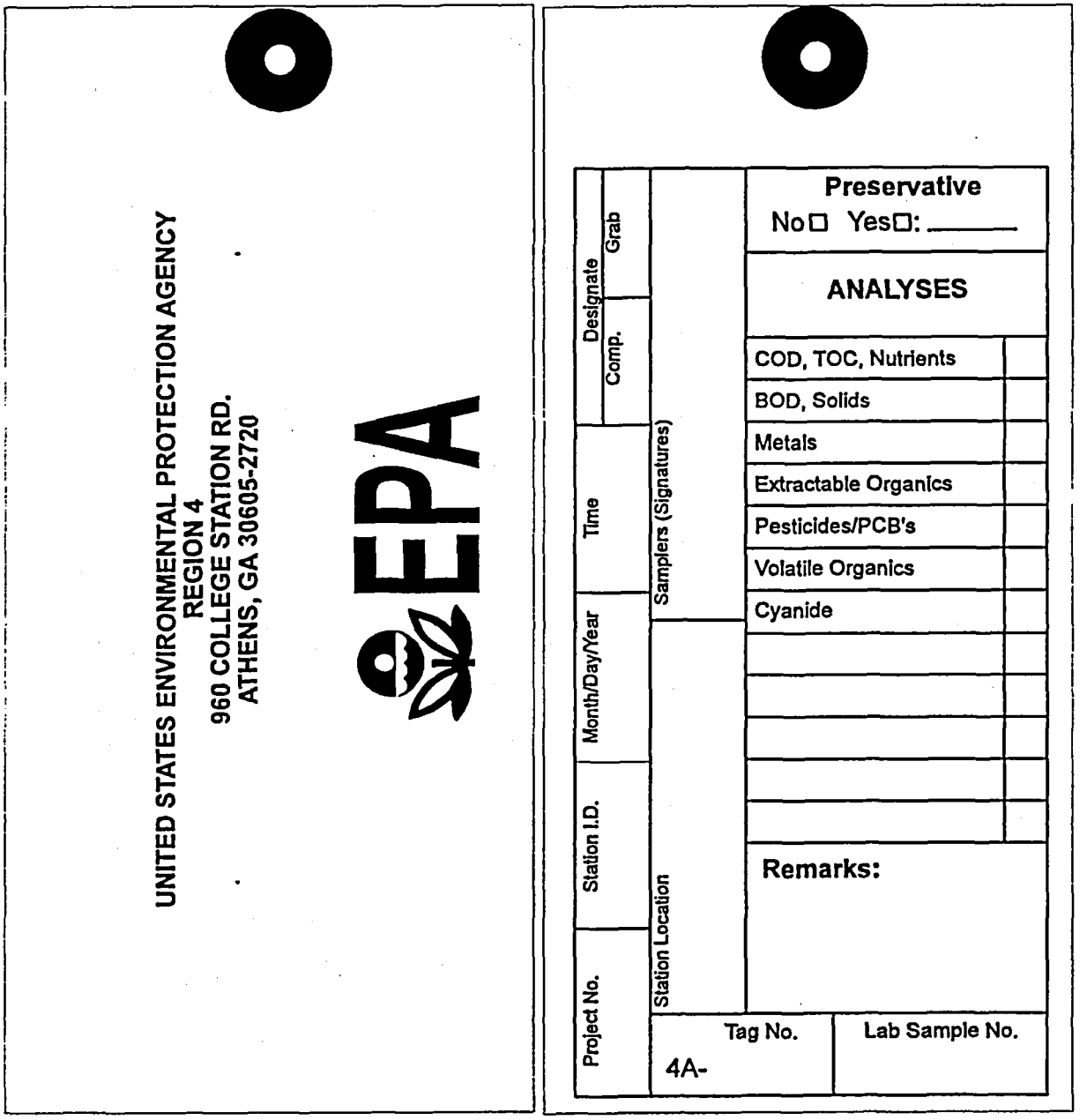


FIGURE 3-2
EPA CUSTODY SEAL

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICIAL SAMPLE SEAL	SAMPLE No.	DATE	SEAL BROKEN BY	EPA FORM 1500-2(R7-75)
	SIGNATURE			
	PRINT NAME AND TITLE (*INSPECTOR, ANALYST or TECHNICIAN*)		DATE	

US EPA RETGION 4
960 COLLEGE STATION ROAD
ATHENS, GEORGIA 30605-2720

CHAIN OF CUSTODY RECORD

PROJECT NO.

PROJECT LEADER

REMARKS

PROJECT NAME/LOCATION

SAMPLE TYPES
1. SURFACE WATER
2. GROUND WATER
3. POTABLE WATER
4. WASTEWATER
5. LEACHATE
6. SOIL/SEDIMENT
7. SLUDGE
8. WASTE
9. AIR
10. FISH
11. OTHER_____

SAMPLERS (SIGN)

CIRCLE/ADD parameters desired. List number of containers submitted.

ANALYSES: COD, TOC, NUTRIENTS; BOD, TOC; METALS; EXT ORG/PEST/PCBs; VOA; CYANIDE

STATION NO.	SAMPLE TYPE	19__ DATE	TIME	COMP	GRAB	STATION LOCATION/DESCRIPTION	TOTAL CONTAINERS	COD, TOC, NUTRIENTS	BOD, TOC	METALS	EXT ORG/PEST/PCBs	VOA	CYANIDE				TAG NO./REMARKS	LAB USE ONLY

RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)	RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)
RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)	RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)

DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory; Pink copy retained by laboratory
White copy is returned to samplers; Yellow copy retained by samplers

*U.S. GPO 1989-732 0 188

4-17906 (10/89)

FIGURE 3-3
CHAIN-OF-CUSTODY FORM

US EPA REGION 4
960 COLLEGE STATION ROAD
ATHENS, GEORGIA 30605-2720

RECEIPT FOR SAMPLES

PROJECT NO.	PROJECT NAME	NAME & LOCATION OF FACILITY/SITE
SAMPLERS: (SIGNATURE)		

STATION NO.	19__ DATE	TIME	COMP	GRAB	STATION LOCATION/DESCRIPTION	NO. OF EPA CONTAINERS	SPLIT SAMPLES Y OR N	EPA SAMPLE TAG NO.S/REMARKS

SPLIT SAMPLES TRANSFERRED BY: (PRINT)	DATE	SPLIT SAMPLES RECEIVED BY ☐ OR DECLINED BY ☐ (PRINT)	DATE/TIME
(SIGN)	TIME	(SIGN)	TELEPHONE
		(TITLE)	

DISTRIBUTION: Original to Coordinator Field Files; Copy to Facility/Site Representative

*U.S. GOVERNMENT PRINTING OFFICE: 1990-531-617 (12/89)

No. 4 4609

FIGURE 3-4
RECEIPT FOR SAMPLES FORM

ATTACHMENT C

SECTION 11
SEDIMENT SAMPLING

PERFORMANCE OBJECTIVE:

- To collect a representative sample of sediment from a surface water body.

11.1 Introduction

Sampling techniques and equipment are designed to minimize effects on the chemical and physical integrity of the sample. If the guidance in this section is followed, a representative sample of the sediment should be obtained.

The physical location of the investigator when collecting a sample may dictate the equipment to be used. Wading is the preferred method for reaching the sampling location, particularly if the stream has a noticeable current (is not impounded). However, wading may disrupt bottom sediments causing biased results. If the stream is too deep to wade, the sediment sample may be collected from a boat or from a bridge.

To collect a sediment sample from a streambed, a variety of methods can be used:

- Dredges (Peterson, Eckman, Ponar),
- Coring (tubes, augers)
- Scoops (BMH-60, standard scoop) and spoons

Regardless of the method used, precautions should be taken to insure that the sample collected is representative of the streambed. These methods are discussed in the following paragraphs.

11.2 Sediment Sampling Equipment

11.2.1 Scoops and Spoons

If the surface water body is wadeable, the easiest way to collect a sediment sample is by using a stainless steel scoop or spoon. The sampling method is accomplished by wading into the surface water body and while facing upstream (into the current), scooping the sample along the bottom of the surface water body in the upstream direction. Excess water may be removed from the scoop or spoon. However, this may result in the loss of some fine particle size material associated with the bottom of the surface water body. Aliquots of the sample are then placed in a glass pan and homogenized according to the quartering method described in Section 5.13.8 of this SOP.

In surface water bodies that are too deep to wade, but less than eight feet deep, a stainless steel scoop or spoon attached to a piece of conduit can be used either from the banks if the surface water body is narrow or from a boat. The sediment is placed into a glass pan and mixed according to Section 5.13.8 of this SOP.

If the surface water body has a significant flow and is too deep to wade, a BMH-60 sampler may be used. The BMH-60 is not particularly efficient in mud or other soft substrates because its weight will cause penetration to deeper sediments, thus missing the most recently deposited material at the sediment water interface. It is also difficult to release secured samples in an undisturbed fashion that would readily permit subsampling. The BMH-60 may be used provided that caution is exercised by only taking subsamples that have not been in contact with the metal walls of the sampler.

11.2.2 Dredges

For routine analyses, the Peterson dredge can be used when the bottom is rocky, in very deep water, or when the stream velocity is high. The dredge should be lowered very slowly as it approaches bottom, since it can displace and miss fine particle size sediment if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where the bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high velocities. It should not be used from a bridge that is more than a few feet above the water, because the spring mechanism which activates the sampler can be damaged by the messenger if dropped from too great a height.

The Ponar dredge is a modification of the Peterson dredge and is similar in size and weight. It has been modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing turbulence around the dredge. The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The "mini" Ponar dredge is a smaller, much lighter version of the Ponar dredge. It is used to collect smaller sample volumes when working in industrial tanks, lagoons, ponds, and shallow water bodies. It is a good device use when collecting sludge and sediment containing hazardous constituents because the size of the dredge makes it more amenable to field cleaning.

11.2.3 Coring

Core samplers are used to sample vertical columns of sediment. They are particularly useful when a historical picture of sediment deposition is desired since they preserve the sequential layering of the deposit, and when it is desirable to minimize the loss of material at the sediment-water interface. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand push tubes to weight or gravity driven devices.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines of the sediment-water interface are only minimally disturbed; the sample is withdrawn intact permitting the removal of only those layers of interest; core liners manufactured of glass or Teflon® can be purchased, thus reducing possible sample contamination; and the samples are easily delivered to the lab for analysis in the tube in which they were collected.

The disadvantage of coring devices is that a relatively small surface area and sample size is obtained often necessitating repetitive sampling in order to obtain the required amount of material for analysis. Because it is believed that this disadvantage is offset by the advantages, coring devices are recommended in sampling sediments for trace organic compounds or metals analyses.

In shallow, wadeable waters, the direct use of a core liner or tube manufactured of Teflon®, plastic, or glass is recommended for the collection of sediment samples. (Plastic tubes are principally used for collection of samples for physical parameters such as particle size analysis). Their use can also be extended to deep waters when SCUBA diving equipment is utilized. Teflon® or plastic are preferred to glass since they are unbreakable which reduces the possibility of sample loss. Stainless steel push tubes are also acceptable and provide a better cutting edge and higher strength than Teflon®. The use of glass or Teflon® tubes eliminates any possible metals contamination from core barrels, cutting heads, and retainers. The tube should be approximately 12 inches in length if only recently deposited sediments (8 inches or less) are to be sampled. Longer tubes should be used when the depth of the substrate exceeds 8 inches. Soft or semi-consolidated sediments such as mud and clays have a greater adherence to the inside of the tube and thus can be sampled with larger diameter tubes. Because coarse or unconsolidated sediments such as sands and gravel tend to fall out of the tube, a small diameter is required for them. A tube about two inches in diameter is usually the best size. The wall thickness of the tube should be about 1/3 inch for Teflon®, plastic, or glass. The inside wall may be filed down at the bottom of the tube to provide a cutting edge and facilitate entry of the liner into the substrate.

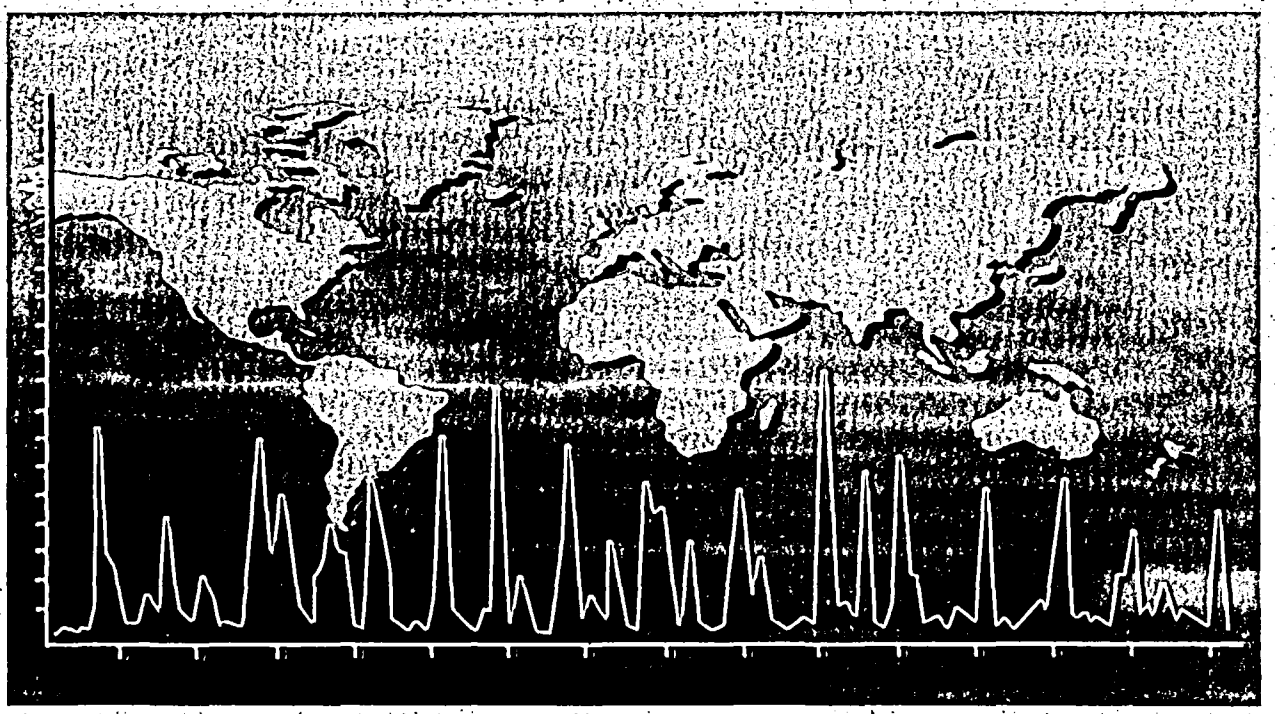
Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The core tube is pushed into the substrate until four inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction. The top of the tube is then capped to provide a suction and reduce the chance of losing the sample. A Teflon® plug or a sheet of Teflon® held in place by a rubber stopper or cork may be used. After capping, the tube is slowly extracted with the suction and adherence of the sediment keeping the sample in the tube. Before pulling the bottom part of the core above the water surface, it too should be capped.

When extensive core sampling is required, such as a cross-sectional examination of a streambed (with an objective of profiling both the physical and chemical contents of the sediment), a whole core must be collected. A strong coring tube such as one made from aluminum, steel or stainless steel is needed to penetrate the sediment and underlying clay or sands. A coring device can be used to collect an intact sediment core from streambeds that have soft bottoms which allows several inches of penetration. It is recommended that the corer have a checkvalve built into the driving head which allows water and air to escape from the cutting core, thus creating a partial vacuum which helps to hold the sediment core in the tube. The corer is attached to a standard auger extension and handle, allowing it to be corkscrewed into the sediment from a boat or while wading. The coring tube is easily detached and the intact sediment core is removed with an extraction device.

Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by simply turning the core tube to its side, and gently pouring the liquid out until fine sediment particles appear in the waste liquid. The loss of some of the fine sediments usually occurs with this technique.

ATTACHMENT D

TRIANGLE LABS



Quality Assurance

YOUR ANALYTICAL SOURCE

1999

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MISSION STATEMENT

Triangle Laboratories is in the business of applying scientific knowledge and measurements to the solution of health, environmental and other issues confronting society.

Beliefs

We believe that we must excel in relationships with our customers, our employees, and our investors while establishing leadership in our technology and operations management.

We believe that in all things and at all times our behavior must follow the highest ethical standards. This includes commitments made to customers, suppliers, employees, investors, and to one another.

We believe that to our customers, we must be the laboratory of choice. Our marketing program will always honestly inform. We will set the quality and timeliness standards in our markets. We will structure our company so that we have the flexibility and versatility required to be responsive to customer's needs. We will work until the customer is satisfied.

We believe that for our employees, we must be the employer of choice. Through the application of high ethical standards, maintenance of efficient operations and a respect for diversity, we will provide a work environment that enriches and builds people while giving them an opportunity to excel and enjoy the dignity, pride, and material rewards of being part of a winning team.

We believe that for our investors, we must commit to the development of long term value in their investment. This will be accomplished by taking those risks that have an appropriate probability of reward, controlling expenses to maintain high profitability and aggressively seeking opportunities to achieve growth through expansion of existing business and developing new business opportunities.

We commit ourselves to conducting research and development so that we are always a leader in technology, to apply the knowledge gained to maintain efficient operations and to service our customers needs in a timely manner while providing a reasonable profit for our investors.

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4	Containers, Preservatives, and Holding Times	April 15, 1999	1

Section 1

INTRODUCTION

This manual is a description of the quality assurance program employed at Triangle Laboratories, Inc., referred to hereafter as Triangle Labs. It is intended to provide employees, accrediting agencies, and clients with the information needed to understand how an effective quality assurance system is maintained at Triangle Labs. The QA Manual is divided into fifteen sections and several appendices. The first three sections pertain to the manual itself. Sections 4 - 7 provide general descriptions of Triangle Labs, including its objectives, policies, facilities, organization, personnel, and services. The remaining sections describe specific quality assurance activities as practiced within different functions or work units. The order of sections 8 - 12 closely follows that of the production process at Triangle Labs. The appendices provide supplemental materials that support the descriptions in the QA Manual sections.

Written procedures for implementing the activities described in this manual are maintained as standard operating procedures (SOP's) and as department specific training procedures. The SOP's are made available to the operating staff through the widely distributed SOP Manuals. The training procedures are maintained by the department managers. The provisions of this manual are binding upon all laboratory personnel assigned responsibilities described herein. All laboratory personnel must adhere implicitly to the Standard Operating Procedures.

Section 2

AUTHORIZATION

The quality assurance system described in this Quality Assurance Manual has the absolute support of the management at Triangle Labs.

The provision of quality analytical services to our customers has given us an enviable reputation and has made us a leader in the industry. Assuring that we maintain this status in providing quality products to our customers is the responsibility of every member of the laboratory staff. It is expected that everyone concerned will use this manual as a guide to quality improvement and to maintenance of our current standing as a quality-oriented laboratory.

Signature:	Date:
J. Ronald Hass J. Ronald Hass, Ph. D. President and Chief Executive Officer	4/19/99
Donald J Harvan Donald J. Harvan Quality Assurance Officer	4/19/99
Phil Albro Philip W. Albro, Ph. D. Technical Director	4/19/99

Section 3

MANAGEMENT OF THE QUALITY ASSURANCE MANUAL

The Quality Assurance Department is responsible for the publication and distribution of the Quality Assurance Manual. The manual is submitted to senior management for review and authorization annually. As major changes are implemented in the quality assurance system, the Quality Assurance Manual is revised and submitted to management for authorization. The assistance of laboratory management is essential for the publication of the QA Manual. Department specific information is supplied by the department supervisors for inclusion in the manual.

The authorization signatures found in Section 2 of the manual signify management review and approval of the Quality Assurance Manual. The authorization section must be kept current and reflect any organizational changes affecting the authorizing positions.

Document control procedures are applied to the distribution of the Quality Assurance Manual. Controlled copies are serially numbered and are updated each time a section is revised. Controlled copies of the manual may be distributed to an individual or a department. Uncontrolled copies may be issued to persons or organizations outside of Triangle Labs. These copies are distinctly marked "uncontrolled" and are not subject to updates upon revision of the manual. A distribution list is maintained for all controlled copies of the Quality Assurance Manual.

Upon revision, all text added or changed since the last issue of each section is marked with a vertical bar in the margin.

Section 4

OBJECTIVE AND POLICIES

Objective

The objective of the staff at Triangle Labs is to provide products and services which satisfy our clients' expectations and definitions of quality and which are legally defensible.

Policies

The management of Triangle Labs supports the following policies in order to achieve the objective and promote the overall quality assurance program:

- Standard operating procedures shall be implemented in order to determine client requirements and to clearly communicate these requirements within the laboratory.
- Organizational emphasis on quality improvement will take place through strong management commitment and leadership, employee empowerment and teamwork.
- A comprehensive quality control system shall be established and maintained in order to verify and assure continued precision and accuracy of analytical results.
- Adequate training on laboratory operations shall be available to all employees whose decisions may affect the quality of laboratory products.
- A comprehensive program of documentation shall be implemented to ensure maintenance of accountability and traceability throughout the analytical process.
- Measures shall be implemented to ensure that sample integrity is protected.
- Validation studies shall be performed for each analytical method, including extensive evaluations whenever major modifications have been implemented.
- The instrumentation, equipment, and materials used in the production process shall be controlled (i.e., purchased, verified, calibrated, maintained, monitored, and evaluated) to ensure that required standards are met.

- A comprehensive program for data reduction, validation, reporting, and archival shall be implemented.
- Preventive and corrective actions shall be taken to eliminate the causes of potential or actual nonconformance. Emphasis shall be placed on preventive measures.
- Measures shall be implemented in order to meet the requirements set forth by agencies from whom certifications and accreditations have been granted.

Section 5

LABORATORY DESCRIPTION

Triangle Laboratories, Inc.

The location, mailing address, and phone numbers for Triangle Laboratories, Inc. are:

Triangle Laboratories, Inc.
801 Capitola Drive
Durham, North Carolina 27713

P.O. Box 13485
Research Triangle Park, North Carolina 27709

(919) 544-5729
(919) 544-5491 (Facsimile)

Triangle Laboratories, Inc. is a privately held subchapter C Corporation registered and incorporated in the state of Delaware. Triangle Laboratories has been in business since 1984 and has established an unparalleled reputation for integrity and quality while undertaking the most challenging work in its industry. The company experienced rapid growth during the emergence of the environmental market. Recognizing the necessity of diversification even while the environmental business was in full swing, the company expanded internationally as well as moving into new markets. Triangle Laboratories currently serves two major market areas, environmental and pharmaceutical.

Facilities and Instrumentation

Triangle Laboratories, Inc. currently occupies more than 50,000 square feet. The facility is divided according to work function, including separate areas for sample receipt; sample, standard, and glassware preparation; sample and data storage; instrumentation; report generation, quality assurance; shipping; maintenance; and business/management offices.

Analytical instrumentation at Triangle Labs includes: high resolution gas chromatograph/high resolution mass spectrometers (HRGC/HRMS); high resolution gas chromatograph/low resolution mass spectrometers (HRGC/LRMS); high pressure liquid chromatograph/mass spectrometer/mass spectrometers (HPLC/MS/MS); high pressure liquid chromatograph (HPLC) with ultraviolet detector (UV); gas chromatographs (GC) with electron capture detectors (ECD) and flame ionization detectors(FID); AOX/TOX adsorption module and microcoulometric titration systems; ion chromatographs (IC); inductively coupled plasma atomic emission spectrophotometers (ICP) and atomic absorption spectrophotometers (AA).

Well maintained equipment is essential in assuring the timely delivery of complete, high quality analytical data to clients. This is facilitated through a program of regular maintenance for all equipment, equipment redundancy, an ample stock of spare parts, and an inventory of specialized test equipment to support rapid repair when unscheduled maintenance is required. Service technicians are available through contracts with local providers for most of the instruments. Procedures and schedules for preventive maintenance are available in several SOP's. All instrument maintenance, both preventative and corrective, is recorded in the dedicated maintenance logbook assigned to each instrument.

Environmental and Security Systems

Triangle Labs provides a secure environment for our employees, guests, clients, samples and analytical data.

Access

Standard procedures require that all exterior doors remain locked via keylock or combination lock unless manned. Visitors are required to sign the Visitor Log and must be accompanied by an employee of Triangle Labs.

The defined high security areas include all laboratories, data archives, computer system, data reduction offices, and quality assurance offices. Entry into these areas of the building are controlled by combination locks on the internal and external entry doors. Visitors must be accompanied by an employee of Triangle Labs at all times inside the high security area.

Several rules apply to protecting the combination lock codes. The combinations are changed periodically. New combinations are supplied to the active employees only by the employee's supervisor or the facility manager. When accompanied by visitors, employees obscure the punch lock combination from view.

Security

All doors are locked after hours and require a key for entry.

Archives

Limited access archive facilities are maintained that house all Triangle Labs copies of analytical reports, raw data, inactive logbooks, magnetic tapes and other data which facilitate traceability of analytical results. Materials housed in the archives are packaged to reduce potential damage from fire and water.

Chemical Storage and Disposal

All chemicals are stored in appropriate cabinets and are properly disposed of when necessary. All flammable solvents are kept in OSHA and NFPA approved cabinets. Acids are stored in OSHA approved acid cabinets. An authorized waste carrier is contracted to pick up lab waste monthly and dispose of it, usually by incineration, meeting all regulatory requirements. Post-analysis disposition of samples is dependent upon client requests. Remaining sample material may be returned to the client, safely discarded, or archived for a specific period of time.

Environmental Control

The working and storage environments are maintained in a safe and appropriate manner. Heating, ventilation and air-conditioning systems satisfy the needs of personnel, equipment and supplies. Lighting, noise and other environmental factors are also considered and kept at appropriate levels. Safety measures which protect personnel and property from injury or illness include the following: fume hoods, fire extinguishers and blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes and spill control kits. Triangle Laboratories has contracts which provide an occupational health program.

Accreditations, Certifications, Licenses and Registrations

Triangle Laboratories, Inc. has received approval from several state and national agencies. The American Association for Laboratory Accreditation has conferred accreditation upon Triangle Labs for technical competence in environmental testing. The laboratory has been validated by the United States Army Corps of Engineers, and while not currently under contract, Triangle Labs has performed organic analyses under the United States Environmental Protection Agency (USEPA) Contract Laboratory Program. Triangle Labs is registered under current Food and Drug Administration (FDA) regulations to engage in the testing of drugs; has received registration under the provisions of the Clinical Laboratory Improvement Amendments of 1988 (CLIA) to perform high complexity testing (dioxin and PCB's) of human samples; has been licensed, and has been provisionally certified by several USEPA regions to analyze drinking water samples for dioxin.

Section 6

ORGANIZATION AND PERSONNEL

Organization

Responsibility and Authority

At Triangle Labs, the management structure is shown in the Organizational Chart in Appendix 1A. Responsibilities and authority of key personnel are summarized later in this section. Brief resumes of key Triangle Labs personnel may be found in the company's Statement of Qualifications.

Verification Resources and Personnel

Verification activities include inspection and monitoring of process and product quality and auditing of the quality system, processes and products. Provision is made for personnel to be trained and have responsibility for these activities.

Production personnel, under the direct supervision of team leaders, are responsible for the inspection and monitoring of in-process and final products. Audits of the laboratory systems and products are performed by personnel independent of those performing the laboratory work. Quality system audits are carried out by Quality Assurance Department personnel, while data audits (audits of the final product) are carried out by employees in both Production and Quality Assurance.

Effective verification activities are achieved by the provision of adequate resources to personnel. These resources include adequate training, time for verification activities, knowledge about requirements, documented procedures, access to quality records, and adequate supplies and equipment necessary to perform verification.

Management Representative for Quality Assurance

The Quality Assurance Officer reports directly to the President, functions independently of production, and has the authority to implement and maintain the quality system. The management of Triangle Labs presents a strong commitment towards the important role of quality assurance in its organization. The Quality Assurance Officer and other members of the Quality Assurance Department interact frequently with personnel at all levels throughout the organization.

Management Review

A formal management review of the quality system occurs annually. The purpose of this review is to ensure that the quality system remains effective, meets the quality objectives and policies stated in Section 4 of this manual, and satisfies the requirements of state, national, and international certifications held by Triangle Labs. Records of management reviews shall be maintained in the Quality Assurance Department.

Personnel

Job Descriptions of Key Technical Personnel

While not all-inclusive of assigned duties, the following are brief descriptions of the chief technical personnel at Triangle Labs.

President/Chief Executive Officer: management of administrative, business, quality assurance, personnel and production activities; direct supervision of the Production Manager, the Quality Assurance Officer; and the Technical Director, minimum qualifications - education: Ph.D. Chemistry, experience: 10 years analytical chemistry.

Quality Assurance Officer: coordination and management of the Quality Assurance Department; reports directly to the President; responsible for overseeing all quality aspects of the laboratory; specific elements to be maintained are: the Standard Operating Procedures, Quality Assurance Manual; coordination of internal and external audits, performance samples and laboratory certification data; minimum qualifications - education: B.S. Chemistry or equivalent, experience: 5 years in scientific field.

Technical Director: consultation and guidance on specific technical and scientific questions and issues; performs audits of the technical apects of program operations; reports directly to the President; minimum qualifications - education: Ph.D. Chemistry, experience: 5 years analytical chemistry.

Production Manager: The production manager is responsible for developing production plans to meet commitments made to clients, identifying and resolving issues which impede success, and promptly reporting to the president any issues which cannot be resolved with available resources.

Team Leaders: management of a defined production area, instrumentation, reporting and/or sample preparation; minimum qualifications - education: B.S. Physical Science, experience: 2 years general analytical chemistry.

Recruitment Policy

The Personnel Department of Triangle Labs uses several methods of recruitment. Current employees are offered the earliest opportunity to apply for openings within the facility by posting available positions on the bulletin boards before outside sources are considered for candidates. Then, announcements are made in local newspapers, placement agencies (temporary and permanent), colleges and the Employment Security Commission offices. The recruitment process consists of collecting applications and resumes, distributing them to the appropriate supervisors, scheduling interviews as requested by supervisors and having candidates meet with relevant staff, a representative from the Personnel Department and senior Management. The references of promising candidates are investigated prior to making job offers.

Training

Training is provided for new employees and as continuing education for veteran employees, both at the Triangle Labs facility and off-site.

On-Site Training: Training goes on at different levels throughout the facilities. Numerous manuals, texts, videos, SOP's, journals, analytical protocols and in-house instructors are available to trainees. On-the-job training related directly to the position is done by team leaders or other qualified staff. Typically, a trainee goes through a stepwise method to learn procedures pertaining to such areas as analytical methodology, report generation or quality assurance activities: the trainee is given an SOP to read, the trainee observes the trainer performing the procedure, the trainee assists the trainer in performing the procedure several times, the trainee performs the procedure without assistance but with the trainer's frequent inspection of his work, and finally, the individual may perform the procedure without supervision. The Quality Assurance Manual is available to all employees whose activities have a direct impact on product quality. Cross training, supervisory training and other related training takes place on a scheduled basis and is documented for training files.

Off-Site Training: This type of training takes place on an as-needed basis. Recommendations and suggestions about promising educational programs come from all levels of staff. Completed studies are documented and updated regularly in the training files. Courses may be taken at local colleges and universities. Workshops and seminars are often made available by instrument manufacturers, software companies and national associations specializing in analytical chemistry or laboratory quality assurance.

Training Records Maintenance

Résumés, education and experience records, job descriptions and training records are maintained by the personnel department: Résumés are put in a uniform format upon hire. These résumés are updated on an annual basis or as needed. Additional education and experience is updated with the résumés. There is a job description for each position existing within the company. Active training records are kept on file in the work areas. Employees are responsible for maintaining their own training records. These training files contain records for any pertinent on- or off-site educational experiences, orientation records, SOP competence records or self help courses.

Safety and Health Policies

All personnel undertake a one day orientation upon initial employment and on-the-job intensive training concerning health and safety issues. Triangle Labs complies with the OSHA requirement that safety and health training takes place on an annual basis, with a careful introduction to new principles. We have contracted with Concentra to provide us with recommendations for the improvement of the safety and health practices at Triangle Labs. Triangle Labs' policy with respect to health and safety issues is presented in detail in several documents, which are provided to employees.

Section 7

ANALYTICAL SERVICES

Triangle Labs has assembled an international staff of unparalleled expertise in analytical sciences with particular specialization in mass spectrometry and the analysis of complex biological matrices. The skills of the staff are routinely applied to environmental samples, including of air, water, solid and tissue matrices, and to biological samples associated with studies supporting the research efforts of the pharmaceutical industry.

Pharmaceutical Services

Triangle Labs serves the research pharmaceutical industry by providing analytical results for drugs of interest in a variety of biomatrices. This work is typically associated with pharmacokinetic Phase I through Phase IV studies for reporting to the Food and Drug Administration (FDA).

GC/MS and LC/MS/MS methods are typically employed for these analyses. High resolution mass spectrometers and alternate ionization methods are frequently utilized to achieve low detection limits. The staff is also experienced in assays, both GC and LC based, for chiral compounds.

Environmental Services

Triangle Labs provides environmental analytical services which include the preparation and analysis of a wide variety of sample matrices for such analytical categories as:

Volatile and Semivolatile Organic Compounds, including Polychlorinated Biphenyls, by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry

Pesticides and Herbicides by High Resolution Gas Chromatography

Polychlorinated Dibenzo-*p*-Dioxins, Polychloro-dibenzofurans, Polychlorinated Biphenyls, and Polynuclear Aromatic Hydrocarbons by High Resolution Gas Chromatography/ High Resolution Mass Spectrometry

Polychlorinated Dibenzo-*p*-Dioxins and Polychloro-dibenzofurans by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry

Adsorbable Organic Halides and Total Organic Halides by Adsorption and Microcoulometric Titration

Inorganics by Ion Chromatography, Atomic Absorption Spectrophotometry, and

Inductively Coupled Plasma-Atomic Emission Spectrophotometry

Triangle Labs is experienced in the analysis of many matrices, including air, aqueous, plant and animal tissues, soils, and other solids. Air matrices currently analyzed include Modified Method 5 (MM5) samples and Volatile Organic Sampling Trains (VOST). Several auxiliary services are also offered, such as the provision and preparation of sampling containers (e.g., XAD traps, VOST tubes, and bottles).

Analytical Methodology and Target Compounds

Triangle Labs utilizes a variety of published and in-house analytical methods. In some cases minor modifications of methodology may be employed. Such modifications are validated prior to implementation in the laboratory. Target Compound Lists (TCL's) are chosen from the analytical methods. Published methodology utilized for each category of analytical services is listed below:

Volatile Organic Compounds (VOA) - Method 8260B

Semivolatile Organic Compounds (SVOA) - Method 8270C

Pesticides - Methods 8081

Polychlorinated Biphenyls (PCB's) - Modified Method 680 and 8081

Polychlorinated Dibenzo-p-Dioxins (PCDD's) and Polychlorodibenzofurans (PCDF's) - Methods 8290, 23, 0023A, 1613, 8280, 613 and NCASI 551

Adsorbable Organic Halides (AOX)/Total Organic Halides (TOX) - Methods DIN 38409, DIN 38414, EPA 9020, EPA 1650, PTS-RH: 012/90, SCAN-W 9:89, ISO/DIS 9562, and APHA 5320B

Inorganics - Ion Chromatography by Methods 7D, 26, 26A, 218.6, 300.0, and 9057; Trace Metals analyses by Methods 200.7, 6010, 7020, 7040, 7041, 7060, 7080, 7091, 7131, 7140, 7200, 7210, 7380, 7420, 7421, 7450, 7460, 7470, 7471, 7481, 7520, 7610, 7740, 7760, 7770, 7840, 7841, and 7870

Triangle Labs has developed in-house methods for the analyses for Polynuclear Aromatic Hydrocarbons (PAH's) by CARB Method 429 and Polychlorinated Biphenyls (PCB's by High Resolution Gas Chromatography/High Resolution Mass Spectrometry, using Triangle Labs Method TLI035. These methods are proprietary and utilize state-of-the-art technologies.

Additional information about analytical services and methodology can be found elsewhere in this manual. Selected analytical methods are summarized in

Appendices 2 and 3 of this manual.

Contract Review

For all analytical services to be provided by Triangle Labs, contract review is accomplished through the generation of a written quote or contract. Written quotes are utilized for short-term contracts, usually consisting of one analytical project. Written contracts are utilized for long-term contracts consisting of multiple analytical projects. Sales and Client Services personnel are responsible for implementing and documenting contract review. Client requirements, including special needs that are not normally provided by Triangle Labs, are defined and documented in the written quote or contract. Project scientists, who each have expertise in specific analytical services, are consulted to ensure special requirements can be met by the laboratory. If it is decided that the special requirements cannot be met, this is discussed with the client, and a counterproposal may be offered. Information about the capacity of the lab is made available to Sales and Client Services personnel on a regular basis. This practice allows the sales staff to make informed decisions regarding contracted delivery times.

Subcontracted Analyses

In dealing with any analyses that Triangle Labs cannot perform, there are established procedures for subcontracting. Depending on the nature of the client's requests for analyses, two courses of action may be followed. The client may be referred directly to another laboratory, or work may be subcontracted by Triangle Labs to another laboratory. The latter usually takes place at client request. When the subcontracted analysis is one that Triangle Labs has been certified to perform, the subcontract lab must have a quality assurance system in place that is consistent with Triangle's system. Incoming samples which will be subcontracted are subjected to normal sample receipt procedures by the sample custodian. The samples are prepared and shipped to the subcontract laboratory. Results are received at Triangle Labs, a copy is sent to the client, and the original is archived. Triangle Labs invoices the client for the subcontracted work.

Section 8

LABORATORY MATERIALS—PURCHASING AND HANDLING

Purchasing, Receiving, Inspection, Inventory and Storage of Laboratory Materials

Practices utilized for the purchase, receipt, inspection, inventory, and storage of laboratory materials are described in several SOP's. A completed purchase requisition form provides a clear description of the product ordered. This includes, where applicable, a precise identification and reference to any specifications that must be met. Purchases are pre-approved by department heads. The purchasing department orders the material, from an approved supplier whenever possible. Upon receipt of the goods, receiving personnel examine them for damage before signing the bill of lading. Within two days, items and quantities in all shipments are compared with what was ordered and this information is communicated to purchasing and accounts payable. All stocked items are stored in the warehouse and a monthly inventory is performed. Non-stocked inventory is forwarded to the requisitioning person. Reagent materials are assigned expiration dates and placed on shelves so that the older materials will be used first.

Sample Container Cleaning, Storage, Preparation and Shipping

While Triangle Labs does not perform sampling, sampling kits may be provided upon client request. The vials, jars, and bottles contained in the kits are purchased and must be QC class, precleaned, with a certificate of analysis. The certificates of analysis are maintained by Triangle Labs. Since kits are assembled only upon clients' requests, no "ready for shipping" kits are stored. Precleaned glassware is stored in small quantities in house. Sampling materials, such as XAD traps, PUFs and VOST tubes, are also provided to or owned by the client. These are prepared, stored and handled as detailed in several SOP's.

Prior to shipping, glass containers are wrapped in sheets of bubble wrap to prevent breakage. The containers are placed in plastic coolers with non-frozen ice packs and Chain-of-Custody forms, seals and labels enclosed in a ziplock bag. The kit is filled with additional packing material and sealed with tape for shipping.

Glassware Cleaning

All glassware used for the preparation of samples is cleaned as described in written standard operating procedures. These procedures include pre-rinses and soapy water washes. The pre-rinse may be solvent, water or acid solution depending on the analysis for which the glassware will be used. Basins and brushes are kept segregated so that cross contamination is minimized. Glassware used for high concentration analyses is kept segregated from glassware used for low concentration analyses, as is the glassware used for volatile, extractable organic compound and metals analyses. Glassware used for the analysis of extractable organic compounds, including

dioxins and furans, is subjected to a solvent soak and rinses with several solvents. All clean glassware is covered with aluminum foil and transferred to a proper storage location, taking care that the glassware is not intermixed with other types of glassware. In the Inorganic area, glassware is cleaned by a washing procedure that exceeds EPA guidelines. This glassware is washed with detergent, followed by acid soaks and multiple rinses with deionized water. The clean glassware is air-dried and stored in plastic bags.

Vendor Qualification

Vendors subject to qualification are those who provide critical laboratory supplies, chemicals, and calibration services which directly impact on the quality of our product. Placement on the approved vendor list is based on the vendor's ability to meet one or more qualification factors which cover the purchased product. These factors include but are not limited to:

1. the vendor's quality system or product meets an applicable state, national, or international standard, based on third party certification
2. an acceptable quality assurance plan/survey, or on-site audit;
3. the vendor provides quality inspection documentation with each shipment or batch lot of product;
4. the vendor passes comprehensive inspections of three consecutive product shipments;
5. a demonstrated history of acceptable product supply.

A vendor may be provisionally approved until qualification factor(s) are met, but in-house inspection of each batch lot of material is required. Previously approved vendors may be disqualified due to unacceptable performance.

Client Verification

When required by contract, the client or a representative may verify that purchased products conform to contract specifications. This verification may take place at the vendor's premises or at Triangle Labs. Client verification shall not be used as evidence of effective control of quality by the vendor and shall not absolve Triangle Labs of responsibility to provide an acceptable product.

Section 9

ANALYTICAL STANDARDS

During the analytical process, it is possible to obtain a variety of measurements. These include such measurements as volume, weight, concentration, pH, and temperature, to name just a few. The laboratory must implement practices that facilitate the traceability of these measurements to recognized standards of measurement.

Chemical Standards

The procurement, preparation, handling and storage of chemical standards is critical to the analytical process. It is through these chemical standards that reported analyte measurements in samples are traceable to reference values. Only the highest quality chemicals are used as reference materials at Triangle Labs. Whenever possible, standard solutions will be traceable to national standards, such as NIST, EPA or A2LA certified reference materials. Numerous written procedures describe the management of these analytical standards. These procedures are written to ensure consistency with the requirements of analytical methods and current certifications and accreditations.

Sources of Standards, Traceability and Verification

Triangle Laboratories purchases standards from approved suppliers of chemical standards. Occasionally, clients supply standards specifically for use in the preparation and analysis of their samples. Prior to using these standards, an agreement must be reached with the client about the handling and disposition of their standards. Information about these standards and any client requirements are recorded in the pertinent standards logbook. The chemist receiving a chemical standard shipment verifies that the information on the standard label is consistent with that on the supplier paperwork. Information about the standard is recorded in a standards logbook. Traceability of standard solutions is facilitated by the use of codes that unambiguously identify the supplier, materials and all derived preparations. Non-certified standard materials are verified against certified reference standards, when the latter are available.

Types of Standards

Analytical methodologies define a variety of standard solutions which are used by the laboratory. Included among them are: surrogate spikes, matrix spikes, internal standards, QC check standards, recovery standards, and calibration solutions. The composition and concentration of these solutions must conform to method specifications.

Standards are categorized at Triangle Labs according to the following definitions:

Primary Standard	A neat standard received from a supplier.
Stock Standard	A solution of a primary standard at a high concentration, used to prepare secondary standards. These may be prepared in-house or received from a supplier.
Secondary Standard	A solution of one or more stock standards, with each analyte prepared at a selected concentration, to be used as a beginning mixture for preparation of calibration or spike solutions. These may be prepared in-house or received from a supplier.
Working Standard	A solution that will be used without dilution for instrument calibration or sample fortification. These may be prepared in-house from secondary standards, or purchased from a supplier.

Preparation of Standards

The preparation of any standard solution is performed by an experienced chemist, and is documented in the appropriate standards logbook. New standard solutions are prepared as needed. The manner of preparation for a standard solution depends upon the required amount and concentration and its intended application. Several SOPs are utilized to assure the correct preparation and documentation of standard solutions.

All standards are assigned an expiration date. The supplier's assigned expiration date, if provided, is used for neat or primary standards. Otherwise, the expiration date is assigned based upon the supplier's date of preparation and the known stability of the analyte. (Some analytes are known to be highly volatile or to easily degrade or react.) When applicable, assigned expiration dates meet the requirements of analytical methods. A standard mixture is assigned an expiration date no later than that of the oldest components. The expiration date is only a guideline. Standards are removed from production prior to the assigned expiration date if deterioration is observed visually or analytically or if the integrity of the material can no longer be assured.

Analyte or standard components common to calibration solutions and associated sample fortification solutions may be of the same primary source or an independent source. Some methodologies require that primary standards of the same supplier batch or lot number be used for both. Certain spiked QC samples must be prepared from reference material that is independent of the associated calibration standards. New standards are prepared as necessary to meet these

requirements.

Inventory and Storage Documentation for all standards is carefully recorded in relevant standards logbooks and/or computer inventory system. The manner of storage for a standard is determined by its type and expiration date or shelf life. All light sensitive standards are stored in amber vials or bottles. Environmental organic standards are kept in designated refrigerators/freezers. Pharmaceutical standards are stored according to the conditions specified in the associated protocol, validation report or stability report. Analytical standards are never stored together with samples or extracts.

Measurement Equipment

All equipment used for measurement and testing shall meet the specific requirements of pertinent analytical methods and applicable certification agencies. This includes small equipment, such as thermometers, analytical balances, pH meters, autopipetors, and volumetric glassware; as well as large equipment, such as gas chromatographs and mass spectrometers.

Written procedures for the operation of measurement equipment, large or small, shall contain the information described below, where applicable. In addition, Section 11 on "Instrumental Analysis" of this manual contains more specific information about the calibration and operation of large measurement equipment.

- What equipment the procedure is to be performed on, including equipment type
- How the equipment is to be calibrated and used for measurement
- What measurements are to be made
- Acceptance criteria for the calibrations, including the accuracy and precision required
- Corrective action for failed acceptance criteria, including assessment of previous calibration results
- Basis used for calibration (e.g., national standards of measurement, such as NIST, ASTM, and A2LA; participation in EPA and state performance evaluations; round-robin studies with other laboratories)
- Frequency at which the equipment will be calibrated, adjusted and checked

- What records will be maintained to document the calibration and use of measurement equipment
- How the calibration status for equipment is determined (e.g., a sticker or logbook entry)
- What environmental conditions are necessary before measurement equipment may be calibrated or used for measurement
- What adjustments to measurement equipment, including software, cannot be made due to possible invalidation of the calibration setting
- How measurement equipment is to be handled, preserved, and stored in order to maintain accuracy and fitness for use

Section 10

SAMPLE RECEIPT, HANDLING AND PREPARATION

Sample Receipt and Chain-of-Custody

The Sample Custodian or a designated assistant receives deliveries of all samples. A unique project number is assigned to each shipment of samples received from a client, and the first in-house records for the new project, including an internal Chain-of-Custody, are initiated. When samples are hand delivered by a customer, the individual's name is recorded on the internal Chain-of-Custody. The shipping containers, their contents, and accompanying client documentation are examined by the Sample Custodian. Information about the presence and condition of custody seals and the state of preservation of the samples is noted on the internal Chain-of-Custody. Any discrepancies in documentation or problems with sample condition are also noted and brought to the attention of the client, who may provide clarification or further instructions. The Sample Custodian assigns an internal sample ID to each sample, which is labeled on the sample container. The following information pertinent to each sample is recorded on the internal Chain-of-Custody: internal sample ID, client sample ID, sample matrix and storage location. The original internal Chain-of-Custody is placed in storage with the samples. The sample receipt and handling SOP's describe procedures for sample receipt and log-in, chain-of custody, along with those for handling sample shipment containers provided by clients.

Sample Preservation and Security

Samples are stored in a manner which ensures their integrity and security. Samples are stored at temperatures which meet specifications of the methodology and client. Depending on the nature of the sample and the requirements of the method, samples may be stored in a freezer at -70° ± 20° C or at -20° ± 10° C, in a refrigerator or cooler at 4° ± 2° C, or in a cabinet at room temperature. Required preservation techniques may be found in Appendix 4 for most methods employed at Triangle Labs. Quality Assurance Project Plans (QAPP's) and protocols often give specific preservation requirements that must be observed. Addition of chemical preservative to sample containers normally takes place at the time of sample collection. Sample storage facilities at Triangle Labs are located within laboratory areas which are secured by locked doors. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage and transfer are outlined in written procedures. Care is taken to ensure that cross-contamination does not occur during sample storage. Temperatures of cold storage areas are monitored and recorded at least twice a day, and corrective action is taken as necessary. Walk-in coolers housing environmental samples and freezers used for pharmaceutical samples and standards are monitored electronically 24 hours a day. Further details about sample storage and preservation may be found in the sample receipt and handling SOP's.

Sample Preparation Procedures

Samples are prepared in a way that is method and matrix specific. Most environmental samples must be prepared within a method-specified time after sampling. These preparation holding times are complied with to the extent possible. Samples are occasionally received near or beyond the expiration of these holding times. For most methods employed at Triangle Labs, holding times may be found in Appendix 5. Applicable Quality Assurance Project Plans (QAPP's) and protocols must be consulted for project-specific holding time requirements. Many primary extracts require clean-up procedures before they may be injected into a GC or GC/MS analytical system. All sample preparation procedures employed at Triangle Labs are covered by appropriate SOP's.

Sample, Extract, and Digestate Archival and Disposal

The Sample Custodian and other authorized personnel are responsible for the archiving and disposal of raw samples, extracts, and digestates. Raw and prepared samples may not to be archived or disposed of until all of the designated analyses are complete and resultant analytical data are sent to clients. Samples in cold storage are retained there until at least 30 days after receipt. Archive samples are placed in boxes, labeled with the project numbers, and retained in a secured sample archive area for a specific length of time, prior to disposal. Written procedures describe routine archival and disposal practices. Clients are informed about these procedures and are given an opportunity to request exceptions to these routine practices. There is a storage fee for the retention of samples in cold storage or archive longer than the time established by routine practices. The client will be contacted prior to the issuance of this fee.

Sample Return to the Client

When a client has requested the return of samples, the Sample Custodian prepares and ships the samples according to written procedures. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of Triangle Labs. Clients are informed that a commercial carrier will deliver their samples.

Sample Loss, Damage, or Unsuitability

It is possible for samples or sample containers to be lost, damaged or determined to be unsuitable, for whatever reason, after initial receipt at Triangle Labs. Whenever this happens, the event is recorded in the sample handling documentation by the observer. The problem is brought to the attention of a Project Scientist, who reports it to the client. Plans for disposition of the affected sample(s) or containers are agreed upon with the client, carried out, and recorded in the project records.

Section 11

INSTRUMENTATION AND EQUIPMENT

Instrumental analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations and other instrument performance tests, analyzing prepared samples, and collecting data from the analyses. Instrumental analysis procedures, frequencies and acceptance criteria are described in several SOP's. A description of data collection and reduction at Triangle Labs is given in Section 12.

Instrument Operating Conditions

The published analytical methods normally define the instrument operating conditions (e.g., temperature programs, column conditions, flow rates). Where applicable, these guideline will be followed. However, they may be modified, for improved performance.

Calibration Procedures and Frequencies

Equipment used for inspection, measuring and testing must meet all specific requirements for proper measurement capability as identified in the pertinent analytical method and applicable certification agency. This includes small equipment and instruments as well as large analytical instruments such as gas chromatographs and mass spectrometers. Calibration procedures and frequencies specific to types of equipment are briefly described below.

The instrumental performance requirements of the published methods will be followed unless otherwise specified for a project. Other performance tests may also be executed to further demonstrate proper functioning of instrumentation.

Small equipment

Thermometers Laboratory thermometers are routinely checked for accuracy against certified, NIST-traceable thermometers. These calibrations are performed annually for mercury or alcohol in glass thermometers, and quarterly for metal thermometers. Infrared thermometer calibrations are verified daily. Correction factors derived from the annual and quarterly calibrations are applied to temperature readings where applicable. NIST-traceable thermometers are professionally calibrated and re-certified annually.

Balances Calibration checks are performed for each day of use for each balance. The calibration consists of a minimum of two weights which encompass the weight the balance will be used to measure. Calibration weight measurements must meet the acceptance criteria listed in the associated balance calibration log book. Each balance is serviced and calibrated by a certified professional, semiannually. The accuracy of the calibration weights are verified annually.

Volumetric Glassware All volumetric glassware used at Triangle Laboratories, Inc. must be type "class A". Volumetric glassware is never heated or placed in an oven.

Automatic Pipettes Delivery volumes for the automatic pipettes are checked gravimetrically monthly. Each pipette is checked throughout the volume range of use. Acceptance criteria for continued use is 2% RSD and 97.5 - 102.4% accuracy. Pipettes which fail to meet these criteria are tagged and removed from service until repaired.

pH Meters pH meters are calibrated prior to use each day. The meter is calibrated using a single buffer solution at mid-range and the pH of two other solutions (at low and high range) is measured and recorded to verify the accuracy over the range of the meter.

Conductivity Meters A five point calibration curve using potassium chloride (KCl) solutions is analyzed annually. A single KCl standard solution is used as a check standard each day the meter is used. Acceptance criteria is ±20% of the true value.

Gas Chromatography/Mass Spectrometry (GC/MS) and Liquid Chromatography/Mass Spectrometry (LC/MS/MS)

Tuning and Mass Calibration For high resolution, selected ion monitoring analyses, the high resolution mass spectrometer is tuned to give the required static resolving power, which is checked visually, using an oscilloscope. This measurement is confirmed by the use of a data system. The instrument is then mass calibrated using perfluorokerosene (PFK) or perfluorotributylamine (PFTBA). Mass calibration is adjusted automatically by the data system, to within ± 5 parts-per-million (ppm), approximately once per second during the course of all quantitative analyses.

The mass calibration of a quadrupole mass spectrometer is checked daily through the use of the perfluorotributylamine reference compound (FC-43/PFTBA). The instrument is adjusted to give specified peak ratios for this compound, consistent with the type of analysis to be performed. The GC/MS is tuned prior to performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, 50 ng of bromofluorobenzene (BFB) is used, and for semivolatiles analyses, 50 ng of decafluorotriphenylphosphine (DFTPP) is used.

Initial Calibration For environmental samples, the mass spectrometer response is typically calibrated by analyzing a set of five or more initial calibration solutions, as appropriate for each GC/MS method. Typically each solution is analyzed once, unless the method requires multiple analyses. The relative response factor for each analyte (target compounds, surrogate / internal / alternate standards) is calculated using the expression in Formula 11-1. The mean relative response factor for each analyte is then obtained using the expression in Formula 11-2. Integrated ion currents are utilized for these expressions. An acceptable

calibration must meet the method specified criteria for percent relative standard deviations (% RSD) of the mean relative response factors, calculated for each analyte. Failure to meet the criteria will result in corrective action (e.g., locating the source of the problem and adjusting the instrument tuning parameters) before repeating the rejected analysis. Triangle Labs does not analyze any samples unless the performance criteria for calibrations are satisfied.

For pharmaceutical samples, the calibration curve normally consists of a minimum of five standard concentrations analyzed at the beginning and end of the analytical sequence, or are dispersed throughout the analytical run depending on the client's requirements. All standards are used for the regression, with exclusion criteria defined in each method SOP.

Continuing Calibration

For environmental analyses, the initial calibration is verified through the analysis of a continuing calibration standard every 12 hours. The concentration of continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated using the expression in Figure 11-3. An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, either instrument maintenance is performed such that a new continuing calibration analysis meets all criteria or a new initial calibration will be established before any samples can be analyzed. No samples may be analyzed unless acceptance criteria have been met.

For pharmaceutical analyses, the calibration is verified through the analysis of quality control samples which are interspersed throughout the analytical sequence. The quality control samples are matrix spikes which contain known levels of analyte and are extracted with the samples.

Formula 11-1

$$RRF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where

RRF = *the relative response factor for the analyte*
A_{is} = *integrated area or ion current of the internal standard*
A_s = *integrated area or ion current of the analyte*
C_{is} = *amount of the internal standard*
C_s = *amount of the analyte*

Formula 11-2

$$\overline{RRF} = \frac{1}{n}\sum_{i}^{n}\frac{A_s C_{is}}{A_{is} C_s}$$

where: $\overline{RRF}$ = *the mean value of the relative response factors for the analyte*
n = *the total number of data points derived from the initial calibration*
A_{is}, A_s, C_{is} *and* C_s *have the same meaning as in formula 11-1.*

Formula 11-3

$$\%D = \frac{RRF_{cc} - \overline{RRF}}{\overline{RRF}} \times 100$$

where:

$\overline{RRF}$ = *mean relative response factor for the analyte in the initial calibration*
RRF_{cc} = *relative response factor for the analyte from the continuing calibration*

Gas Chromatography/Electron Capture Detector (GC/ECD)

Initial Calibration

Internal standard calibration is utilized for the analysis of pesticides amd PCBs by GC/ECD. The method-specified number of calibration standards are be used. Each solution is analyzed once and the analyte relative response factors are calculated using the expression in Formula 11-1. The mean relative response factor for each analyte is then obtained by using the expression in formula 11-2. Integrated areas are utilized for these expressions. For multiple response pesticides/PCB's, quantitation consists of an average of the quantitated values for five selected peaks, if possible. The percent relative standard deviation (% RSD) must be less than ±20% in order to use the mean relative response factor for quantitation. If it is greater than ±20%, one more attempt is made to meet criteria. If the second attempt is unsuccessful, the analyst takes corrective action, such as instrument maintenance, and begins the sequence again.

Atomic Absorption Spectrophotometry (AA)

An initial calibration is performed daily with freshly prepared working standards. A four-point calibration curve is acquired which must have a correlation coefficient of 0.995 or better. The initial calibration is verified every 10 samples or 2 hours, whichever is more frequent. The continuing calibration is required to be within 10% or 20%, depending on the analytical method utilized. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 10% of the true value.

Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed every 8 hours and continuing calibrations are performed every 10 samples or 2 hours, whichever is more frequent. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 10% of the true value. The continuing calibration is required to meet the criteria of the analytical method.

Ion Chromatography (IC)

The ion chromatograph is typically calibrated by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. Procedures for verifying the calibration curve are method specific.

AOX/TOX Instrumentation

Instrumentation for the determination of AOX/TOX consists of a column adsorption module, titration cell and combustion/microcoulometric system. Several system performance tests are conducted and must meet acceptance criteria prior to sample analysis. The following performance tests are typically conducted, with slight variations between the different analytical methods. Granular activated carbon utilized in the column adsorption module is tested for purity. The titration cell is tested and adjusted based on the results of an injection of sodium chloride solution. Calibration of the combustion/microcoulometric system is accomplished through the analysis of 2,4,6-trichlorophenol. Verification of system performance and calibration is performed during sample testing according to specifications in the analytical methods.

Sample Analysis Procedures

Techniques for quantitative analysis of samples are specific to the analytical methods and sample matrices. Samples may either be subjected to a series of preparation steps prior to instrumental analysis, or they may be ready for analysis upon arrival at Triangle Labs. Most samples must be analyzed within a defined period of time following their collection, receipt at the lab and/or preparation. These analysis holding times are complied with to the extent possible (samples are occasionally received near or beyond the expiration date of holding time). Holding times for most methods employed at Triangle Labs may be found in Appendix 4.

After sample analysis is completed and the data is processed, the analyst reviews the resultant data. If established acceptance criteria are not met, corrective action is taken to resolve problems. Once all the samples in a project have been analyzed and the data have met the criteria, the project documentation (instructions, raw data, reports, etc.) is sent to the next stage for preparation of the final report.

Section 12

DATA HANDLING AND SOFTWARE MANAGEMENT

Data Collection and Reduction

Quality assurance principles are applied in the acquisition of raw data related to chemical measurements. Raw data is "primary data" which will be used to generate "secondary" data (the final analytical report). Data can be acquired manually or electronically. Manually acquired data is hand written on data sheets and in logbooks. Electronically acquired data is acquired from an instrument and instrument/computer interface. Specific definitions and data requirements are detailed in the Raw Data SOP.

Manually Acquired Data

Manually acquired data: is recorded on data sheets or in notebooks. The data must be recorded immediately by the analyst in permanent ink. Each entry must be signed and dated immediately after entry. Corrections must not obscure any original entries. Corrections are made by canceling with one line through the original. Each correction must be dated and initialed by the person who made the correction and a reason for the correction must be stated. Data sheets are standardized, preprinted forms which are subject to document control. Data sheets may be bound into a book or may be used as loose sheets depending on the application. Notebooks are bound, consecutively numbered, and subject to a controlled distribution and archival system.

Electronic Data

Electronically produced data may consist of chromatograms, spectra, data printouts, and raw quantitation reports. The first accepted hard copy report constitutes the raw data for each sample and calibration. Acceptance is signified by the dated signature of the analyst. The accepted hard copy report must contain the full sample ID or calibration name, file name, as well as date and time of acquisition. In the case of inorganic data, all replicate and dilution data is included in the documentation. Any changes to the raw data hard copies and computer files must be fully documented and clearly attributable to the person making such alterations (e.g., manual integrations are hard-copied for inclusion in the raw data file, with area changes fully documented on the data printouts). No ambiguity in data system printouts as to what peak on a chromatogram corresponds to an analyte of interest is allowed. Computer-collected data is reduced to hard copy as soon as possible. The signed and dated hard-copies of the data files are retained in the project file and are maintained for a minimum of 10 years. The electronic files are safeguarded by a system of disk storage and backup disks to protect loss of data and programs.

There are several different means of data collection, review and reduction, which are dependent upon specific methodology and instrumentation. Data review and reduction of pharmaceutical data normally consists of data acquisition via a dedicated computer with further reduction and data reporting utilizing validated spreadsheets. Regression and sample calculations are verified independently for each pharmaceutical data set.

Data review and reduction of environmental analyses normally follow the guidelines of relevant EPA reference methods to the extent possible. For HRGC/HRMS analyses, established procedures consist of data acquisition and reduction on a Digital Micro VAX and VAX 3100 and further reduction and data reporting using dBase software on a PC. For HRGC/LRMS analyses, established procedures consist of data acquisition and reduction using PC-based software or a PDP-11/24 system followed by further data reduction and reporting using dBase software. For HRGC analyses, established procedures consist of data acquisition and reduction using PC-based software followed by further data reduction and reporting using dBase software. For AOX/TOX analyses, manual data acquisition from instrument panel readings is followed by data reduction and reporting using spreadsheet software.

All GC, GC/MS, and inorganic data go through several levels of review and inspection, starting with an initial examination in the Instrumentation area, followed by a thorough review before preparation of the report. After preparation of a report, an independent review is performed by a chemist other than the one who prepared the report. At each stage of the analytical process, data are reviewed for completeness, adherence to protocol requirements, and credibility. Results are fully validated, possible compromises of data quality are evaluated, and deviations from protocol requirements are documented. To the greatest extent possible, computer programs are utilized for data reduction. Where manual data procedures are required, data review is performed according to standard operating procedures. This ensures that the results are as independent of the chemist performing the duties as possible. Corrective actions are implemented at the earliest possible opportunity.

Data Validation

The tests performed by Triangle Labs typically involve the performance of complex chemical analyses by a number of chemists. For this reason data validation and coordination are very important. At the conclusion of the analyses, data are checked against the original shipping information and analytical request to be sure that the required analyses have been performed on all samples.

The validity of the data are verified through the analysis of blank samples, duplicate samples and laboratory control or matrix spikes. The blank sample results demonstrate the absence of laboratory contamination of the samples. Duplicate analyses give a measure of analytical precision. The analysis of spike samples permits a measure of accuracy. Data for these QC samples are reviewed as soon as possible after analysis. For example, in the inorganic area, a data quality checklist is used by the instrument operator at the time of analysis, to verify that all

calibration verifications are within tolerance, and that other QC indicators such as spike recoveries and blanks, are acceptable for a project.

Data Reporting

The data are reported as components identified and the quantities present. The final report includes example calculations and descriptions of the equipment and procedures utilized. Complete data packages of all raw sample and calibration data are prepared and archived. These are furnished to the client upon request. Sample flagging procedures for HRGC/HRMS analyses are summarized in the final report.. While sample flagging is not done directly on most HRGC/LRMS analytical reports, problematic results are discussed in the case narrative which accompanies each data package. Several standard report formats are used in the inorganic area, tailored to the data structure for the specific project type (e.g., TCLP, Multi-Metals Train or CLP).

Data Package Delivery

Data packages are prepared for delivery by the Shipping and Archive department according to their SOP's. Unless otherwise requested by the client, a copy of the data package is shipped, while the original is retained in a secured archive facility. Reports are fully paginated prior to copying. The data packages are packed to meet the requirements of the commercial carrier chosen for delivery. Packages are delivered by a commercial carrier whose procedures for protecting the data packages are not within the control of Triangle Labs. Should the shipped data package be lost or damaged during delivery, a copy can be quickly prepared as a replacement. Clients are made aware that a commercial carrier will deliver their data packages.

Corrections and Additions to Documentation

The policy for handling additions/corrections of reports already issued is as follows. The Project Scientist requests an addition/correction in writing to the appropriate data review/report preparation personnel, who make the requested change in a timely manner and internally verify the change. An authorized Chemist reviews and approves the addition/correction, and the Data Package Assembly Department mails or faxes the new report, which is then stored with the original data package for a minimum of ten years. In all cases, revised pages are clearly noted as such, as are additional pages added to the report.

Software Management

Triangle Labs has a formal validation program of its computer systems. Ultimately, the validation program is intended to be of a level such that all computer systems will meet the scope of any computer system audit. The validation approach is three pronged. First, new software is developed according to appropriate internal validation guidelines. Second, a validation committee has been appointed to oversee specific validation efforts of existing systems. Finally, systems are kept validated through a system of change controls. This includes the *Computer Systems Services Request* (CSSR) forms which employees use to make known to the MIS department, desired changes to software and hardware. CSSR forms include personnel sign-off for each step

of the change process; and depending on the nature of the change, specify increasingly stringent required levels of authorization. Change controls also include software version control; changes to existing software are announced, uniquely labeled, documented, and old versions are archived for future reference.

The goals of the software development methodology, existing system validations, and the change control system are to ensure that the software systems perform the required functions accurately, that the users understand how to use the system, and that auditors can assure themselves of the validity of the analytical methods utilized. This in turn insures the ability to deliver accurate analyses in a timely fashion.

Section 13

DOCUMENTATION FOR QUALITY ASSURANCE

Objectives of Documentation

The objectives of documentation for quality assurance are: to provide a standardized, written program of policies, procedures and instructions; to demonstrate that adequate quality assurance and quality control procedures have been implemented; to demonstrate that accountability of the data is maintained; and to ensure traceability of analytical results.

Document Control

The laboratory maintains control over the possession and distribution of documents that directly impact the quality of a product or service. It is the responsibility of team leaders to ensure that document control files are created and maintained for all applicable documents originating in their areas. This includes documents such as the Quality Assurance Manual, Standard Operating Procedures (SOP's), Work Area Guidelines (WAGs), Quality Assurance Project Plans (QAPP's), and client instructions. It also includes standard forms, such as laboratory bench sheets, project communication forms, and corrective action reports.

A written procedure describes document control practices. Full or limited document control is applied, depending upon the purpose of the document. Those publications which document the quality assurance system at Triangle Labs, specifically the QA Manual and Standard Operating Procedures, are subject to full document control practices. Limited document control procedures are employed for other relevant documents, such as forms and flow charts . The procedure for limited document control allows for the retention of a previous version for historical information and purposes.

Every controlled document is assigned a unique identification (usually a title, file ID and creation/revision date) which must be present on each page of the document. This unique identification is entered on a master list of documents, along with a distribution list for each document to ensure that pertinent documents are made available wherever they are essential. A master set of current documents is maintained along with the master list. The status of each document, active/current or inactive/obsolete is indicated on the master list. Each document and any subsequent revisions must be reviewed and approved by authorized personnel prior to issue. Personnel authorized to review and approve a document are to have access to all necessary information on which to base their review and approval. Obsolete documents are to be retrieved from distribution points and replaced with current versions.

Standard Operating Procedures (SOP's)

Standard Operating Procedures (SOP's) are controlled documents in which instructions for standard operations performed by the laboratory are detailed. The author of an SOP should be the person most familiar with the topic being addressed. The standard format for writing SOP's is fully described in the SOP on SOPs. Each SOP is reviewed by senior level staff and authorized by management prior to distribution.

It is important that SOP's receive evaluation and input by laboratory supervisors and key technical personnel. The content of each SOPs must conform to applicable requirements of analytical methods and certification agencies, and be consistent with the Good Laboratory Practice standards. Within these constraints, the content of an SOP may be customized to meet the needs of a particular area of the laboratory. The performance of laboratory operations is subject to audit for compliance with written SOP's. If an SOP is impractical, hard to follow, or no longer meets laboratory needs, it must be modified or replaced.

The need for new or revised SOP's can be determined when a new method is implemented, when the scope of the existing method is extended or when some activities are being performed without adequate SOP's. Such a need can be identified by the analyst involved in the production or by someone from management. Also, the QA Department may identify the need and request new or revised SOP's, usually as a corrective action for deficiencies found during an internal inspection. SOP's are created to provide a clear, concise, description of the procedure with explanatory information to enable a person with the appropriate background to perform the procedure. Revisions are made to SOP's as necessary to reflect changes in procedures.

While team leaders are responsible for the operating SOPs, the administrative staff assists with the typing on an as-needed basis. Once technical approval is obtained for a new or revised SOP, the SOP is reviewed by the Quality Assurance Department for compliance with all requirements. The Quality Assurance Department also maintains a database of SOP distribution and version status, as well as maintaining the original copies of each active SOP and the historical files of each revision. The administrative staff distributes copies of the authorized SOPs to area SOP coordinators according to the distribution plan contained in the SOP database. The area SOP coordinator is responsible for discarding copies of obsolete SOPs upon receipt of revisions. Team leaders are responsible for training staff in all applicable new or revised SOPs.

Work Area Guidelines

WAGs are supplements to the SOPs and as such contain additional detail and guidance. Work Area Guidelines (WAGs) are training documents which entail step-by-step instructions for specific tasks. The WAGs are comprised almost entirely of proprietary information and are restricted to use by Triangle Labs employees. These documents cannot be distributed to clients or other non-employees.

Quality Records

Quality records must be maintained to prove that the quality assurance system is being effectively applied. At Triangle Labs, specific procedures for the identification, collection, indexing, filing, storage, maintenance, and disposition of various quality records are described in several SOP's. All quality records must be recorded in permanent (indelible) ink, legible, attributable to those personnel who wrote them, and protected so they may not be adversely affected by an unsuitable environment. They are stored and maintained in a manner that facilitates rapid retrieval for a period of at least ten years after completion. With the exception of internal audit reports, project specific quality records are available for evaluation by the client or his representative during the archive period of ten years. In fact, certain quality records, as specified by SOP or contract, are delivered to the client with the final product.

Project specific quality records are maintained to prove that adequate quality control procedures are being implemented, accountability of the project data is maintained, and traceability of analytical results is facilitated. Accountability means that reported data reflect the sample as it was received, that sample mix-up was avoided, and the sample was properly preserved after receipt. Traceability means that reported data may be reconstructed at a later date. Through proper documentation, a laboratory is able to demonstrate or prove to clients or government agencies that the quality of the data is what the laboratory says it is. Records must contain sufficient information to permit the reconstruction of calibrations, sample preparations and sample analyses.

Quality records that are maintained at Triangle Labs include, but are not limited to, the following.

- records for sample receipt, preparation and handling
- field sample and quality control sample analysis data
- project communication tracking forms
- inspection reports for receiving, in-process and final product
- subcontractor records
- vendor qualification records
- logbooks: run logs, maintenance logs, temperature logs, balance logs, etc.
- method validation records: MDL studies, initial precision and accuracy demonstrations
- recovery data for samples, blanks and spiked samples (maintained in a database)
- system and data audit reports
- corrective action reports
- QA reports to management

Many of these quality records are discussed at length in other sections of this manual. Laboratory notebooks (or "logbooks") are utilized throughout Triangle Labs for many different purposes. All logbooks are maintained according to written procedures. New logbooks are issued by a system of signing them out in a designated logbook. Information that must be documented, both in the new logbook and the sign-out logbook, includes the assigned owner, the date issued, and the name and subject of the logbook. Logbooks must be maintained in accordance with the raw data SOP. Logbooks are kept to document all monitoring, maintenance and calibration of analytical instrumentation, and such laboratory equipment as balances, refrigerators and ovens. Software and hardware records for computers are also kept in logbooks. Logbooks specific to a piece of equipment are kept near that equipment to ensure that the work is recorded concurrently. Logbooks used for personal notes and telephone logs are distributed and tracked in the same

manner as laboratory notebooks. When no longer in use, distributed notebooks are sto n Archive Room for a minimum of ten years.

Archive

The Archive Room is locked at all times and only trained, designated staff have access. All oth personnel may enter the room only in the presence of a trained Archivist and must sign and da a logbook in the Archive Room. Any materials removed from the Archive Room must be signed out by the Archivist.

All magnetic and hard copies of data, calibrations, equipment maintenance records, calculation records of original observations, final test results and any other miscellaneous quality records directly associated with sample analyses are stored in a secured facility for a minimum ten (10) years after completion of a project. They may be stored in the Archive Room or at a secure, off site storage facility.

Section 14

QUALITY ASSURANCE

Through a formal quality assurance system, Triangle Laboratories, Inc. is able to prove that products and services meet specific quality standards. These quality standards are defined to meet the needs and requirements of our clients, the analytical methods utilized, government agencies, and senior management of Triangle Labs.

Quality assurance is a very broad and multifaceted concept. It is composed of quality control and quality assessment. Quality control is the most important component of quality assurance. The need for quality assessment would be negligible if the laboratory always achieved perfect quality control.

> *Quality control* is a system of activities applied at each stage of the production process. Its purpose is to assure that products meet defined quality standards. This system includes the following: employee education, training, and experience; documentation (e.g., instructions, document control, records); instrument calibration and maintenance; laboratory accommodations; and inspection.
>
> *Quality assessment* is a system of activities employed to assure that quality control takes place at each stage of the production process. This system includes the following: system, data, and performance audits; reference materials; statistical evaluations; retests; and measurement bias investigation (when measurements may be operator-, instrument-, or methodology-dependent).

The success of a quality assurance system is dependent upon acknowledgment by all personnel of their responsibility for the system. The management of the laboratory is ultimately accountable for product quality, but no one person or group (e.g., the QA Department) is responsible for the greater part of quality assurance program activities. Details of the program may be found throughout this QA manual. The remainder of Section 14 will be limited to a discussion of the Quality Assurance Department, and the major activities performed and/or administered by this group.

The Quality Assurance Department

At Triangle Labs, the QA Department monitors the quality assurance system, as it is implemented throughout the laboratory, and reports the results of its observations to senior management. The Quality Assurance Officer reports directly to the President and the QA Department has no direct responsibility for production in the laboratory. The objective of this independence is to eliminate conflicts of interest in the performance of QA duties. Major activities performed and/or administered by the QA Department are summarized below. Each activity is discussed in greater detail elsewhere in the QA manual, as indicated.

- Performance of internal audits and coordination of external audits (see this section)
- Administration of a system for formal Corrective Action Reports (see this section and Section 15)
- Performance of Quality Assurance Unit (QAU) duties required for GLP-regulated studies (see this section)
- Administration of the system for document control, with emphasis on the maintenance of Standard Operating Procedures (see Section 13)
- Performance of statistical evaluations for selected quality indicators, and maintenance of quality records (e.g., control charts, summary reports) generated to document selected statistical evaluations performed throughout the laboratory (see Section 15)
- Publication of the QA Manual and other documents that describe the quality assurance system at Triangle Labs (see Section 3)

Audits and Inspections

There are several different types of audits. These may be internal, in which the laboratory reviews and examines itself, or external, in which the laboratory is audited by outside organizations, such as accrediting or regulatory agencies and clients.

Internal System Audits and Phase Inspections

A system audit is an on-site inspection and review of the quality assurance system as it is employed in the laboratory. During an audit, verification may be sought that: adequate written instructions are available for use; that analytical practices performed in the laboratory are consistent with SOP's; that adequate quality control practices are applied during production; that corrective actions are applied as necessary; that deviations from approved protocols are occurring only with proper authorization and documentation; that SOP's, quality records, analytical records, magnetic tape, etc., are properly maintained; and that personnel training records are satisfactory and current.

Internal system audits are implemented by the Quality Assurance Department to assess the functioning of one or more department(s) of the laboratory. These audits consist of real time inspections of the analytical process, comparing the daily operation to the applicable SOPs and policies. Formal inspection reports are issued detailing the extent of the inspection and any non-conformance issues noted. The production staff is required to correct all noted deficiencies and a second acceptable inspection is required for acceptance of the corrections.

Inspection reports may be routed to management at any point in the process depending on the severity of the problem. Major problemsare reported to management immediately while minor ones are normally communicated in a summary report dealing with several inspections. The original of each completed

inspection report, with management notification dates, is kept on file in the QA files.

Phase inspections are internal system audits that are used to verify that critical points of analysis during a pre-clinical or clincal study are being performed as specified in the applicable SOP. These inspections are performed at intervals adequate to assure the integrity of the study.

External System Audits

Representatives of clients, government agencies, and accrediting agencies frequently perform system audits of Triangle Labs. These audits are usually announced inspections, but sometimes are conducted without forewarning. QA Department personnel usually accompany such audit teams through the lab. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors typically provide an account of their findings shortly after the audit. This account is evaluated by QA personnel and reported to management, along with recommendations for actions in response to any cited deficiencies.

Data Audits

Data audits are performed by technical personnel (in Client Services or the QA Department) on a random sampling of the data reports produced at Triangle Labs. It is a goal to perform a comprehensive evaluation of a representative sampling of data reports. A data report is carefully evaluated for technical, clerical and administrative accuracy. Primary emphasis is placed on the ability of the data report to meet customer requirements. Data audits are utilized for several purposes, including: identification of opportunities for process improvement, evaluation of the efficiency of the system, detection of inadequate execution of quality control procedures, early warning of potential system deficiencies, corrective action recommendations, and reports to upper level management.

Performance Audits

A performance audit is the analysis of a fortified blank sample, for the purpose of evaluating laboratory or analyst performance. There are several examples of performance audits, which may be of internal or external origin. Performance Evaluation (PE) samples have analyte concentrations unknown to Triangle Labs, and are submitted by external organizations. PE's may be analyzed as part of multi-laboratory round robin studies, in conjunction with accreditation programs, or as blind check samples submitted by clients. Internal performance audits are fortified blanks with known analyte concentrations, the values of which may or may be known to the analyst. Examples of internal performance audits include initial precision and accuracy studies, QC check samples, laboratory control samples, and blind samples. The results of performance audits are utilized for several purposes other than the evaluation of laboratory performance, including: to fulfill accreditation requirements, to serve as analyst proficiency tests, and to facilitate laboratory improvement efforts.

Non-Conformance Reports

All instances of failure to comply with acceptance criteria are documented in a non-conformance report (NCR). Each report contains a description of the "failure", details of the resulting investigation, and the determined impact on the associated sample(s). A summary of these NCR reports is reviewed by the Production Manager, the Technical Director and the Quality Assurance staff. NCRs are maintained as part of the raw data file of the project. A non-conformance issue may be caused by a particular sample independent of the analytical process or it may have been caused by a faulty analytical process with minimal adverse impact on the particular samples. The staff at Triangle strives to identify both types of situations and deal with them accordingly.

Corrective Action Reports

All major non-routine problems, deficiencies, or irregularities must be reported to management. A formal Corrective Action Report (CAR) system, administered by the QA Department, is in place at Triangle Labs. The QA Department issues CAR forms, monitors the progress of corrective actions, maintains completed documentation, and provides reports to senior management on the status of formal corrective action activities. CAR's may be originated by anyone responsible for the quality of a product. A completed form is sent to an appropriate person or group to whom responsibility for corrective action is assigned. One person is designated the Corrective Action Analyst. This person records the corrective action plans, implementations and follow-up actions completed by the responsible person(s). During the corrective action process, several measures may be taken. These include: determination of the root cause through careful analysis of processes, specifications, quality records, customer complaints, etc., using statistical process control when applicable; implementation of measures that prevent recurrence of the problem; implementation of process controls to ensure that effective corrective action is taken; application of remedial actions to products affected by the identified problem; and revision of documentation for procedures that have undergone change as a result of corrective action.

Certification and Accreditation

Triangle Labs has been granted numerous certifications and accreditations, based upon compliance with standards set forth by the granting agencies. These credentials have enabled Triangle Labs to expand and retain a substantial client base. More information about specific credentials can be found in Section 5, page 3. The nature of the quality assurance program implemented at Triangle Labs is profoundly affected by requirements of certification agencies. The administrative staff is responsible for the administration application and renewal activities associated with the various certification programs, while the QA Department is responsible for the coordination of the technical and quality issues associated with the certification programs. The QA Department is directly responsible for the coordination of:

- On-site audits by outside agencies
- Analysis of blind performance evaluation (PE) samples
- Responses to deficiencies cited in audit reports and performance evaluation results.

- Dissemination of requirements and status of certifications to relevant laboratory personnel.

GLP Regulated Studies

The Good Laboratory Practices (GLP's) are a set of regulations decreed by the United States Food and Drug Administration (FDA) and the United States Environmental Protection Agency (EPA). Compliance with these regulations is required for certain projects ("studies") completed at Triangle Labs. The GLP's define some specific responsibilities for the Quality Assurance Department. Briefly summarized, these QAU duties include the following:

- Maintenance of a copy of the master schedule sheet for all studies
- Maintenance of copies of all protocols pertaining to all studies
- Inspection of each study at adequate intervals
- Preparation of written status reports on each study with reports to management and the study director
- Determination that no deviations from approved protocols or SOP's were made without proper authorization and documentation
- Review of the final study report
- Preparation of a signed statement of the inspections performed and the dates each was reported to management for inclusion in the final study report

Section 15

QUALITY CONTROL

At Triangle Labs, quality control is achieved through the application of several procedures. Quality control activities commence before production is initiated, and are implemented at each stage of the production process. The purpose of these activities is to assure that all required standards of quality are met. Quality control activities are described in many sections of this manual. The remainder of this section will describe a subset of quality control activities that may be considered a discrete process, summarized as follows:

Prior to the initiation of production activities, required quality standards are defined. These are derived from several sources, including: requirements of the analytical methods, needs stated by the clients, and standards established within Triangle Labs.

During production, verification activities are performed to determine that defined quality standards have been met. Also, preventive measures are applied to avoid the possibility of nonconformity.

When defined quality standards have not been met (nonconformities), corrective actions are applied and verified to determine that the results meet requirements.

Data Quality Objectives

Data are produced for clients at Triangle Labs. Defined quality standards for these data may be expressed as data quality objectives (DQO's). These are established prior to sample preparation and analysis. Quality assurance indicators common to all DQO's include, but are not limited to: accuracy, precision, completeness, representativeness, and comparability. Examination of the QA indicators is performed to demonstrate that the data are scientifically valid, legally defensible and that they adequately meet established DQO's. The QA indicators may be summarized as follows:

Accuracy A quantitative measure of the relationship of reported data compared to the "true" or expected values. This measurement may be accomplished by evaluation of the recoveries of analytes spiked into samples. Specific accuracy measurement activities include surrogate spikes, matrix spikes and Quality Control Check Samples. .

Precision A quantitative measure of the reproducibility of measurements made under controlled conditions. This measurement may be accomplished by comparison of recoveries of analytes in replicate samples or injections. These analytes may be spiked or native to the duplicate samples. Specific precision measurement activities may include field replicates, lab replicates, matrix spike replicates and replicate injections

Completeness A qualitative measure of the amount of valid data obtained from the analytical process compared to the amount that was expected to be obtained. Valid data must meet all data quality objectives for precision and accuracy.

Representativeness A qualitative measure of the degree to which data represents the characteristics of the population from which samples were collected. This is usually dependent upon sampling techniques not controlled by the analytical laboratory. However, the issue of the representativeness of subsamples prepared within the laboratory is addressed by thorough homogenization prior to subsampling.

Comparability A qualitative measure of the confidence with which one set of data can be compared to another. Characteristics that make comparison possible include standardized report format, consistency of units (e.g., mg/L, ppm), and standardized sample preparation and analysis.

Quality Control Samples and Spikes

Analytical performance is monitored through quality control samples and spikes, such as laboratory method blanks, surrogate spikes, quality control check samples, matrix spikes, matrix spike duplicates, duplicate samples and duplicate injections. Many of these quality control measures, as applied at Triangle Labs, are summarized below.

Laboratory Method Blank A laboratory method blank consists of a sample that is processed in a manner identical to that of a regular sample, except that the matrix is replaced with distilled water for aqueous matrices, sodium sulfate for solid matrices, XAD-2 resin for MM-5 and PUF filter for PUF air sampling cartridges. The laboratory method blank sample is fortified and prepared along with the field samples, at a frequency of one laboratory method blank per batch of 20 (or less) samples of a given matrix type. The laboratory method blank serves to demonstrate a contamination free environment in the laboratory.

Surrogate Standards For certain methods, all samples, including the laboratory method blank, are spiked with a set of specific surrogate standards to monitor accuracy of the analytical determination for each particular sample. QC criteria for surrogate recoveries are method and matrix specific. Typically, laboratory QC criteria are established upon acquisition of a sufficient number of data points (20 or more) and used for evaluation of sets of data via control charts, while method specified limits are utilized for individual samples.

Quality Control Check Sample A quality control check sample consists of a blank matrix sample which is fortified not only with appropriate internal and/or surrogate standards, but also with target analytes. QC check samples are analyzed at a frequency dependent on the method. They serve as an estimation of system precision and accuracy. Results of QC check samples are monitored on control charts, with QC requirements for recoveries being established as they are for surrogate recoveries.

Matrix Spike Sample

A matrix spike (MS) sample consists of a field sample, identified by the client, that is split into two parts and processed in a manner identical to that of the rest of the field samples. However, in addition to the regular fortification with the standards (internal, surrogate and/or alternate), the chemist will add a set of the target analytes to one part of the chosen sample before the preparation. The fortification levels for the target analytes are defined by the analytical method or the client's request. At the request of the client, one such sample will be prepared for every batch of 20 samples (or less) for a given matrix. To be able to run matrix spikes, the client must provide Triangle Labs with extra sample amounts.

The analytical report for the matrix spike will contain a tabulation of the analyte concentrations as expected and as measured, along with the calculated percent recoveries based on the expected concentrations. The percent recoveries actually represent a measurement of the method accuracy for that particular sample and matrix. Accuracy is established and updated for a particular analyte and method. In the absence of observable quantitative interferences, the MS sample showing accuracies falling outside the QC limits must be reanalyzed unless the matrix spike duplicate (MSD), which was processed along with the MS, shows similar deviations as a result of a "matrix effect." This type of corrective action can only be implemented if the sample selected for the MS (and MSD) was proven to be free of the target analytes, or did not contain high concentrations that significantly exceed the MS fortification level of these analytes. "Matrix effect" is further substantiated by acceptable recoveries in a QC check sample processed along with the field samples. Matrix spike recoveries, and the possible effects on data quality when accuracies fall outside the QC limits, are discussed in the Case Narrative.

Matrix Spike Duplicate Sample

The matrix spike duplicate (MSD) sample is commonly prepared (at the Client's request) in conjunction with the matrix spike (MS) sample. The analytical report will summarize the data from the MS and MSD analyses in a format allowing determination of the precision of the analyses. As for the matrix spike sample, the client must provide Triangle Labs with extra sample amounts.

Duplicate Sample

A duplicate sample (DUP) consists of a set of two identical samples obtained during a single sampling session. At the client's request one such sample per batch of 20 samples (or as specified by the client) per matrix type will be analyzed, provided the client supplies Triangle Labs with the necessary samples.

The analytical report for the duplicate analyses will contain a tabulation of the results showing the precision as relative percent difference (RPD). Precision exceeding any specified target values will necessitate a non-conformance report and an evaluation of the associated data. The influence of the sampling procedure will be included in the data evaluation. The RPD is calculated as:

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

where: RPD = the relative percent difference

$X_i(i=1,2)$ = the analyte concentration in the original sample (1) and the duplicate sample (2)

Duplicate Injection Upon client request, a duplicate injection of a single sample extract will be performed. In the absence of observable interferences, the RPD is expected to be within ± 30% or the injections will be repeated after identification of the cause of the poor precision. Field samples analyzed during a suspected out-of-control situation will be reinjected as well.

Statistical Evaluation

Statistical evaluations can be made of selected analytical quality indicators, including spike recoveries, calibration responses, contamination levels, and method detection limits. Production units monitor levels of compliance with many criteria on a "real time" basis. Control charts are used to identify shifts in the analytical process. All identified performance shifts are investigated and causes of adverse shifts are eliminated. Causes of positive shifts are also identified and incorporated in SOPs and staff training as applicable. In-house QC criteria may be determined through historical trend analysis of data collected on QC charts. Statistical evaluations can be performed by both the QA department and production units.

QC Inspection

Quality control inspections are built into the production process. These inspections consist of peer review at each step of the process the ensure compliance with process and product specifications. Acceptance criteria are included in the production SOP's. Written documentation of the analytical process is maintained beginning with sample receipt and preparation, through instrument calibration, sample analysis, data review and report preparation. This documentation is reviewed for completeness, compliance with written procedures and consistence with client documentation.

Written records of all QC inspections are required indicating the date, inspector and results of the inspection. Detected nonconformances must be recorded during the inspection. Corrective action must be taken and documented whenever nonconformance is detected. The identity of the inspection authority responsible for releasing the product is documented in the inspection records. Until required inspections are performed on the intermediate and final product, it is not permitted to progress further along the production process, except by special, documented, client request.

In-process Inspection and Testing

Each department is responsible for a segment of the production process and for all in-process inspection and testing that takes place within the department. In-process inspection is accomplished through 100% screening for all areas. Each client sample that goes through the analytical process is unique and can be considered a separate lot.

Final Inspection and Testing

The last stage of the production process is the preparation of a final data report. This requires a thorough review of all records generated for a client sample set since its receipt, including inspection records and any client documentation that may have originated before sample receipt. A chemist performs this function during the preparation of the data package. This inspection serves as both an in-process and final inspection of the product. In addition, a second chemist performs another final inspection of the data package and quality records. As in any other part of the process, any nonconformances found during these inspections must be documented and corrected before the data package is released. Approval of the data package for release to the client is indicated by the signatures of the chemists on the case narrative.

Nonconformity

Each field sample that is incorporated into the analytical process is unique. Laboratory procedures are designed to introduce as much standardization as possible. Whenever conformance to standards is uncertain, the product is reviewed to determine the nature and cause of nonconformance. If it is judged to be nonconforming due to the unique nature of a sample, there may be little recourse other than to inform the client and discuss the options that are available.

Each case of failure to comply with written acceptance criteria must be recorded in a non-conformance report (NCR). The failure must be recorded by the person who detected or observed it. All investigative efforts are recorded on the NCR with an evaluation of the impact the non-conformance had on the associated samples. Impact on the analytical process is also noted. If needed, recommendations for corrective action are made. A copy of the NCR is kept with the project data. Rework and reanalysis is subject to the same inspection procedures as the initial work. Nonconformity, its review, and its disposition must be documented in the quality records as prescribed by the written procedures.

Corrective and Preventive Action

Appropriate actions must be taken to prevent or correct nonconformities in products and problems in analytical systems. When actions result in permanent procedural changes, pertinent documentation (e.g., SOP's) must also be modified to reflect these changes. Cost-effective preventive measures are applied whenever possible. In specific cases, the cost of applying preventive measures would exceed the cost of applying routine corrective actions. Because every client sample possesses unique and unknown properties, some predisposition to unpredictable, unpreventable nonconformities exists.

Corrective Action

Specific corrective actions are of two types: routine corrective actions applied to solve minor or commonplace problems, and formal corrective actions taken to eliminate major or non-routine problems.

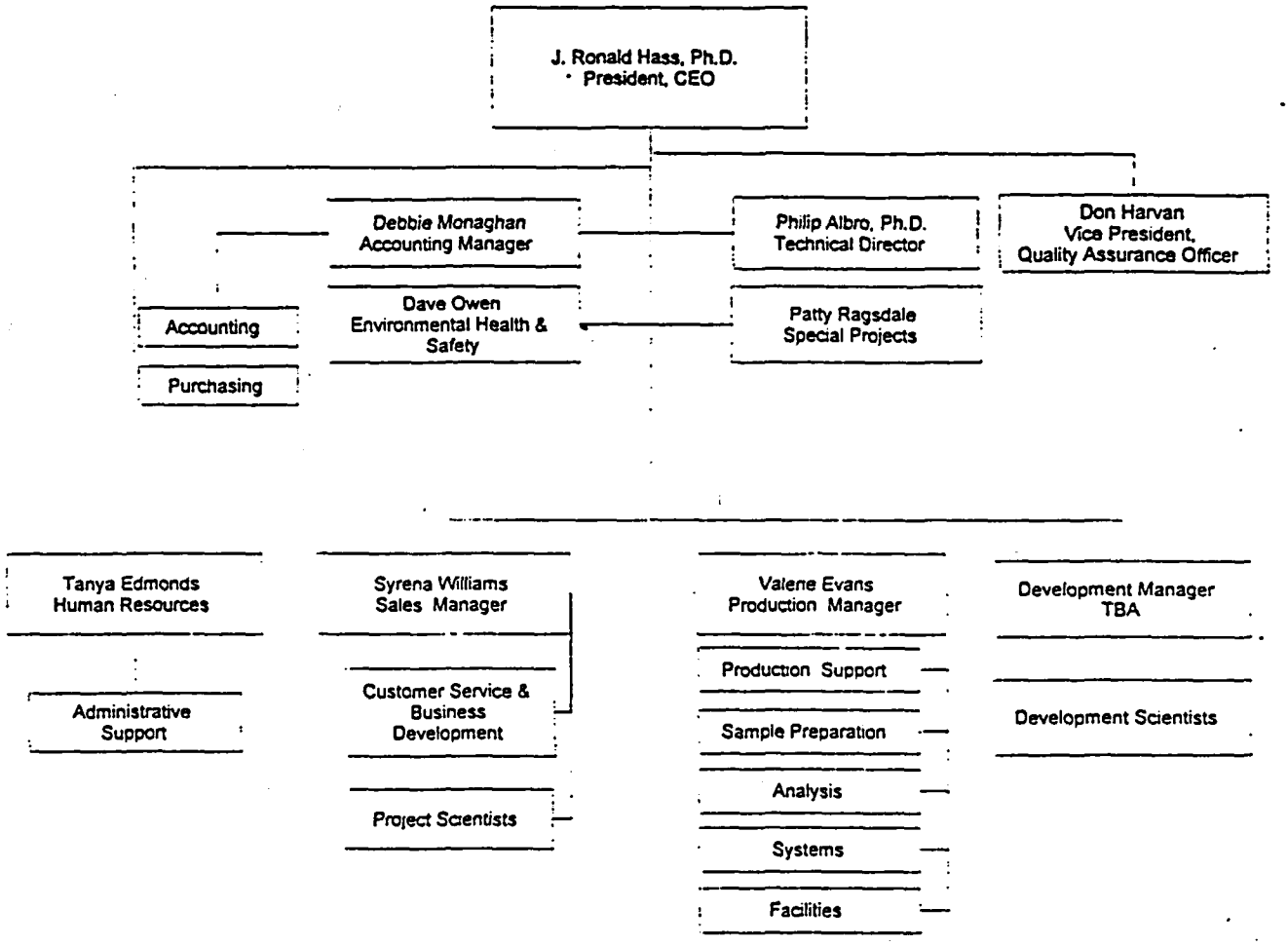
Routine corrective actions are usually made by the chemists, technicians or instrument operators who detect minor problems or product nonconformances. These actions are taken in response to observed non-conformance issues are recorded on the associated NCRs.

There are three procedures for conducting formal corrective actions. The first is corrective action in response to a system audit report from the Quality Assurance Unit. This procedure is more thoroughly described in Section 14. The second procedure is the formal Corrective Action Report, which may be initiated by anyone who detects a significant quality problem. This procedure is also administered by the Quality Assurance Unit. Further information about it can be found in Section 14. The third practice is described in a written procedure on "Problem Sample Communication." It is initiated in response to client complaints about specific projects.

Preventive Action

Preventive actions are implemented as part of standard operating procedures, process improvement efforts and corrective actions. When circumstances inherent to a procedure are known to have a high potential for error, the SOP must define measures to prevent the error from occurring. Preventive actions are an integral part of corrective actions, because resultant changes in procedures often prevent recurrence of problems.

ORGANIZATIONAL CHART



Appendix 2A
VOLATILE COMPOUNDS SW-846 Method 8260B

Pentafluorobenzene (Internal standard)
Acetone
Acrylonitrile
Allyl chloride
Bromomethane
Bromochloromethane
Carbon disulfide
Chloroethane
Chloroform
Chloromethane
cis-1,2-Dichloroethene
1,1-Dichloroethane
1,1-Dichloroethene
1,2-Dichloroethane
1,1,1-Trichloroethane
cis-1,2-Dichloroethene
2-butanone (methylethylketone)
2,2- Dichloropropane
Iodomethane
Methylene chloride
trans-1,2-Dichloroethene
Trichlorofluoromethane
Vinyl chloride
Vinyl Acetate
Dibromofluoromethane (surrogate)
1,4-Difluorobenzene (Internal standard)
Benzene
Bromodichloromethane
Bromoform
2-Butanone
Carbon Tetrachloride
Chlorodibromomethane
Dibromomethane
Dichlorodifluoromethane
1,1-Dichloropropene
1,2-Dichloroethane
1,2-Dichloropropane
cis-1,3-Dichloropropene
Ethyl methacrylate
Methyl methacrylate
trans-1,3-Dichloropropene
4-Methyl-2-pentanone
trans-1,3-Dichloropropene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Toluene
4-Bromofluorobenzene (surrogate)
Toluene-d_6 (surrogate)
Chlorobenzene-d_5 (Internal standard)
Bromoform
Chlorobenzene
Dibromochloromethane
1,2-Dibromoethane
Ethylbenzene
1,1,1,,2-Tetrachloroethane
1,3-Dichloropropane
2-hexanone
o-Xylene
m-/p-Xylene
Styrene
Tetrachloroethene
1,4-Dichlorobenzene- d_4 (Internal standard)
4-Chlorotoluene
Benzyl Chloride
n-Butylenzene
sec-Butylenzene
tert-Butylenzene
trans-1,4-Dichloro-2-butene
n-Propylenzene
Naphthalene
p-Cymene
Bromobenzene
Cumene
Hexachlorobutadiene
1,1,2,2-Tetrachloroethane
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-chloropropane
1,3,5-Trimethylbenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
2-chlorotoluene
4-chlorotoluene

Additional single point calibration compounds:
1,3-butadiene; n-hexane; Vinyl bromide; 1,2-Epoxybutane; Iso-octane; Heptane; Ethyl acrylate; methyl-t-butyl ether (MTBE)

Appendix 2B
SEMIVOLATILE COMPOUNDS
SW-846 Method 8270C

1,4-Dichlorobenzene-d_4
Benzyl alcohol
Bis(2-Chloroethyl)ether
Bis(2-Chloroisopropyl)ether
2-Chlorophenol
1,4-Dibromobenzene-d_4 (surrogate)
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
1,2-Dichlorobenzene-d_4 (surr)
Hexachlorophenol
2-Methylphenol
3/4-Methylphenol
N-Nitroso-di-n-propylamine
Phenol
Phenol-d_5 (surrogate)
2-Fluorophenol (surrogate)

Naphthalene-d_8
Benzoic acid
4-Chloroaniline
Bis(2-Chloroethoxy)methane
4-Chloro-3-methylphenol
2,4-Dichlorophenol
2,4-Dimethylphenol
Hexachlorobutadiene
Isophorone
Naphthalene
Nitrobenzene
2-Nitrophenol
1,2,4-Trichlorobenzene
1,3,5-Trichlorobenzene-d_3 (surrogate)
Nitrobenzene-d_5 (surrogate)

Acenaphthene-d_{10}
Acenaphthene
Acenaphthylene
2-Chloronaphthalene
4-Chlorophenyl phenyl ether
Dibenzofuran
Diethylphthalate
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Fluorene
Hexachlorocyclopentadiene
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
4-Nitrobenzene
2,4,5-Trichlorophenol
2,4,6-Trichlorophenol
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Phenanthrene-d_{10}
Anthracene
Anthracene-d_{10} (surrogate)
4-Bromophenyl phenyl ether
Di-n-butylphthalate
4,6-Dinitro-2-methylphenol
Hexachlorobenzene
N-Nitrosodiphenylamine
Pentachlorophenol
Phenanthrene

Chrysene-d_{12}
Benzo(a)anthracene
Bis(2-ethylhexyl)phthalate
Butylbenzylphthalate
Chrysene
3,3'-Dichlorobenzidine
Pyrene
Pyrene-d_{10} surrogate)
Terphenyl-d_{14} (surrogate)

Perylene-d_{12}
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Di-n-octylphthalate
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene

Non-target compounds known as tentatively identified compounds (TIC's) are identified by a computer generated searcch of the National Institute of Standards and Technology (NIST) Mass Spectral Library

Internal standards are underlined in this list.

Appendix 2C

DIOXIN/FURAN COMPOUNDS

Table 1- Method 551 Target Analytes

Specific Isomers	Total Isomers
2,3,7,8-TCDD	Total TCDD (22 isomers)
2,3,7,8-TCDF	Total TCDF (38 isomers)

Table 2- Methods 8280, 8290, 23, 0023A, 1613B

Specific Isomers	Total Isomers	
2,3,7,8-TCDD	Total TCDD	(22 isomers)
1,2,3,7,8-PeCDD	Total PeCDD	(14 isomers)
1,2,3,4,7,8-HxCDD	Total HxCDD	(10 isomers)
1,2,3,6,7,8-HxCDD		
1,2,3,4,6,7,8-HpCDD	Total HpCDD	(2 isomers)
OCDD		
2,3,7,8-TCDF	Total TCDF	(38 isomers)
1,2,3,7,8-PeCDF	Total PeCDF	(28 isomers)
2,3,4,7,8-PeCDF		
1,2,3,4,7,8-HxCDF	Total HxCDF	(16 isomers)
1,2,3,6,7,8-HxCDF		
2,3,4,6,7,8-HxCDF		
1,2,3,7,8,9-HxCDF		
1,2,3,4,6,7,8-HpCDF	Total HpCDF	(4 isomers)
1,2,3,4,7,8,9-HpCDF		
OCDF		

TCDD	tetrachlorodibenzo-p-dioxin
PeCDD	pentachlorodibenzo-p-dioxin
HxCDD	hexachlorodibenzo-p-dioxin
HpCDD	heptachlorodibenzo-p-dioxin
OCDD	octachlorodibenzo-p-dioxin
TCDF	tetrachlorodibenzo furan
PeCDF	pentachlorodibenzofuran
HxCDF	hexachlorodibenzofuran
HpCDF	heptachlorodibenzofuran
OCDF	octachlorodibenzofuran

Appendix 2D

PESTICIDE COMPOUNDS

Method 8081

Aldrin
α-BHC
β-BHC
δ -BHC
γ -BHC (Lindane)
Chlordane (technical)[a]
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Methoxychlor
Toxaphene
Aroclor 1016
Aroclor 1221
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260
Tetrachloro-meta-xylene (TCMX) - surrogate
Decachlorobiphenyl (DCBP) - surrogate

Appendix 3A

GC/MS ANALYTICAL METHODS: VOLATILES - SW-846 8260B

Matrices	VOST tubes, solids (by dilution method only) , water
Compounds	See Appendix 2A
Initial Calibration	5 point minimum; SPCC compounds RF > 0.30 for chlorobenzene and 1,1,2,2-tetrachoroethane and RF > 0.10 for chloromethane, 1,1-dichlorethane and bromoform ; CCC compounds RSD <30%
Continuing Calibration	mid-level standard analyzed at the beginning of every 12 hours of analysis time, SPCC compounds - same as initial calibration; CCC compounds %D < 20% from initial calibration average
Internal Standards	Pentafluorobenzene 1,4-Difluorobenzene Chlorobenzene-d_5 1,4-Dichlorobenzene- d_4
Standard Solution Lifetime	Stock Solutions (>1000 ppm): gases in methanol 2 months liquids in methanol 6 months Dilutions (<1000 ppm): in methanol 2 weeks in water 1 week
Holding Time	14 days from sample collection
Validation	Initial performance analysis (water): four 5 mL aliquots composed of reagent water spikes with all analytes at 20 µg/L. Results must meet all method criteria.
QC Check Sample	Blank matrix spiked with equivalent of 20 µg/L all analytes. Must meet all method criteria. Two (2) are analyzed each day of analysis or once per 20 samples whichever is greater.

Appendix 3B

GC/MS ANALYTICAL METHODS: SEMIVOLATILES SW-846 Method 8270C

Matrices	solid waste, soil, water, and air	
Compounds	See Appendix 2B	
Initial Calibration	5 point minimum, SPCC compounds RF > 0.050, CCC compounds RSD <30%	
Continuing Calibration	mid-level standard analyzed at the beginning of every 12 hours of analysis time, SPCC compounds RF > 0.050, CCC compounds %D < 30% from initial calibration average	
Internal Standards	1,4-Dichlorobenzene-d_4 Naphthalene-d_8 Acenaphthalene-d_{10}	Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}
Surrogate Standard	Nitrobenzene-d_5 2-Fluorobiphenyl Terphenyl-d_{14} Phenol-d_5 2-Fluorophenol	2,4,6-Tribromophenol Pyrene-d_{10} 1,3,5-Trichlorobenzene-d_3 1,4-Dibromobenzene-d_4 Anthracene-d_{10}
Standard Solution Lifetime	Stock Solutions 1 year	
Holding Time	Extraction: water 7 days from sample collection soils 14 days from sample collection Analysis: 40 days from extraction	
Validation	Initial performance analysis (water): four (4) 1 L samples composed of reagent water spikes with all analytes at 100 µg/L, extracted and analyzed. Results must meet all method criteria.	
QC Check Sample or Laboratory Control Sample	Blank matrix spiked with equivalent of 100 µg/L all analytes. Must meet all method criteria. Two (2) are analyzed each day of analysis or once per 20 samples whichever is greater.	

Appendix 3C

GC/MS ANALYTICAL METHODS: DIOXIN/FURAN

	Methods 8290 & 23	Method 1613B	Method 551
Matrices	water, soil, sludge, tissue, pulp, paper, ash, MM5, PUF (Method 23- MM5 only)		
Compounds	See Appendix 2C, Table 2	See Appendix 2C, Table 2	See Appendix 2C, Table 1
Initial Calibration	6 points- 20/30% RSD (Method 23- 25/30% RSD)	6 points- 20/25% RSD	5 points in duplicate - 20/25% RSD
Continuing Calibration	mid-level standard every 12 hours, 20/30% RPD (Method 23- 25/30% RPD)	Mid-level standard every 12 hours, approximately 20/25% RPD	Mid-level standard at the beginning of every 12 hours and 4th point at the end of injection sequence, 20% RPD
Internal Standards	$^{13}C_{12}$-2,3,7,8-TCDD $^{13}C_{12}$-2,3,7,8-TCDF $^{13}C_{12}$-1,2,3,7,8-PeCDD $^{13}C_{12}$-1,2,3,7,8-PeCDF $^{13}C_{12}$-1,2,3,6,7,8-HxCDD $^{13}C_{12}$-1,2,3,6,7,8-HxCDF $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF $^{13}C_{12}$-1,2,3,4,6,7,8-OCDD	same as Method 8290 plus: $^{13}C_{12}$-2,3,4,7,8-PeCDF $^{13}C_{12}$-1,2,3,4,7,8-HxCDD $^{13}C_{12}$-1,2,3,4,7,8-HxCDF $^{13}C_{12}$-1,2,3,7,8,9-HxCDF $^{13}C_{12}$-2,3,4,6,7,8-HxCDF $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	same as Method 8290 (except tetra isomers only)
Surrogate Standards	$^{37}Cl_4$-2,3,7,8-TCDD $^{13}C_{12}$-2,3,4,7,8-PeCDF $^{13}C_{12}$-1,2,3,4,7,8-HxCDD $^{13}C_{12}$-1,2,3,4,7,8-HxCDF $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	$^{37}Cl_4$-2,3,7,8-TCDD (cleanup standard)	same as Method 8290 (except tetra isomers only)
Recovery Standards	$^{13}C_{12}$-1,2,3,4-TCDD $^{13}C_{12}$-1,2,3,7,8,9-HxCDD	$^{13}C_{12}$-1,2,3,4-TCDD $^{13}C_{12}$-1,2,3,7,8,9-HxCDD	$^{13}C_{12}$-1,2,3,4-TCDD

Standard Solution Lifetime	Based on concentration- ≥10 µg/mL: 15 years or according to supplier specifications <10 µg/mL: 1 year
Holding Time	8290 Extract within 30 days of collection; analyze within 45 days of extraction Water - 1 year at 0-4° C Solids - 1 year at <-10 ° C. 23 Analyze within 60 days of collection
Validation	Initial performance analysis (water): four (4) 1L aliquots composed of reagent water spiked with all analytes: tetra at 200 pg/L, penta - hepta at 1000 pg/L, and octa at 2000 pg/L.
QC Check Sample	Blank matrix spiked in the same manner as the validation series. Two (2) are analyzed each day of analysis or once per 20 samples whichever is greater. (Only 1 per day or once per 20 samples Method 1613)

Appendix 3D

GC/MS ANALYTICAL METHODS: PESTICIDES/PCBs

Matrices	groundwater, soil, non water-miscible waste
Compounds	See Appendix 2D
Initial Calibration	5 point minimum, RSD ≤20%, use average RF
Continuing Calibration	mid-level standard every 10 samples, ≤15% D
Internal Standards	Decafluorobiphenyl 2,4,5,6-Tetrachloro-meta-xylene
Standard Solution Lifetime	Stock Solutions (≥1000 ppm): According to supplier specifications Working Solutions (<100 ppm): 6 months
Holding Time	Extraction: water 7 days from sample collection soils 14 days from sample collection Analysis: 40 days from extraction
Validation	Initial performance analysis (water): four (4) one L aliquots composed of reagent water spikes with all analytes at equivalent of 10 and 2 µg/L. Results must meet all method criteria.
QC Check Sample	Blank matrix spiked with all analytes, equivalent of 10 and 2 µg/L. Must meet all method criteria. Two (2) are analyzed each day of analysis or once per 20 samples whichever is greater.

Appendix 4

CONTAINERS, PRESERVATIVES AND HOLDING TIMES

Parameter	Matrix	Holding time	Recommended Volume	Preservative[d]
Volatile[a] organics	Water	14 days from collection	two 40 mL glass vials with Teflon lined septa, no headspace present	4 drops conc. HCl 4°C
	Soil - Method 5035 not available	14 days from collection	20 g. glass vials with teflon septa, no headspace present	Cool, 4°C
Extractable[b] organics	Water	7 days from collection 40 days from extraction	2.0 L in glass bottles	Cool, 4°C, $Na_2S_2O_3$ if residual chlorine (80 mg/L)
	Soil	14 days from collection 40 days from extraction	100 g in glass jar	Cool, 4°C
AOX/TOX	Water	≥3 days but <6 months after collection	250 mL in glass bottle	HNO3 to pH <2, $Na_2S_2O_3$ if residual chlorine present (20 mg/ 250 mL)
	Soil	≥3 days but <6 months after collection	50 g in glass jar	none
Dioxin	Water	method specific, see Appendix 3C	Two 1.0 L aliquots in glass bottles	Cool, 4°C 1613 - $Na_2S_2O_3$ if residual chlorine (80 mg/L)
	Soil	method specific, see Appendix 3C	100 g in glass jar	Cool, 4°C
Metals	Water	28 days Hg 6 months all other metals	0.5 L in glass or plastic	HNO3 to pH <2
	Soil	28 days Hg 6 months all other metals	50 g in glass jar	none

[a]For SW-846: Free chlorine must be removed prior to addition of HCl by addition of sodium thiosulfate ($Na_2S_2O_3$). Adjust pH <2 for purgeable aromatic hydrocarbons with H_2SO_4, HCl or solid $NaHSO_4$. Adjust pH to 4-5 for acrolein and acrylonitrile.

[b]For SW-846: Preserve phenols, benzidines, nitrosamines, nitroaromatics and cyclic ketones, PAHs, haloethers, chlorinated hydrocarbons and pesticides with 0.008% $Na_2S_2O_3$. Nitrosamines, Nitroaromatics, cyclic ketones and PAHs should be stored in the dark. Pesticides pH=5-9.

[c]For CLP: Dissolved metals require filtration before pH adjustment.

[d]Preservation temperatures are approximate with an acceptable range of ±2.0°C.