QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR Triangle Lake Forestry Pesticides Project Environmental Sampling and Analysis for Pesticide Exposure Assessment

Date: September 15, 2011
Revision: 0.0

APPROVAL OF QAPP:

Scott E Downey
Scott Downey, Manager
Pesticides and Toxics Unit
Office of Compliance and Enforcement
U.S. EPA Region 10

Date: 9/15/11

Sheila, Fleming, P.E., Manager
Risk Evaluation Unit
Office of Environmental Assessment
U.S. EPA Region 10

Date: 9/15/2011

Gina Grepo-Grove, QA Manager
Office of Environmental Assessment
U.S. EPA Region 10

Date: 9/15/11

TABLE OF CONTENTS

A. Project Management

A1 – Distribution List

Electronic copies of the completed/signed quality assurance project plan (QAPP) should be distributed to the following:

Table 1 – Distribution List

Name	Affiliation	Phone/E-mail
Scott Downey	EPA Manager, Pesticides and Toxics Unit	206-553-0682 Downey.Scott@epa.gov
Gina Grepo-Grove	EPA Regional Quality Assurance Manager (RQAM)	206-553-6395 Grepo-Grove.Gina@epa.gov
Raymond Wu	EPA Field Health and Safety	206-553-1413 Wu.Raymond@epa.gov
Jed Januch	EPA - Environmental Protection Specialist	360-871-8731 Januch.Jed@epa.gov
Elizabeth Allen	EPA Human Health Risk Assessor	206-553-1807 Allen.Elizabeth@epa.gov
Jennifer Crawford	Regional Sample Control Coordinator	206-553-6261 Crawford.Jennifer@epa.gov
Greg Pettit	ODEQ Laboratory Director	503-229-5983 Pettit.Greg@deq.state.or.us
Joshua Seeds	ODEQ Project Manager	503-229-5081 Seeds.Joshua@deq.state.or.us
Brian Boling	ODEQ Organic Laboratory Manager	503-693-5745 Boling.Brian@deq.state.or.us
Chris Bayham	ODEQ Willamette River Basin Coordinator	541-687-7356 Bayham.Chris@deq.state.or.us
Aaron Borisenko	ODEQ Water Monitoring Manager	503-693-5723 Borisenko.Aaron@deq.state.or.us
Scott Hoatson	ODEQ Quality Assurance Officer	503-693-5786 Hoatson.Scott@deq.state.or.us
Richard Myzak	ODEQ Field Support	503-229-5983 Myzak.Richard@deq.state.or.us
Jae Douglas	OHA Research and Education Section Manager	971-673-1139 Jae.P.Douglas@state.or.us
Dave Farrer	OHA Public Health Toxicologist	971-673-0971 David.G.Farrer@state.or.us
Richard Kauffman	ATSDR Senior Regional Representative	206-553-2632 Kauffman.Richard@epa.gov
Link (Grant) Smith	ODF Western Lane District Forester	541-935-2283 Grant.S.Smith@state.or.us

Dale Mitchell	ODA Pesticides Division Assistant Administrator	503-986-4646 Dmitchel@oda.state.or.us
Kathleen Wickman	ODA Laboratory Manager	503-872-6633 Kwickman@oda.state.or.us

At the conclusion of sampling and analysis, electronic copies of the project narrative and analytical data should be provided to:

Scott Downey, EPA Project Manager	OCE-084
Sheila Fleming, Risk Evaluation Unit Manager	OEA-095
Elizabeth Allen, Risk Assessor	OEA-095

A2 – Project/Task Organization

The following individuals are EPA, ODEQ, and ODA staff with responsibility for design and implementation of this project:

- **Elizabeth Allen**, EPA, (206) 553-1807, human health risk assessor responsible for sampling plan design and data interpretation.
- **Jed Januch**, EPA, (360) 871-8731, field team lead responsible for preparation of the QAPP and sample collection.
- **Donald M. Brown**, EPA, (206) 553-0717, quality assurance staff responsible for preparation of the QAPP.
- **Raymond Wu, EPA,** (206) 553-1413, field staff responsible for health and safety plan and sample collection.
- **Richard Myzak, ODEQ,** 503-229-5983, field staff responsible for sample collection.
- **Jennifer Crawford**, EPA, (206) 553-6261, Regional Sample Control Coordinator (RSCC) residing in the Quality Assurance (QA) Office. The RSCC will provide sample numbers for samples generated for this study.
- **Brian Boling**, **ODEQ**, 503-693-5745, Organic Laboratory Manager responsible for directing analysis of samples at ODEQ Laboratory.
- **Kathleen Wickman, ODA**, 503-872-6633, Laboratory Manager responsible for directing analysis of samples at ODA Laboratory.

A3 – Problem Definition/Background

Residents along the Highway 36 corridor in the Triangle Lake area (the Lake Creek watershed) in Oregon have been raising concerns about exposure to herbicides used in nearby commercial timber operations. Recent tests conducted by Emory University revealed the presence of two herbicides, 2, 4-dichlorophenoxy acetic acid (2,4-D) and atrazine, in samples of urine submitted for analysis by concerned residents. The levels suggest an ongoing exposure that could be from spray drift, revolatilization, or possible contamination of drinking water. This sampling effort will explore whether pesticide residues can be detected in drinking water as well as homegrown garden produce and vegetation, animal products (i.e. milk and eggs), and surface soil in the Triangle Lake/Hwy 36 area.

This multi-agency study is being led by ATSDR and Oregon Health Authority with technical support from EPA (sample collection), Oregon Department of Environmental Quality (sample collection and laboratory analysis), Oregon Department of Agriculture (laboratory analysis), and Oregon Department of Forestry.

A4 – Project Task Description

Collect water samples from private sources of drinking water, vegetation (edible fruits and vegetables), animal products, and soil from approximately forty residential locations. Analyze water samples for herbicides and general water chemistry parameters including temperature, pH, conductivity, and dissolved oxygen and field screen for chlorine residual. Analyze soil, vegetation, and animal product samples for herbicides.

Table 2 includes a schedule for conducting tasks related to this project. The information in Table 2 is a guideline only as it is possible that unforeseen circumstances and conditions will require adjustment to some or all of the following proposed dates.

Table 2 – Schedule of Tasks

Activity	Estimated Start Date	Estimated Completion Date	Comments
Project-Specific QAPP Review/Approval	July 21, 2011	September 15, 2011	QAPP will be reviewed and approved by EPA Region 10 OEA.
Sampling	September 19, 2011	September 29, 2011	Sampling will be conducted by EPA Region 10 OEA and ODEQ.
Laboratory Receipt of Samples	September 20, 2011	September 30, 2011	Water samples will be delivered to the ODEQ Laboratory. Vegetation, food, product and soil samples will be delivered

			to the ODA Laboratory.
Laboratory Analysis of Samples	September 20, 2011	November 16, 2011	Analysis of water samples by ODEQ. Analysis of vegetation/animal product samples by ODA.
Data Verification and Validation	November 17, 2011	December 16, 2011	EPA will verify/validate all laboratory analyses.

A5 – Quality Objectives and Criteria

Data Quality Objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of the data to be collected. EPA has determined a seven-step process for establishing DQOs and for developing QAPPs to help ensure that data collected during a study will be adequate to support reliable decision making. The seven-step DQO process is detailed below.

Step 1: State the Problem

Describe the Problem

There is concern that off target movement of pesticides applied to commercial timber operations has drifted onto local residential properties resulting in possible human exposure to pesticides. Results of recent testing of urine samples collected from residents living near the pesticide application areas indicate possible exposure to herbicides 2, 4-D and atrazine.

Planning Team

This QAPP has been planned by a team of scientists including laboratory personnel, quality assurance personnel, toxicologists, and other technical specialists. Section A2 identifies the key personnel, data users, and decision makers for the project.

Data Needs and Use

This QAPP will guide the collection and analysis of environmental samples including water, soil, vegetation, and animal products. The data collected during this project will support an exposure assessment being conducted by ATSDR and OHA and help determine concentrations of pesticides/pesticide residues that may be present at the study locations.

Resources, Constraints, and Deadlines

Sample collection resources for this project will be provided by EPA Region 10 OEA and ODEQ. Laboratory analytical resources will be provided by ODEQ and ODA. Table 2 includes a schedule for

conducting tasks related to this project. As noted above, the schedule may be adjusted as appropriate to accommodate unforeseen circumstances that influence timing of the sample collection and analysis.

Step 2: Identify the study goals

The primary goal of this study is to determine whether residents near timber spraying areas are exposed to pesticides because of spray operations.

Step 3: Identify the Types of Data Needed

In order to consider various possible sources of exposure to pesticides, representative samples of drinking water, vegetation (edible plant materials), animal products (milk, honey, eggs), and surface soil will be collected from approximately 40 locations. Water sampling will be supplemented by direct field measurements of general water chemistry parameters including temperature, pH, conductivity, and dissolved oxygen and field screening for chlorine residual. In addition to the field samples, the sampling team will also collect quality assurance/quality control (QA/QC) samples (field blanks and duplicates) at the frequency specified in this QAPP. Analytical data from the laboratory will be reported in standard international (SI) units.

Step 4: Define the Study Bounds

This sampling effort is limited to a single event. Sampling locations in the Triangle Lake area are being identified by OHA. Samples will consist of representative drinking water, vegetation, animal product, and soil samples collected according to the specifications in this QAPP.

Step 5: Define the Analytical Approach

Measurement Quality Objectives (MQOs) are the quantitative and qualitative terms field personnel and project managers use to describe how good the data need to be in order to meet the project's objectives. MQOs for measurement data are precision, accuracy, representativeness, completeness, comparability, and measurement range. The overall QA objective for analytical data is to ensure that data of known and acceptable quality are provided. To achieve this goal, data must be reviewed for 1) representativeness, 2) comparability, 3) precision, 4) accuracy (or bias), and 5) completeness. Precision, accuracy, completeness, sample representativeness, and data comparability are necessary attributes to ensure that analytical data are reliable, scientifically sound, and legally defensible.

Precision: Field precision is typically estimated by the collection of field duplicate or co-located samples.

Lab precision and accuracy can be measured by the laboratory measuring Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples and the analysis of laboratory duplicate samples. The laboratory usually

performs analysis of at least one set of MS/MSD and duplicate from the field samples per matrix. Laboratory precision will be determined by the spike recoveries and the RPDs of the MS/MSD samples, respectively.

$$RPD = \frac{ABS\ (R1 - R2)}{\left(\frac{R1 + R2}{2}\right)} \times 100$$

R1 = Recovery for MS or duplicate 1
R2 = Recovery for MSD or duplicate 2

Accuracy in the lab will be evaluated by the use of percent recovery (%R) of the target analyte in spiked samples and surrogates in all samples and QC samples.

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

Field accuracy is measured by conducting flow rate calibrations on the instruments at specified frequencies.

Representativeness is the degree to which data from the project accurately represent a particular characteristic of the environmental matrix which is being tested. Representativeness of samples is ensured by adherence to standard field sampling protocols and standard laboratory protocols. For this project we intend to use EPA field sampling methods and validated analytical methods in conjunction with approved EPA, ODEQ, or ODA developed extraction and analytical procedures. The design of the sampling scheme and number of samples should provide a representativeness of each matrix being sampled.

Comparability is the measurement of the confidence in comparing the results of one experiment with the results of different experiments using the same matrix, sample location, sampling techniques and analytical methodologies. Since the sampling, extraction, and analytical techniques and methodologies prescribed by the analytical laboratories, our results should be comparable to other studies.

Completeness: Completeness is the percentage of valid results obtained compared to the total number of samples taken for a parameter. The goal for this study is to collect valid results at better than 75% completeness.

$$\%\ \text{Completene ss} = \frac{\#\ \text{of valid results}}{\#\ \text{of samples taken}} \times 100$$

The QA objectives specified, above, will be evaluated during the data validation process.

Step 6: Define the acceptance criteria

The acceptance criteria for all analyses are described in Table 7 at the end of this document.

Step 7: Optimize the study design

This sample collection effort has been designed to meet the data needs identified by OHA to conduct an exposure assessment. The number and type of samples to be collected has been optimized based on available resources and laboratory capacity.

A6 – Special Training and Certification

The sampling team collecting samples for this project are trained and experienced sampling personnel and have completed at minimum the 40-hour training in Basic Health and Safety. In addition, they have completed an 8-hour health and safety-training refresher course within the last year.

The laboratories performing the sample analysis of drinking water analytes for this program are certified and/or accredited. Scientists (Chemists) performing the analytical work for this project have extensive knowledge, skill, and demonstrated experience in the execution of the analytical methods being requested.

A7 – Documents and Records

It will be the responsibility of the QA officer to ensure that appropriate project personnel have the most current approved version of the QAPP including addenda. The final signed version of the QAPP and any addenda will be distributed in portable document file (pdf) format.

Processing documentation may include the projects field sample data form (see Appendix A) and sample custody and analysis required form (see Appendix B). Laboratory documentation may include, but is not limited to hard copy bench sheets, electronic data deliverable (EDD) spreadsheets, sample preparation and analysis logs, and results of calibration and quality control (QC) checks.

The project documentation will be kept in a case file and submitted to OEA's Risk Evaluation Unit for inclusion in the final narrative report. The following documents will be archived at the laboratory: (1) signed hard copies of chain-of-custody records (2) electronic and hard copies of analytical data. The

laboratory will store all sample receipt, sample login, and laboratory instrument documentation for a minimum of seven years.

B. Data Generation and Acquisition

The elements in Sections B1-B10 are designed to ensure that appropriate methods for sampling, measurement and analysis, data collection, data handling, and QC activities are employed and documented.

B1 – Sampling Process Design (Experimental Design)

OHA will provide the locations where samples are to be collected. Samples may also be obtained as a result of visual examination of the site. Field analysis and sample collection processes will be based on EPA or United States Department of Agriculture (USDA) standard operating procedures (SOPs) (see Appendices C-G). **Note: Due to the nature of several sample media, deviations from the sampling SOPs may occur and should be noted in the field notebooks.**

B2 – Sampling Methods

Drinking Water Samples

Drinking water samples will be obtained from the residences identified by OHA. The procedure for obtaining samples will include purging which is necessary to remove stagnant water from the source (including pipes used to convey water), immediately prior to sampling, causing its replacement by ground water from the adjacent formation, which is representative of actual aquifer conditions. In order to determine when a well has been adequately purged, samplers will use a multi-parameter water quality checker to monitor the pH, conductivity, temperature, and turbidity of the ground water removed during purging. In addition, the sampling team will test for residual chlorine with the aid of a chlorine detection kit. Samplers will record the results of field analysis in the project notebook. The multi-parameter water quality checker used for these measurements will be calibrated and results from that calibration recorded in the project notebook prior to daily sampling.

Water samples will be collected following purging from a valve or cold water tap as near to the well as possible, preferably prior to any storage/pressure tanks or filtration systems that might be present. Samples will be collected directly into 1,250 ml amber glass containers with Teflon lids or 60 ml VOA vials for pesticide analysis.

Vegetation Samples

The sampling team will collect materials (edible foliage, fruits, and other plant parts) from vegetation and place them in clean stainless steel bowl for visual examination. In cases where symptoms indicate herbicide exposed plant tissues, the damaged plant tissue will be isolated and transferred into a sample container (paper bag contained within a plastic Ziploc bag) and submitted to the lab for analysis. The sampling team will collect approximately one pound of each vegetation sample.

If a pesticide drift pattern is apparent, the sampling team will collect samples in a gradient pattern sequentially from the area with least anticipated residue concentration to the greatest anticipated concentration. In cases where there is no apparent pattern, the sampling team will attempt to collect vegetation samples in a grid pattern.

Animal Product Samples

The sampling team will collect animal products (i.e. milk, eggs, and honey) from residences identified by OHA that grow and consume these products as part of their normal diet. Milk samples for glyphosate analysis will be collected in 500 ml polypropylene plastic containers and milk samples for other analyses will be collected in 500 ml glass containers. The sampling team will collect four eggs for each laboratory parameter and these will be placed in an egg carton. Honey samples for glyphosate analysis will be collected in 100 ml polypropylene plastic containers and honey samples for other analyses will be collected in 100 ml glass containers.

Soil Samples

In the event that the sampling team is present during or immediately following a pesticide spraying event, soil samples will be collected. Soil samples should be collected in the open in areas where it does not appear that deposition could be hindered by any structures or natural objects. Soil samples for glyphosate analysis will be collected in 500 ml polypropylene plastic containers and soil samples for other analyses will be collected in 500 ml glass containers.

Table 3 – Sample Locations

Sampling Location	# of locations	# samples per facility	Analytes
Drinking Water (from tap)	38	1	ODEQ: TMP-phenoxy herbicides + DCPA metabolites, Organic Compounds, Pesticides
Vegetation	20	0-1	ODA: Atrazine; Hexazinone; Imazapyr; Sulfometuron-Methyl; Metsulfuron-Methyl; Aminopyralid, 2,4-D; Clopyralid; Triclopyr; Picloram; Glyphosate

Eggs	2	1	ODA: Atrazine; Hexazinone; Imazapyr; Sulfometuron-Methyl; Metsulfuron-Methyl; Aminopyralid, 2,4-D; Clopyralid; Triclopyr; Picloram; Glyphosate
Milk	1	1	ODA: Atrazine; Hexazinone; Imazapyr; Sulfometuron-Methyl; Metsulfuron-Methyl; Aminopyralid, 2,4-D; Clopyralid; Triclopyr; Picloram; Glyphosate
Honey	1	1	ODA: Atrazine; Hexazinone; Imazapyr; Sulfometuron-Methyl; Metsulfuron-Methyl; Aminopyralid, 2,4-D; Clopyralid; Triclopyr; Picloram; Glyphosate
Soil	Up to 38	1	ODA: Atrazine; Hexazinone; Imazapyr; Sulfometuron-Methyl; Metsulfuron-Methyl; Aminopyralid, 2,4-D; Clopyralid; Triclopyr; Picloram; Glyphosate

B3 – Sample Handling and Custody

Each sample will be identified with a unique sample number assigned by the RSCC. EPA Region 10 chain-of-custody procedures and forms will be used. Custody seals will be placed on all sample containers during transit to the laboratory. Samples will be hand carried to the appropriate laboratory by a member of the sampling team. Samples will be chilled in wet ice (to approximately 4^{o}C) and transported to the lab in a covered cooler.

Table 4 – Laboratory Locations and Contacts

Analysis	Location	Contacts
Drinking Water Pesticides	Oregon DEQ Laboratory 3150 NW 229th Avenue Suite 150 Hillsboro, Oregon 97124	Shannon Swantek 503-693-5784 or Brian Boling 503-693-5745
Vegetation, Animal Products, and Soil	ODA Laboratory 1207 NW Naito Parkway Suite 204 Portland, Oregon 97209 503-872-6644	Kathleen Wickman 503-872-6633 (office) 503-872-6644 (cell)

B4 – Analytical Methods

The compounds of concern (COC) identified for this project include the following:

Table 5 – ODEQ Pesticide Suite Analytes and Methods (Drinking Water)

TMP - Phenoxy Herbicides + DCPA metabolites done by method SM 6640 and reported in µg/L (MRL)		
2,4,5-T (0.30)	2,4-D (0.10)	2,4-DB (0.60)
3,5-Dichlorobenzoic acid (0.30)	Acifluorfen (0.20)	DCPA acid metabolites(a) (0.60)
Dicamba (0.30)	Dichloroprop (0.30)	Dinoseb (0.30)
MCPA (20)	MCPP (60)	Pentachlorophenol (0.10)
Picloram (0.60)	Silvex (0.10)	Triclopyr (0.30)
Organic compounds by LC/MS/MS – Rogue/Umatilla done by method 8321 and reported in ng/L (MRL)		
Acetamiprid (4.0)	Acetochlor (10.0)	Alachlor (10.0)
Ametryn (4.0)	Aminocarb (4.0)	Atrazine (4.0)
Atrazine-Desethyl (4.0)	Atrazine-Desisopropyl (4.0)	Azinphos Methyl (20)
Baygon (4.0)	Carbaryl (5.0)	Carbofuran (4.0)
DEET (5.0)	Diuron (4.0)	Fluometuron (4.0)
Imazapyr (40)	Imidacloprid (20)	Linuron (4.0)
Methiocarb (4.0)	Methomyl (4.0)	Metolachlor (10.0)
Metribuzin (4.0)	Mexacarbate (4.0)	Neburon (5.0)
Oxyamyl (4.0)	Prometon (4.0)	Prometryn (4.0)
Propazine (4.0)	Propiconazole (20.0)	Pyraclostrobin (4.0)
Siduron (4.0)	Simazine (4.0)	Simetryn (4.0)
Sulfometuron-Methyl (4.0)	Terbutryne (4.0)	Terbutylazine (4.0)
Pesticide / Herbicides by GC/MS CLLE done by method 8270 and reported in ng/L (MRL)		
4,4’ DDD (25)	4,4’ DDE (25)	4,4’ DDT (25)
Alachlor (30)	Aldrin (25)	alpha-BHC (25)
Atrazine (50)	Azinphos Methyl (40)	beta-BHC (25)
Bromacil (25)	Butachlor (25)	Butylate (25)
Carboxin (50)	Chlorobenzilate(a) (25)	Chloroneb (25)
Chlorothalonil (25)	Chlorpropham (25)	Chlorpyrifos (Dursban) (25)
cis-Chlordane (25)	Cyanazine (25)	Cycloate (25)
Dacthal (25)	delta-BHC (25)	Diazinon (25)
Dichlorvos (25)	Dieldrin (25)	Dimethoate (25)
Diphenamid (25)	Disulfoton (50)	Endosulfan I (25)
Endosulfan II(25)	Endosulfan sulfate (25)	Endrin (25)
Endrin Aldehyde (25)	EPTC (Eptam) (25)	Ethoprophos (25)
Etridiazole (25)	Fenamiphos (30)	Fenarimol (25)
Fenvalerate & Esfenvalerate (500)	Fluridone (25)	Heptachlor (25)
Heptachlor epoxide (25)	Hexazinone (25)	Imidan aka Phosmet (25)
Lindane aka gamma-BHC (25)	Malathion (25)	Methoxychlor (25)
Methyl paraoxon (25)	Methyl Parathion (25)	Metolachlor (25)
Metribuzin (25)	MGK-264 (50)	Molinate (25)
Napropamide (25)	Norflurazon (25)	Pebulate (25)
Pendimethalin (25)	Permethrin (50)	Phosdrin (Mevinphos) (25)
Pronamide (25)	Propachlor (25)	Propazine (25)
Pyriproxyfen (250)	Simazine (25)	Tebuthiuron (25)
Terbacil (25)	Terbufos (40)	Tetrachlorvinphos (25)
trans-Chlordane (25)	trans-Nonachlor (25)	Triadimefon (25)
Tricyclazole (25)	Trifluralin (25)	Vernolate (25)

Table 6 – ODA Pesticide Suite Analytes and Methods (soil, vegetation, milk, eggs, and honey)

Compound (MRL – reported in ppb)	Method
Atrazine (10) Hexazinone (10)	Quechers GD0908
Imazapyr (10) Sulfometuron-Methyl (10) Metsulfuron-Methyl (10) Aminopyralid (10)	
2,4-D (10) Clopyralid (10) Triclopyr (10) Picloram (10)	Phenoxy Quechers GD110112
Glyphosate (10)	GD110325 rev. 1.0

The analytical methods, container specifications, preservative, and holding time requirements are listed in Table 7 – Summary of Data Quality Objectives attached at the end of this QAPP.

B5 – Quality Control

Quality Control (QC) consists of the collection of data that allow a quantitative evaluation of the accuracy and precision of the samples that are analyzed. Each type of QC sample is described in more detail below.

Field QC Samples

Field blanks and duplicates will be collected at a frequency of 5% or 1 in every 20 samples for the drinking water samples only.

Laboratory QC Samples

The laboratory is expected to analyze for QC as indicated by the respective analysis SOPs to include surrogates, matrix spikes, laboratory control samples, blanks, and calibration verifications. The limits for these analyses are outlined in Table 7.

B6 – Instrument/Equipment Testing, Inspection, and Maintenance

Field Equipment

Field equipment will consist of multi-parameter water quality probes, flow through cells, and chlorine test kits. EPA routinely cleans and services this equipment. Other field sampling equipment will include new/clean disposable scoops for soil sampling.

Lab Instruments

Laboratory instruments required by the applicable analytical methods will be maintained according to the manufacturer instructions and the laboratory SOPs. Records for equipment service shall be maintained by the laboratory.

B7 – Instrument/Equipment Calibration and Frequency

Field Instruments

Daily calibration will be required for the multi-parameter probes prior to field use. Records from these calibrations are kept in booklets dedicated to each instrument.

Laboratory Instruments

Laboratory equipment will be calibrated using the method and frequency specified in the laboratory SOPs. Records on calibration of laboratory equipment shall be maintained by the laboratory.

B8 – Inspection/Acceptance of Consumable Supplies

The consumable supplies for the drinking water samples will consist of quality control class sample containers (1250 ml amber bottles and 60 ml VOA vials) provided by EPA Region 10 OEA. The consumable supplies for the vegetation sampling will consist of paper bags and plastic zip lock bags. The consumable supplies for the soil sampling will consist of 500 ml polypropylene plastic containers and 500 ml glass containers. The consumable supplies for the milk sampling will consist of 500 ml polypropylene plastic containers and 500 ml glass containers. The consumable supplies for the honey sampling will consist of 100 ml polypropylene plastic containers and 100 ml glass containers. The consumable supplies for the egg sampling will consist of egg cartons.

Consumable supplies in the laboratory will consist of reagents and standard reference materials (SRMs).

The quality of standards and other consumable supplies used for this project should be documented by the supplier and certificates should be available to EPA on request. In the case of the paper and plastic zip lock bags, blank samples will be provided to the laboratory prior to sampling to determine if these supplies are free of pesticide residues.

B9 – Non-Direct Measurements

Not applicable.

B10 – Data Management

All data generated as part of this study will be maintained in a study-specific Microsoft® Access database or Excel spreadsheet.

C. Assessment and Oversight

C1 – Assessments and Response Actions

Quality assurance (QA) assessments may be conducted during the course of this project. The quality assurance assessment performed during this project may include the following:

1) Oversight of sample processing activities
2) Oversight of sample handling and chain-of-custody procedures
3) Laboratory inspections

QA assessments will be conducted by the EPA Region 10 QA Manager or QA staff delegated by the manager to conduct assessments.

Laboratories routinely perform performance checks using different program specific blind and double blind check standards. Each analytical method requires specific QA/QC runs that must be complied with by the laboratory performing the analysis. An internal assessment of the data and results are also routinely conducted by the appropriate supervisors and the Laboratory QA Coordinator. No additional audits will be performed on the laboratory for this project.

Corrective action procedures that might be implemented from QA results or detection of unacceptable data will be developed if required and documented using the Corrective Action Form (see Appendix H).

C2 – Reports to Management

If, for any reason, the schedules or procedures above cannot be followed, the project manager shall complete a Sample Alteration Form (SAF) (see Appendix I). The SAF should be reviewed and approved by the QA Manager. The laboratory should be given a copy of the approved SAF for reference and project file.

D. Data Validation and Usability

D1 – Data Review, Verification, and Validation

Data review is the in-house examination of the data to ensure that they have been recorded, transmitted, and processed correctly. Data verification is the process for evaluating the completeness, correctness,

and conformance of the data against the requirements specified in the QAPP. Data validation is a sample-specific process that determines the quality of data relative to its end use.

D2 – Verification and Validation Methods

Data review will include checking that results have been transferred correctly from laboratory log books and bench sheets to the EDD. Additional data reviews of all analytical results will be performed at a frequency of 10%.

Data verification will include a review of the findings of all QA assessment activities including:

1) Sample processing procedures
2) Sample labeling methods
3) Chain-of-custody procedures
4) Analytical preparation and analysis procedures

If any deviations are identified, the potential impact of those deviations on the reliability of the data will be assessed, and the information will be provided to the project manager.

Data validation consists of examining the sample data package(s) against pre-determined standardized requirements. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, COC information, raw data, initial and continuing instrument calibration, and other reported information to determine the accuracy and completeness of the data package. During this process, the validator will verify that the analytical methodologies were followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information. Analytical results for each sample will then be qualified as necessary.

All analytical results for samples collected by the EPA will be verified and validated by the EPA Quality Assurance team. Laboratories should submit analytical results with all proper quality control and analytical raw data for each analysis and sample. The following deliverables under subheadings I through IV will be required with submission of the laboratory data:

I. Case Narrative

- Case Narrative per batch of samples per suite of parameters - a summary of samples received, extraction techniques and analytical method used with focus on problems encountered during extraction and analysis, corrective action taken, data limitations (if any), example of calculations and definitions of laboratory qualifiers applied.

II. QC data

- Summary of surrogate recoveries

- Matrix Spike/Matrix spike duplicate recoveries
- Fortified blank recovery results (1 per batch)
- List of samples associated with the method blank
- Calibration summary
- Continuing calibration summary
- Internal Standards area and recovery summary (for dual columns) - compound identification summary (concentrations reported from each column reported with %D)

III. Sample Data

- Summary of Analytical results (surrogate recoveries can be reported here too). Analytical Results should also include a sample specific reporting limit. For solids/tissues, Reporting Units need to be identified as either dry weight (include percent moisture determination) or wet weight
- Instrument Raw Data (Chromatograms, Mass Spectra, etc.)

IV. Miscellaneous Data

- Copy of method SOP
- Sample receipt documentation and sample control
- Extraction Logs
- Instrument Run Logs
- Clean-up calibration
- Other analytical runs (screens, clean-up, etc)

D3 – Reconciliation with User Requirements

Once all samples have been processed and analytical data has been generated, data will be evaluated to determine if study DQOs were achieved. Evaluation of the study data will include a qualitative and quantitative review of all QA checks, QC samples and deviations from procedures described in this QAPP, along with conclusions regarding the reliability of the data for their intended use.

Table 7 – Data Quality Objectives Summary

Analytical Group	# of Samples	# of QA Samples	Matrix	Method	MRLs	Accuracy	Precision (RPD)	Complete-Ness	Volume, Container	Holding Time (days)	Preservation
TMP-phenoxy herbicides + DCPA metabolites	38	2	Water	SM6640	Per Analyte	+/- 30%	+/- 30% RPD	> 90%	2-60 ml, VOA vial w/PTFE lid liner	Extraction – 14 Analysis – 21	4 deg. C
Organic Compounds	38	2	Water	8321	Per Analyte	+/- 30%	+/- 40% RPD	> 90%	1250 ml, amber glass w/PTFE lid liner	Extraction – 7 Analysis – 14	4 deg. C
Pesticides	38	2	Water	8270	Per Analyte	+/- 40%	+/- 30% RPD	> 90%	1250 ml, amber glass w/PTFE lid liner	Extraction – 7 Analysis – 40	4 deg. C
Atrazine Hexazinone Imazapyr Sulfometuron-Methyl Metsulfuron-Methyl Aminopyralid	38/20	2/0	Soil Vegetation	Quechers GD0908	10 ppb	60-130%	N/A	> 90%	Soil 500 ml glass jar; veg. 1 pound, paper bag w/plastic bag cover	Extraction – 14 Analysis – 40	4 deg. C
2.4-D Clopyralid Triclopyr Picloram	38/20	2/0	Soil Vegetation	Phenoxy Quechers GD110112	10 ppb	60-130%	N/A	> 90%	Soil 500 ml glass jar; veg. 1 pound, paper bag w/plastic bag cover	Extraction – 14 Analysis – 40	4 deg. C
Glyphosate	38/20	2/0	Soil Vegetation	GD110325 rev. 1.0	10 ppb	+/- 20%	N/A	> 90%	Soil 500 ml	Extraction – 14 Analysis – 40	4 deg. C

Analytical Group	# of Samples	# of QA Samples	Matrix	Method	MRLs	Accuracy	Precision (RPD)	Complete-Ness	Volume, Container	Holding Time (days)	Preservation
									polypropylene plastic container; veg. 1 pound, paper bag w/plastic bag cover		
Atrazine Hexazinone Imazapyr Sulfometuron-Methyl Metsulfuron-Methyl Aminopyralid	1/1/2	0	Milk Honey Eggs	Quechers GD0908	10 ppb	60-130%	N/A	> 90%	Milk 500 ml glass container; Honey 100 ml glass jar; Eggs 4 in carton	Extraction – 14 Analysis – 40	4 deg. C
2.4-D Clopyralid Triclopyr Picloram	1/1/2	0	Milk Honey Eggs	Phenoxy Quechers GD110112	10 ppb	60-130%	N/A	> 90%	Milk 500 ml, glass container; Honey 100 ml glass jar; Eggs 4 in carton	Extraction – 14 Analysis – 40	4 deg. C
Glyphosate	1/1/2	0	Milk Honey Eggs	GD110325 rev. 1.0	10 ppb	+/- 20%	N/A	> 90%	Milk 500 ml polypropylene plastic container; Honey	Extraction – 14 Analysis – 40	4 deg. C

Analytical Group	# of Samples	# of QA Samples	Matrix	Method	MRLs	Accuracy	Precision (RPD)	Complete-Ness	Volume, Container	Holding Time (days)	Preservation
									100 ml polypropylene container; Eggs 4 in carton		

Table 8 – Field Parameters

Analyte Specifications for Field Parameters	
Field Parameters:	**Methods from 40 CFR Part 136.3, Table 1B, List of Approved Inorganic Test Procedures:**
Turbidity	as NTU, Nephelometric, EPA Method 180.1
Temperature	Degrees C, Thermometric, EPA Method 170.1
Hydrogen Ion	pH Units, Electrometric measurement, EPA Method 150.1
Dissolved Oxygen	mg/L, Electrode, EPA Method 360.1
Specific Conductance	micromhos/cm at 25 degrees C, Wheatstone Bridge, EPA Method 120.1
Chlorine	mg/L, Colorimetric Method, EPA SESD SOP SESDPROC-112-R2

Appendix A - Triangle Lake Forestry Pesticide Project – Field Sampling Data Form (Revision 1)

Date:_____ Team Number 1 2

Location_____

GPS at well Location: Lat_____ Long_____ Waypoint Number_____

Sample Number_____ Duplicate Sample Number_____

Time of Purge Start_____

Minute	TEMP °C	pH	COND	DO	Comments
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					

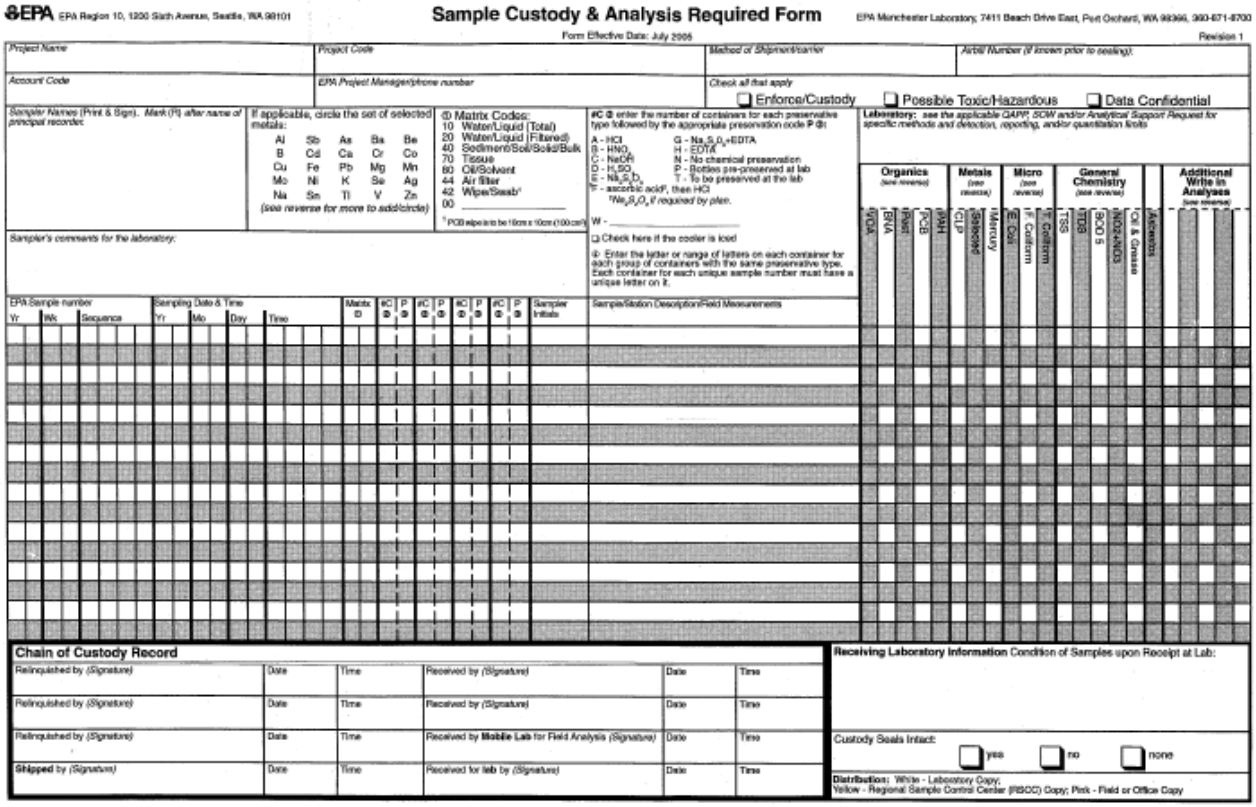
Time of Purge End_____

Water Sample Time_____ Sample Location Description_____

Soil Sample Time_____ Sample Location Description_____

Veg. Sample Time_____ Sample Location Description_____

Appendix B – Sample Custody and Analysis Required Form (Revision 1)



EPA Region 10, 1200 Sixth Avenue, Seattle, WA 98101

Sample Custody & Analysis Required Form

EPA Manchester Laboratory, 7411 Beach Drive East, Port Orchard, WA 98366, 360-871-8700

Form Effective Date: July 2006

Revision 1

Project Name | Project Code | Method of Shipment/carrier | Airbill Number (if known prior to sealing):

Account Code | EPA Project Manager/phone number | Check all that apply: ☐ Enforce/Custody ☐ Possible Toxic/Hazardous ☐ Data Confidential

Sampler Names (Print & Sign). Mark (*) after name of principal recorder.

If applicable, circle the set of selected metals:

Al	Sb	As	Ba	Be
B	Cd	Ca	Cr	Co
Cu	Fe	Pb	Mg	Mn
Mo	Ni	K	Se	Ag
Na	Sn	Tl	V	Zn

(see reverse for more to add/circle)

① Matrix Codes:
10 Water/Liquid (Total)
20 Water/Liquid (Filtered)
40 Sediment/Soil/Solid/Bulk
70 Tissue
80 Oil/Solvent
44 Air filter
42 Wipe/Swab[1]
00 _____

[1] PCB wipe is to be 10cm x 10cm (100 cm²)

#C ② enter the number of containers for each preservative type followed by the appropriate preservation code P ③:

A - HCl; B - HNO_3; C - NaOH; D - H_2SO_4; E - $Na_2S_2O_3$; F - ascorbic acid*, then HCl; G - $Na_2S_2O_3$+EDTA; H - EDTA *; N - No chemical preservation; P - Bottles pre-preserved at lab; T - To be preserved at the lab

* - ascorbic acid*, then HCl
*$Na_2S_2O_3$ if required by plan.

W - _____

☐ Check here if the cooler is iced

④ Enter the letter or range of letters on each container for each group of containers with the same preservative type. Each container for each unique sample number must have a unique letter on it.

Laboratory: see the applicable QAPP, SOW and/or Analytical Support Request for specific methods and detection, reporting, and/or quantitation limits

Organics (see reverse)				Metals (see reverse)		Micro (see reverse)		General Chemistry (see reverse)					Additional Write in Analyses (see reverse)		
VOA	BNA	Pest	PCB	PAH	CLP	Selenium	Mercury	E. Coli	F. Coliform	TSS	TDS	BOD 5	NO3+NO2	Oil & Grease	Asbestos			

Sampler's comments for the laboratory:

EPA Sample number Yr	Wk	Sequence	Sampling Date & Time Yr	Mo	Day	Time	Matrix ID	#C ②	P ③	#C ②	P ③	#C ②	P ③	#C ②	P ③	Sampler Initials	Sample/Station Description/Field Measurements

Chain of Custody Record

Relinquished by (Signature)	Date	Time	Received by (Signature)	Date	Time
Relinquished by (Signature)	Date	Time	Received by (Signature)	Date	Time
Relinquished by (Signature)	Date	Time	Received by **Mobile Lab** for Field Analysis (Signature)	Date	Time
Shipped by (Signature)	Date	Time	Received for **lab** by (Signature)	Date	Time

Receiving Laboratory Information Condition of Samples upon Receipt at Lab:

Custody Seals Intact: ☐ yes ☐ no ☐ none

Distribution: White - Laboratory Copy; Yellow - Regional Sample Control Center (RSCC) Copy; Pink - Field or Office Copy

Appendix C - Calibration and Use of the Horiba U-53G Multi Water Quality Checker OEAFIELDSOP-100

U.S. EPA REGION 10 Office of Environmental Assessment
Field Standard Operating Procedure

Title: Calibration and Use of the Horiba U-53G Multi Water Quality Checker

Effective Date: April 13, 2010

Document Number: OEAFIELDSOP-100

Review and Approvals

Signature _____ Date 10-7-10
Prepared by: Brent Richmond
Office of Environmental Assessment

Signature _____ Date 10-12-10
Peer Review: Don Matheny
Office of Environmental Assessment

Signature _____ Date 10/10/10
QA Manager: Gina Grepo-Grove
Office of Environmental Assessment

Signature _____ Date 10/13/10
Environmental Services Unit Manager: Mark Filippini
Office of Environmental Assessment

Table of Contents

1.0 Scope and Application

This standard operating procedure (SOP) provides guidance for the calibration and use of the Horiba U-53G Multi Water Quality Checker (U-53G) by qualified field inspectors, investigators, or other field personnel.

2.0 Summary of Test method

Following guidance from applicable analytical methods, regulations and publications, field personnel prepare a Quality Assurance Project Plan (QAPP) which includes specifications for appropriate field parameters to be measured by the U-53G. Based on the information in the QAPP, a service request is received by Field Support Center personnel and the instrument(s) are prepared and packed for shipment or pickup. Within OEA, selected members of the Environmental Services Unit on-site with Region 10 Manchester Environmental Laboratory provide this service. The U-53G is a multi-meter instrument that can simultaneously measure: pH, Temperature, Dissolved Oxygen, Conductivity, Total Dissolved Solids, Salinity, Turbidity, Oxidation Reduction Potential, Depth and GPS position. The U-53G is composed of the sensor unit, that is lowered into the water, and the hand held monitor, which allows the user to view and log sensor readings. The U-53G is calibrated using the methods below. It is designed for field use and applications include: rivers, lakes, and ground or tap water using a flow through cell.

3.0 Acronyms

COND - Conductivity
DO – Dissolved Oxygen
DOT – United States Department of Transportation
IATA – International Air Transport Association
NIST – National Institute of Standards and Technology
NTU – Nephelometric Turbidity Unit
OEA – Office of Environmental Assessment
ORP – Oxidation Reduction Potential
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
SOP – Standard Operating Procedure
TDS – Total Dissolved Solids
TURB- Turbidity

4.0 Interferences and Precautions

- Do not use instrument in magnetic fields. Measurement errors will occur.
- Do not immerse in alcohol, organic solvent, strong acid, strong alkaline or other similar solutions.
- Does not support measurement of samples containing fluorine.
- Do not subject to strong shocks. Do not drop.
- Ensure all calibration solutions are the same temperature as ambient air temperature.

- Holding the probe while calibrating may cause the internal probe temperature to rise causing a Dissolved Oxygen (DO) calibration error
- Do not drop instrument into water lower gently dropping will cause sensors to fail and my produce false readings or instrument failure.
- Do not use below depths of 30 meters. Sensor probes are only resistant to 30 meters. Below these depths may give false reading or instrument failure.
- Unit must be turned on for 20 minutes prior to calibrating or using DO measurements.
- For non-flowing water slowly move instrument up and down to induce flow over DO membrane.
- Oxidation Reduction Potential (ORP) standard solutions must be used within one hour at which point it may be transformed. For this reason ORP standard solutions may not be stored.
- When measuring ORP with low concentration of oxidants and reductants such as tap water, well water, or water that underwent treatment there may less repeatability and stability, in general.
- Always use the calibration cup provided. Other containers may create effects from ambient light that cause incorrect turbidity calibrations.
- Avoid turbidity measurement in direct sunlight, since the readout may be affected.

5.0 Safety

Use appropriate personal protective equipment including gloves and safety glasses when using calibration solutions.

6.0 Equipment and Supplies

1. U-53G multi Water Quality Checker, Control Unit and Sensor Probe
   a. pH Sensor
   b. ORP Sensor
   c. Reference electrode
   d. DO Sensor
   e. Turbidity Sensor

2. Calibration Cup(s) or appropriate containers as specified in sensor calibration section
3. pH reference internal solution
4. DO sensor internal solution set
5. DO Membrane spare parts set
6. Spanner wrench for DO sensor
7. Cleaning Brush
8. Back pack
9. Strap
10. Silicone Grease
11. Instruction Manuel
12. De-ionized water (DI water)
13. Bubbler
14. pH 4,7,10 Calibration Standard
15. Sodium Sulfite (zero DO)
16. ORP Solution (ORP Standard)
17. Auto Cal Solutions (0 NTU, pH 4, 4.40 mS/cm)

18. Level Two Solution (40 NTU, pH 6.86, 53.0 mS/cm)
19. Conductivity Calibration Solutions
20. Turbidity Calibration Solutions
21. Sensor Guard and Cap
22. LR14 alkaline dry cell batteries, C-size
23. Coin battery, CR-2032
24. Protective caps for DO, and pH Storage
25. Flow Cell assembly w/ appropriate size tubing
26. Scale
27. Beakers
28. Graduated Cylinders
29. Calibration Solution Disposal Container

7.0 Calibration Solutions and Standards

7.1 pH

- pH standard 4, 7, and 10, NIST traceable with expiration date.
- Auto Cal Solution, NIST traceable with expiration date

7.2 DO

- Zero DO solution is a Sodium Sulfite salt mixed with water (DI or Tap) at a ratio of 50g of sodium sulfite to 1000 ml of water.
- Span DO solution is water saturated with air. This is done by using a bubbler in a bath of water.
- Each solution shall be made fresh just prior to calibration or drift measurement.

7.3 ORP

- ORP standard powder (quinhydrone) mixed with 250 ml of deionized water and/or premixed ORP standard, NIST traceable with expiration date.

7.4 Conductivity

- Auto Cal Solution, NIST traceable with expiration date.
- Conductivity Standards, range of mS/cm, NIST traceable with expiration date.

7.5 Turbidity

- Auto Cal Solution, Referenced to Formazin, with expiration date.
- Turbidity Solutions, Range of NTU's, Auto Cal Solution, referenced to Formazin with expiration date.

8.0 Shipment and Storage

All equipment and calibration standards or solutions shall be shipped in compliance with the appropriate IATA or DOT requirements.

Calibration solutions and standards should be placed in Ziploc bags with taped caps and placed upright in the shipping container to minimize spilling during shipping.

If the equipment will be shipped or delivered, it will be placed into ice chests, cardboard boxes, or other appropriate containers with adequate cushioning material to prevent breakage and custody sealed to confirm not tapering of the standards has take place during shipment.

8.1 Storage For Short Term Use Only

- Prior to use inspect DO and pH for sensor caps are present and remove. DO is a white cap, the pH is a black cap.
- Replace caps filled with DI water between uses and for short term storage of less than two months. Note: Any water may be used in the field to ensure sensors remain wet. Replace water with DI water once available.
- Follow instruction manual for long term storage, which is defined as storage of longer than 2 months.

9.0 Quality Control and Documentation

Calibration and drift measurements (see section 10) should be conducted for each sampling event at a frequency outlined in the QAPP.

The calibration of the instrument sets each parameter to a given standard or solution.

Drift measurements should also be done at the end of each sampling event. Drift is the measurement of the standards or solutions used for calibration. This measurement will indicate if the instrument has "drifted" during the course instrument use. It is used as an indicator to ensure the probes are in good working order and the readings obtained during the sampling event are accurate.

All calibration solutions and standards shall be made fresh prior to use or be used within the labels expiration date.

All commercial standards or solutions shall be NIST traceable with expiration date or referenced to Formazin in the case of Turbidity.

Documentation of the measurements can occur by two methods: electronically by saving the measurements in the instrument for downloading after the sampling event and/or by logging readings manually into a log book. In either case follow the project specific QAPP or SOP. For example: Inspection type activities require a logbook with numbered pages and must be bound.

An equipment log book accompanies the U-53G. Each user shall enter users name, location of equipment use, date equipment used, calibrations that occurred, and other applicable information or failures. This log book remains with the U-53G and should not be used as the data log for the project.

10.0 Calibration and Standardization

The U-53G offers two methods to calibrate. Selection of the calibration method depends on how accurate and the quality of data needed. For general screening the Auto Calibration generally will meet the needs. The second method each parameter is manually calibrated with a zero and a span(s) standard. It is the operator's responsibility to know the limitations of this instrument, any EPA standard methods that apply, and the quality of data required by the project and/or QAPP. Operators should become familiar with the instruction manual for the U-53G. This outline will not duplicate the instruction manual verbatim.

10.1 Auto Calibration

- **PRIOR TO USE REMOVE DO AND pH SENSOR STORAGE CAPS**
- Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize and prior to calibration.
- Using the auto cal solution and fill calibration cup to the "with TURB" line located on the side of the cup.
- Place calibration cup into black calibration cup to eliminate light sources.
- Press the CAL button or right arrow to the Calibration Tab.
- Press the down arrow to Auto Calibration
- Press Enter and follow instructions on screen.
- Instructions should then direct you to place sensors into calibration cup.
- Monitor values of the sensors once stabilized press ENTER to start calibration
- Do not remove the unit from calibration solution the will display "-----"until finished.
- The display will direct you to press MEAS to exit to measurement screen.
- If error occurs make note of error code and refer to instruction manual, Section 4.6 Troubleshooting.

10.2 Manual Calibration

Manual Calibration is used to individually calibrate sensors. It is used to ensure good measurement precision throughout all measurement ranges. It should be noted the temperature calibration should follow the most current version of the Inorganic Chemistry Support Equipment Monitoring SOP along with procedures outlined below. Since the H-53G allows temperature calibration, the temperature obtained from the Inorganic Chemistry Support Equipment Monitoring SOP can be entered directly into the H-53G and no correction factor is necessary.

- **PRIOR TO USE REMOVE DO AND pH SENSOR STORAGE CAPS**
- Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize and prior to calibration.
- Press the CAL button or right arrow to the Calibration Tab.

10.2.1 Temperature (Performed yearly or as needed)

- Following the Inorganic Chemistry Support Equipment Monitoring SOP
- Press the CAL button or right arrow to the Calibration Tab.
- Press the down arrow to Manual Calibration
- Move the cursor to Temp and press ENTER
- Press the up and down arrow to adjust value
- Once value is stable press ENTER to start calibration
- When calibration is finished press ENTER for measurement mode

10.2.2 pH

- Calibrate the zero point (pH 7) first, rinse calibration cup 2-3 times with DI water,
- then fill with pH 7 standard solution to the w/TURB reference line.
- Rinse the senor probe 2-3 times with DI water to remove any dirt and then submerge into calibration cup
- Press CAL or from the Calibration Tab and press the down arrow to "Manual Calibration"
- Move the cursor to pH and press ENT
- Set the number of calibration points for two
- Press the up and down to set the pH value of 7.00 for the pH 7 standard, reference temperature vs. pH standard value chart
- Check that the value has stabilized then press ENTER to start calibration.
- Once complete press Enter to continue to span cal.
- Rinse calibration cup 2-3 times with DI water
- Fill cup with pH 4 or pH 10 to the w/TURB reference line. Place sensor into calibration cup.
- Check that the value has stabilized, reference temperature vs. pH standard value chart, press ENTER to start calibration.
- When calibration complete press ENTER to return to calibration parameter menu.

10.2.3 ORP

- Rinse the sensor probe and calibration cup 2 to 3 times with DI water
- Place sensor probe into solution.
- Fill calibration cup with ORP standard to the w/TURB reference line.
- Press the units CAL key to set calibration mode
- Press down arrow to Manual Calibration and press ENTER
- Move the cursor to ORP and press ENTER
- Press the up or down arrow to adjust the mV to match the standard mV for the given standard temperature.
- Check the measurement value has stabilized and press ENTER
- Once complete press ENTER to return to the calibration selection screen.

10.2.4 Conductivity (COND)

- Rinse the sensor probe 2 to 3 times with DI water to remove any dirt
- Remove as much moisture from the sensor, the zero calibration is done in air.
- Press the units CAL key to set calibration mode
- Press the down arrow to Manual Calibration and press ENTER
- Move the cursor to COND and press ENTER
- Press up or down arrow to select number of calibration points (2 or 4) press ENTER. Depending on the number of calibration points proceed and stop with the number of steps below. The solutions can be modified to bracket the waters being tested.
- Press up or down arrow to set COND to 0.00 mS/cm
- Check the measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to first span calibration procedure
- Wash the sensor probe and calibration cup 2 to 3 times with DI water.
- Fill calibration cup to the w/TURB reference line with a 0.00 to 0.999mS/cm standard.
- Place sensor probe into solution.
- Press up or down to set value to standard.
- Check to ensure measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to the second span calibration procedure.
- Wash the sensor probe and calibration cup 2 to 3 times with DI water.
- Fill calibration cup to the w/TURB reference line with a 1.00 to 9.99 mS/cm standard.
- Place sensor probe into solution.
- Press up or down to set value to standard.
- Check to ensure measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to the third span calibration procedure.
- Wash the sensor probe and calibration cup 2 to 3 times with DI water.
- Fill calibration cup to the w/TURB reference line with a 10.0 to 100.0 mS/cm standard.
- Place sensor probe into solution.
- Press up or down to set value to standard.
- Check to ensure measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to the calibration parameter screen.

10.2.5 Turbidity (TURB)

- Rinse the sensor probe and calibration cup 2 to 3 times with DI water
- Fill calibration cup with zero standard to the w/TURB reference line.
- Place calibration cup into black calibration cup to eliminate light sources.
- Place the sensor probe into calibration cup with standard.
- Press the units CAL key to set calibration mode
- Press down arrow to Manual Calibration and press ENTER
- Move the cursor to TURB and press ENTER
- Press the up or down arrow to select number of calibration points

- Press up or down to set value to the 0.00 standard.
- Check to ensure measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to the first span calibration procedure.
- Wash the sensor probe and calibration cup 2 to 3 times with DI water.
- Fill calibration cup to the w/TURB reference line with a 0.1 to 10.0 NTU standard.
- Place sensor probe into solution.
- Press up or down to set value to standard.
- Check to ensure measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to the second span calibration procedure.
- Wash the sensor probe and calibration cup 2 to 3 times with DI water.
- Fill calibration cup to the w/TURB reference line with a 10.0 to 100.0 NTU standard.
- Place sensor probe into solution.
- Press up or down to set value to standard.
- Check to ensure measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to the third span calibration procedure.
- Wash the sensor probe and calibration cup 2 to 3 times with DI water.
- Fill calibration cup to the w/TURB reference line with a 100 to 1000 NTU standard.
- Place sensor probe into solution.
- Press up or down to set value to standard.
- Check to ensure measurement value has stabilized and press ENTER
- Once complete press ENTER to proceed to the calibration parameter screen.

10.2.6 Dissolved Oxygen (DO)

- Prepare standard solutions
    - Add 50 g of sodium sulfite to 1000 ml of water and stir to dissolve. Generally, 1000 ml of solution is not needed for one calibration. Use the ratios 12.5 per 250 ml or 25 g per 500 ml depending on number of instruments that will be calibrated.
    - Pour 1 to 2 liters of water into a suitable container and feed air into water using a bubbler or air pump until it is oxygen saturated.
- Rinse the sensor probe 2 to 3 times with DI water
- Place sensor into zero standard into the calibration cups (black and clear).
- Fill solution over the DO sensor slot in the cup, this is the area between the black cup and the clear cup.
    - Press the units CAL key to set calibration mode.
    - Press down arrow to Manual Calibration and press ENTER.
- Move the cursor to %DO and press ENTER.

- Press the up or down arrow to selecting two (2) for the number of calibration points.
- Press the up or down arrow to adjust the %DO to match zero.
- Check to ensure the measurement value has stabilized and then press ENTER.
- Once complete press ENTER to proceed to span calibration procedure.
- Place sensor guard on sensor unit to protect sensors.
- Place sensor probe into oxygen saturated bath solution.
- Press up or down to set value to 100%.
- Check to ensure measurement value has stabilized and press ENTER.
- Once complete press ENTER to return to the calibration selection screen.

10.2.7 Water Depth (DEPTH)

- Rinse the sensor probe 2 to 3 times with DI water
- Press the CAL button or right arrow to the Calibration Tab.
- Press the down arrow to "Manual Calibration"
- Move the cursor to Depth and press ENTER.
- Press the up and down arrow to adjust value to 0.00 meters
- Once value is stable press ENTER to start calibration
- When calibration if finished press ENT for measurement mode

10.3 Drift Measurements

- Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize and prior to calibration.
- Using the calibration cup or appropriate container with each successive standard or solution and measure the value by pressing the MEAS button.
- Record and compare to the standard or solution value. The difference is the drift value.
- A percentage of drift can be calculated by subtracting the drift measurement from the standard and dividing by the standard value. This percentage can be used to monitor the equipments sensors and to ensure data quality and repeatability.

11.0 Measurement and Data Collection

Any instrument requests should be made to Field Services staff via phone or email with **AT LEAST TWO WEEKS** notice to ensure adequate standards are available and equipment is serviceable.

- **PRIOR TO USE, REMOVE DO AND pH SENSOR STORAGE CAPS**

11.1 Single Measurement

- Turn unit on using the power button. For DO sensors the unit must remain on for 20 minutes to stabilize, use the internal clock and DO reading, and prior to calibration.

- Check that each sensor and sensor guard is mounted.
- Press the right or left arrow to the Single Measurement tab or screen.
- **NOTE: DO NOT** press MEAS key while unit in the air, this will cause turbidity wiper failure.
- Submerge sensor probe in the sample, gently shake them to remove air bubbles form sensors.
- For non-flowing water gently move sensor probe up and down at a rate of 20 to 30 cm a second.
- When non-turbidity values are stable press MEAS to acquire values.
- Press ENTER to save the values or write values in logbook.
- Press ESC to cancel operation.
- Continue to next measurement or press and hold PWR button to power off.

11.3 GPS Information

- Press the right arrow to the Data Operations tab or screen.
- Press the down arrow to move the cursor to GPS Information then press ENTER.
- When position data exists it will be displayed, if no data is displayed data has not been received.

11.4 Data Operations

- Press the right arrow to the Data Operations tab or screen.
- Press the down arrow to move the cursor to View Data then press ENTER.
- Move the cursor to Site/Date/All and then press ENTER. The Site name or Date will need to be entered depending on your selection. All data will display with the most recent first.
- Press the up or down arrow to scroll though data.
- To delete data from the Data Operation tab or screen cursor to Delete Data and press ENTER.
- Press the left arrow to confirm Yes and press ENTER. All data will be deleted.
- It is the operator's responsibility to save data obtained during the sampling event. The unit may be cleared of all data upon return either by the ESU personnel or the next user.

11.5 Calibration Record Check

- Press the right arrow to the Data Operations tab or screen.
- Press the down arrow to move the cursor to Calibration Record then press ENTER
- The latest calibration record will be displayed. Scroll by pressing the down arrow

11.6 Sensor Information

- Press the right arrow to the Information tab or screen.
- When sensor probe is normal "All sensors are available." will appear.

- When a sensor has a problem a message will be generated. Refer to the instruction manual for troubleshooting information. (Section 4.6 pages 89 to 94)

12.0 Calculations

Principals of measurements for each parameter are outlined in the instruction manual for the instrument. Refer to pages 100-109 or Sections 6.3 thru 6.11

Some of the calibrations solutions need to be mixed. Refer to the calibration section of the instruction manual or section 10 above for mixture ratios.

Refer to appropriate calibration chart for standard value for a given temperature. Usually located on the bottle and/or U-53G instruction manual. There may be a need to interpolate between temperature readings to obtain the correct standard value.

Percentage of Drift = (Calibration Standard – Drift Measurement) / Calibration Standard

13.0 Waste Management and Pollution Prevention

Recycle used containers when possible both in the field and in accordance with Manchester Environmental Laboratory's Waste Management and Pollution Prevention Programs when at the Manchester Lab Facility. Disposal of calibration solution should only occur in an appropriate manner, i.e. not on the ground or in a body of water. All waste solutions should be saved and disposed of once you have returned from the field.

14.0 References

Horiba U-50 series, Multi Water Quality Checker, Instruction Manual, April 2009.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), current version.

Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association and Water Environment Federation, latest edition.

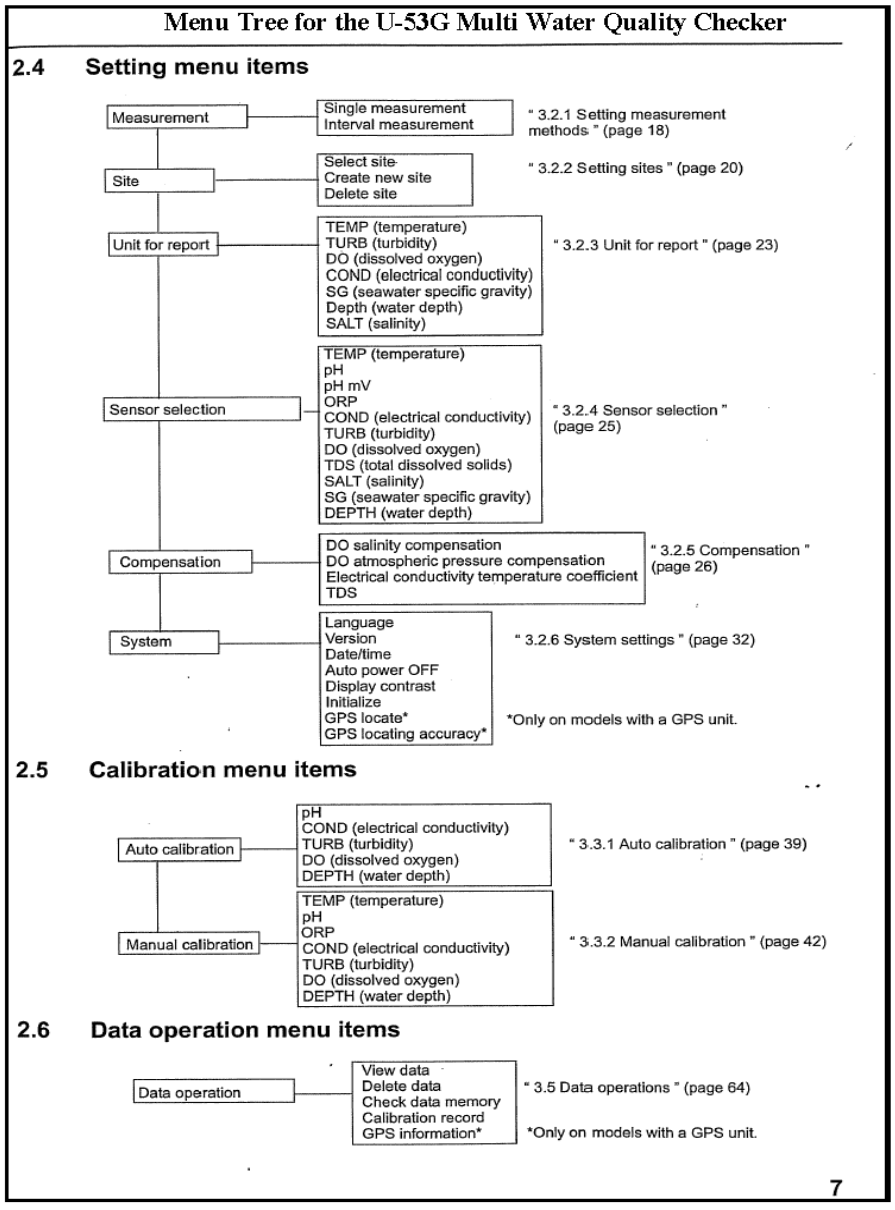
Code of Federal Regulations, Title 40 (40 CFR), Part 136.3, Table IB.

Pollution Prevention Plan of the Manchester Environmental Laboratory," USEPA Region 10,current version.

Manchester Environmental Laboratory Dangerous Waste Disposal Manual," USEPA Region 10, current version.

15.0 Diagram

15.1 Menu Tree for the U-53G



16.0 Change History

Original revision. No changes to date.

Appendix D – Field Measurement of Total Residual Chlorine SESDPROC-112-R2

COPY

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Measurement of Total Residual Chlorine**

Effective Date: April 20, 2011 | **Number:** SESDPROC-112-R2

Author

Name: John Williams
Title: Environmental Scientist
Signature: John Williams Date: 4/14/11

Approvals

Name: Archie Lee
Title: Chief, Enforcement and Investigations Branch
Signature: Date: 4/14/11

Name: Bill Cosgrove
Title: Chief, Ecological Assessment Branch
Signature: Date: 4/14/11

Name: Liza Montalvo
Title: Field Quality Manager, Science and Ecosystem Support Division
Signature: Date: 4/14/2011

COPY

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-112-R2, *Field Measurement of Total Residual Chlorine,* Replaces SESDPROC-112-R1 **Cover Page:** The Author was changed from Ron Phelps to John Williams. The EIB Branch Chief was changed from Antonio Quinones to Archie Lee. The FQM was changed from Laura Ackerman to Liza Montalvo. **Section 1.2:** Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Omitted the reference to the H: drive of the LAN. **Throughout the document:** Replaced "mls" with "ml," and "insure" with "ensure." Corrected typographical errors.	April 20, 2011
SESDPROC-112-R1, *Field Measurement of Total Residual Chlorine,* Replaces SESDPROC-112-R0 **Cover Page:** Author was changed from John Williams to Ron Phelps. **Revision History** Changed Field Quality Manager to Document Control Coordinator. **Section 1.3** Changed Field Quality Manager to Document Control Coordinator. **Section 1.5** Added second paragraph. **Section 2** Removed last sentence in paragraph 3. **Section 3.1 General** Added chemical name for DPD. **Section 3.2** Deleted MDL Study.	June 13, 2008
SESDPROC-112-R0, *Field Measurement of Total Residual Chlorine*, Original Issue	October 19, 2007

COPY

TABLE OF CONTENTS

COPY

1 General Information

1.1 Purpose

This document describes methods and considerations to be used and observed when conducting field measurements of total residual chlorine in surface water and wastewater effluent.

1.2 Scope/Application

On the occasion that SESD field investigators determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another method must be used to obtain a measurement of total residual chlorine, the alternate procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

USEPA Region 4 Environmental Assessment Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

SESD Safety, Health and Environmental Management Program (SHEMP) Manual, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108), Most Recent Version

Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998

Hach Company Pocket Colorimeter Chlorine Manual, Hach, 1994

Code of Federal Regulations, 40 CFR Part 136, Appendix B

1.5 Safety Precautions

Refer to the SESD Safety, Health and Environmental Management Program Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, safety glasses, and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

2 Quality Control

All total residual chlorine meters will be maintained and operated in accordance with the manufacturer's instructions and SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108. The following are general guidelines for maintaining total residual chlorine meters:

- Each meter should be visually inspected before and after each use.
- Check the battery strength.
- Ensure that the reagents are fresh before field trips.

Before a meter is taken to the field, it shall be properly calibrated and verified, according to Section 3.2.2 of this procedure, to ensure it is operating properly. These calibration verifications and maintenance procedures shall be documented and maintained in a logbook.

The ambient temperature in the immediate vicinity of the meter should be measured and recorded in the field logbook to ensure the instrument is operated within the manufacturer's specified range of operating temperatures.

If, at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

3 Field Measurement of Total Residual Chlorine

3.1 General

The chlorination of water supplies and polluted water serves primarily to destroy or deactivate disease-producing microorganisms. Chlorine applied to water in its molecular or hypochlorite form initially undergoes hydrolysis to form free chlorine consisting of aqueous molecular chlorine, hypochlorous acid, and hypochlorous ion. Free chlorine reacts with ammonia and certain nitrogenous compounds to form combined chlorine. With ammonia, chlorine reacts to form the chloramines: monochloramine, di-chloramine, and nitrogen tri-chloride. Total residual chlorine is the sum of the combined available residual chlorine and the free available residual chlorine remaining after a given contact time.

Chlorination may produce adverse effects. Potentially carcinogenic chloroorganic compounds such as chloroform may be formed. To fulfill the primary purpose of chlorination and to minimize any adverse effects, it is essential that proper testing procedures be used. Several methods for measurement of total residual chlorine are available including iodometric methods, amperometric titration methods, and *N*,*N*-diethyl-*p*-phenylenediamine (DPD) methods. This operating procedure will discuss the DPD Colorimetric Method.

3.2 Initial Laboratory Verification

See Standard Methods for the Examination of Water and Wastewater, Method 4500 Cl for directions in preparing the ASTM Standard D1193 "Consumption of Potassium Permanganate."

- Potassium permanganate stock – Prepare a stock solution containing 891 mg/1000 ml. Keep stock cool and store in the dark.

- Potassium permanganate intermediate stock 10 ppm – Prepare intermediate stock solution containing 10 mg/l $KMnO_4$ by diluting 10 ml of stock solution to 1 liter.

  Note: The intermediate stock should be stable for approximately 5 days if kept cool and away from light.

- Potassium permanganate calibration standards – Prepare calibration standards from the intermediate stock solution and/or KMnO4 calibration standard solutions for each day of use. The calibration standards are good for about 2 hours and will fade rapidly (within 15 minutes) if chlorine demand-free water is not used.

Calibration Standard (mg/l)	ml of Intermediate Stock/100 ml
0.05	10.0 of 0.5 mg/l std.
0.10	10.0 of 1.0 mg/l
0.5	5.0 of 10 mg/l
1.0	10.0 of 10 mg/l
2.0	20.0 of 10 mg/l

When checking the instrument verification with the five standards, a linear regression should be performed and the result should correlate to 0.995 or better. Records of this verification will be maintained by ASB staff.

3.2.1 Field Calibration Verification *for Regulatory Monitoring*

For regulatory monitoring, the calibration curve must be verified onsite with a minimum of three points: a blank and two known standards that bracket the expected sample concentrations. The meter's internal calibration scale must be verified daily using a blank, one **high,** and one **low** standard representative of the meter's linear working range. These standard checks must agree to within ±10% of the original curve or a new curve must be prepared. Verification data should be recorded and maintained on file. Use either 1-cm or 2.5-cm cells depending upon concentration range of the sample.

DPD total residual chlorine powder packets – The packets deteriorate in the presence of moisture or high temperature. The packets should be discarded if they have caked or have turned brown, or the expiration date has expired.

Note: Always wear gloves when handling the DPD oxalate, and do not ingest. If accidentally spilled on skin, rinse off immediately. Additionally, EPA might elect to use the facility's equipment to verify chlorine residual values that may be outside of the permit limits. The procedure and equipment used to document the findings should be written in the field logbook.

3.3 Sample Measurement Procedure *for EPA Field Screening*

Total or free residual chlorine measurements should always be conducted within 15 minutes of sample collection. The pH of the source should be checked and documented in the field logbook. When using the DPD colorimetric method for total residual chlorine, gel standards can be used for meter verification. The gel standards should be verified by the SESD laboratory personnel (ASB) to ensure accuracy prior to use. The tolerance ranges for the gel standards used for verification are as follows: 0.2 ± 0.09; 0.81 ± 0.10; 1.53 ± 0.14. If other gel standards are used, the tolerance ranges must also be verified by the SESD laboratory personnel.

For sample screening purposes, the following steps should be followed using a 2.5 cm sample cell for total residual chlorine concentrations ranging between 0 - 2 mg/l.

1. Fill a clean sample cell to the 10-ml mark with a sample blank. Cap the sample cell.

2. Press the **Power** key to turn the meter on.

3. Remove the meter's cover. Wipe off any excess liquid and fingerprints from the sample cell. Place the blank in the cell holder with the diamond mark facing the keypad. Fit the meter cover over the cell compartment to completely cover the cell.

4. Press the **ZERO** button. The display will show "- - - -" then go to "0.00". Record the blank's value in the logbook. Remove the blank from the cell holder.

5. Fill a second 10-mL cell to the 10-mL line with the sample.

6. Open a DPD total chlorine powder packet and add the contents to the sample cell.

7. Replace the cap on the sample cell and swirl or mix for approximately 20 seconds. **Note**: Wipe off any excess liquid and fingerprints from the sample cell.

8. Wait three to six minutes (3-6) after adding the DPD.

9. Press **READ/ENTER**. The instrument will show "- - - -" followed by the actual results in mg/L chlorine. Record the sample's value in the logbook.

Also for screening purposes, all of the above steps should be followed using a 1.0 cm sample cell for total residual chlorine concentrations ranging between 0 - 3.5 mg/l.

3.4 Units

Measurements for total residual chlorine are reported in mg/l.

3.5 Limitations

Do not use with or in the presence of any oxidizing agents including bromine, chlorine dioxide, iodine, permanganate, hydrogen peroxide, and ozone. Sample color and turbidity may also interfere.

Appendix E – Potable Water Supply Sampling
SESDPROC-305-R1

COPY

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Potable Water Supply Sampling**

Effective Date: November 1, 2007 | **Number:** SESDPROC-305-R1

Authors

Name: Maria Labrador
Title: Environmental Engineer
Signature: M Labrador Date: 11/2/07

Approvals

Name: Antonio Quinones
Title: Chief, Enforcement and Investigations Branch
Signature: Date: 11/02/07

Name: Laura Ackerman
Title: Field Quality Manager, Science and Ecosystem Support Division
Signature: Laura Acker Date: 11/01/07

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Field Quality Manager.

History	Effective Date
SESDPROC-305-R1, *Potable Water Sampling*, replaces SESDPROC-305-R0 **General** Updated referenced operating procedures due to changes in title names and/or to reflect most recent version. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch **Section 1.3** Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities. **Section 1.4** Alphabetized and revised the referencing style for consistency. **Section 1.5.1** Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual.	November 1, 2007
SESDPROC-305-R0, Potable Water Supply Sampling, Original Issue	February 05, 2007

TABLE OF CONTENTS

COPY

Contents

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting potable water supply samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling potable water supply samples in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a potable water supply sample, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the H: drive of the SESD local area network. The Field Quality Manager (FQM) is responsible for ensuring the most recent version of the procedure is placed on the H: drive and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

Puls, Robert W., and Michael J. Barcelona. Filtration of Ground Water Samples for Metals Analysis. *Hazardous Waste and Hazardous Materials* 6(4): 385-393 (1989).

Puls, Robert W., Don A. Clark, and Bert Bledsoe. Metals in Ground Water: Sampling Artifacts and Reproducibility. *Hazardous Waste and Hazardous Materials* 9(2): 149-162 (1992).

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-104, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field pH Measurements, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurements, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Temperature Measurements, SESDPROC-102, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurements, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, Most Recent Version

SESD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

US EPA. 1975. Handbook for Evaluating Water Bacteriological Laboratories. Office of Research and Development (ORD), Municipal Environmental Research Laboratory. Cincinnati, Ohio.

US EPA. 1977. Sampling for Organic Chemicals and Microorganisms in the Subsurface, EPA-600/2-77-176

US EPA. 1978. Microbiological Methods for Monitoring the Environment, Water and Wastes, ORD, Environmental Monitoring and Support Laboratory. Cincinnati, Ohio

US EPA. 1995. Ground Water Sampling - A Workshop Summary. Proceedings from the Dallas, Texas November 30 - December 2, 1993 Workshop. Office of Research and Development Robert S. Kerr Environmental Research Laboratory. EPA/600/R-94/205.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version

US EPA. April 13, 1981. Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples. Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273)

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting potable water supply samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting potable water supply samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Always sample from the anticipated cleanest, i.e., least contaminated location, to the most contaminated location. This minimizes the opportunity for cross-contamination to occur during sampling.
- Collected samples must remain in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) and/or International Air Transportation Association (IATA) hazardous materials shipping requirements.

- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and stored in a secure place.

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

Potable water supply samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vials may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two week holding time, whereas unpreserved samples have only a seven day holding time. In the great majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations however, it may be necessary to use the unpreserved vials. For example, if the potable water supply has a high amount of dissolved limestone, i.e., is highly calcareous, there will most likely be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

2.2 Special Precautions for Trace Contaminant Potable Water Supply Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.
- Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating

Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

The following procedures should be followed when collecting samples from potable water supplies:

1. Potable water supply samples will typically be collected from a tap or spigot located at or near the well head or pump house and before the water supply is introduced into any storage tanks or treatment units. Efforts should be made to reduce the flow from either the tap or spigot during sample collection to minimize sample agitation.
2. During sample collection, make sure that the tap or spigot does not contact the sample container.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 2.1, Volatile Organic Compound Analysis). All other sample containers must be filled with an allowance for ullage.
4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by Analytical Support Branch (ASB) personnel prior to departure for the field investigation. All other chemical preservatives required for the remaining suite of analytes will be supplied by ASB personnel and will be added to the samples by SESD field personnel or other authorized persons. The adequacy of sample preservation will be checked after the addition of the preservative for all samples except for the samples collected for VOC analysis. Additional preservative should be added to achieve adequate preservation. Preservation requirements for groundwater samples are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version.

2.4 Quality Control

Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by any sampling equipment.

2.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in the SESD Operating Procedure for Control of Records (SESDPROC-002). Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in

accordance with the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005) and SESD Operating Procedure Logbooks (SESDPROC-010).

3 Potable Water Supply Sampling – Sample Site Selection

3.1 General

The following should be considered when choosing the location to collect a potable water sample:

- Taps selected for sample collection should be supplied with water from a service pipe connected directly to a water main in the segment of interest.

- Whenever possible, choose the tap closest to the water source, and prior to the water lines entering the residence, office, building, etc., and also prior to any holding or pressurization tanks.

- The sampling tap must be protected from exterior contamination associated with being too close to a sink bottom or to the ground. Contaminated water or soil from the faucet exterior may enter the bottle during the collection procedure since it is difficult to place a bottle under a low tap without grazing the neck interior against the outside faucet surface. If the tap is too close to the ground for direct collection into the appropriate container, it is acceptable to use a smaller container to transfer sample to a larger container. The smaller container should be made of glass or stainless steel, and should be decontaminated to the same standards as the larger container.

- Leaking taps that allow water to discharge from around the valve stem handle and down the outside of the faucet, or taps in which water tends to run up on the outside of the lip, are to be avoided as sampling locations.

- Disconnect any hoses, filters, or aerators attached to the tap before sampling. These devices can harbor a bacterial population if they are not routinely cleaned or replaced when worn or cracked.

- Taps where the water flow is not constant should be avoided because temporary fluctuation in line pressure may cause clumps of microbial growth that are lodged in a pipe section or faucet connection to break loose. A smooth flowing water stream at moderate pressure without splashing should be used. The sample should be collected without changing the water flow. It may be appropriate to reduce the flow for the volatile organic compounds aliquot to minimize sample agitation.

Occasionally, samples are collected to determine the contribution of system-related variables (e.g., transmission pipes, water coolers, water heaters, holding tanks, pressurization tanks, etc.) to the quality of potable water supplies. In these cases, it may be necessary to ensure that the water source has not been used for a specific time interval (e.g., over a weekend or a three- or four-day holiday period). Sample collection may consist of collecting a sample of the initial flush, collecting a sample after several

minutes, and collecting another sample after the system being investigated has been completely purged.

When sampling for bacterial content, the sample container should not be rinsed before use due to possible contamination of the sample container or removal of the thiosulfate dechlorinating agent (if used). When filling any sample container, care should be taken that splashing drops of water from the ground or sink do not enter into either the bottle or cap.

When sampling at a water treatment plant, samples are often collected from the raw water supply and the treated water after chlorination.

Obtain the name(s) of the resident or water supply owner/operator, the resident's exact mailing address, and the resident's home and work telephone numbers. The information is required so that the residents or water supply owner/operators can be informed of the results of the sampling program.

4 Potable Water Supply Sampling Methods - Purging

4.1 General

4.1.1 Purging and Purge Adequacy

Purging is the process of removing stagnant water immediately prior to sampling. In order to determine when an adequate purge has occurred, field investigators should monitor the pH, specific conductance, temperature, and turbidity of the water removed during purging. For potable water supply sampling it is recommended to purge the system for at least 15 minutes when possible.

An adequate purge is achieved when the pH, specific conductance, and temperature of the potable water have stabilized and the turbidity has either stabilized or is below 10 Nephelometric Turbidity Units (NTUs) (twice the Primary Drinking Water Standard of 5 NTUs). Although 10 NTUs is normally considered the minimum goal for most water sampling objectives, lower turbidity has been shown to be easily achievable in most situations and reasonable attempts should be made to achieve these lower levels. Stabilization occurs when, for at least three consecutive measurements, the pH remains constant within 0.1 Standard Unit (SU), specific conductance varies no more than approximately 10 percent, and the temperature is constant. There are no set criteria establishing how many total sets of measurements are adequate to document stability of parameters.

If, after 15 minutes, the in situ chemical parameters have not stabilized according to the above criteria, additional water can be removed. If the parameters have not stabilized after 15 minutes, it is at the discretion of the project leader whether or not to collect a sample or to continue purging.

4.2 Potable Water Samples Collected from Wells with In-Place Plumbing

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. The objective of purging wells with in-place pumps is the same as with monitoring wells without in-place pumps, i.e., to ultimately collect a water sample representative of aquifer conditions. Among the types of wells identified in this section, two different approaches are necessary.

A permanent well with an in-place pump should, in all respects, be treated like a well without a pump. One limitation is that in most cases the in-place pump is "hard" mounted, that is, the pump is suspended in the well at a pre-selected depth and cannot be moved up or down during purging and sampling. In these cases, well volumes are removed, parameters are measured and the well is sampled from the pump discharge, after volume removal and parameter conditions have been met.

In the case of the other types of wells, i.e., municipal, industrial and residential supply wells, however, not enough is generally known about the construction aspects of the wells to apply the same criteria as used for monitoring wells, i.e., 3 to 5 well volumes. The volume to be purged in these situations, therefore, depends on several factors: whether the pumps are running continuously or intermittently and whether or not any storage/pressure tanks are located between the sampling point and the pump. The following considerations and procedures should be followed when purging wells with in-place plumbing under the conditions described.

4.2.1 Continuously Running Pumps

If the pump runs more or less continuously, no purge (other than opening a valve and allowing it to flush for a few minutes) is necessary. If a storage tank is present, a spigot, valve or other sampling point should be located between the pump and the storage tank. If not, locate the valve closest to the tank. Measurements of pH, specific conductance, temperature, and turbidity are recorded at the time of sampling.

4.2.2 Intermittently or Infrequently Running Pumps

If the pump runs intermittently or infrequently, best judgment should be utilized to remove enough water from the plumbing to flush standing water from the piping and any storage tanks that might be present. Generally, under these conditions, 15 to 30 minutes will be adequate. Measurements of pH, specific conductance, temperature and turbidity should be made and recorded at intervals during the purge and the final measurements made at the time of sampling should be considered the measurements of record for the event.

4.3 Investigation Derived Waste

Purging generates quantities of purge water or investigation derived waste (IDW), the disposition of which must be considered. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for guidance on management or disposal of this waste.

5 Potable Water Supply Sampling Methods – Sampling

5.1 General

Sampling is the process of obtaining, containerizing, and preserving (if required) a potable water supply water sample after the purging process is complete. It is recognized that there are situations, such as industrial or municipal supply wells or private residential wells, where a well may be equipped with a dedicated pump from which a sample would not normally be collected. Discretion should always be used in obtaining a sample.

5.2 Collecting Samples from In-Place Plumbing

Samples should be collected following purging from a valve or cold water tap as near to the well as possible, preferably prior to any storage/pressure tanks or physical/chemical treatment system that might be present. Remove any hose that may be present before sample collection and reduce the flow to a low level to minimize sample disturbance, particularly with respect to volatile organic constituents. Samples should be collected directly into the appropriate containers (see the ASBLOQAM for a list of containers). It may be necessary to use a secondary container, such as a clean 8 oz. or similar size sample jar or a stainless steel scoop, to obtain and transfer samples from spigots with low ground clearance. All measurements for pH, specific conductance, temperature, and turbidity should be recorded at the time of sample collection.

1. Ideally, the sample should be collected from a tap or spigot located at or near the well head or pump house and before the water supply is introduced into any storage tanks or treatment units. If the sample must be collected at a point in the water line beyond pressurization or holding tank, a sufficient volume of water should be purged to provide a complete exchange of fresh water into the tank and at the location where the sample is collected. If the sample is collected from a tap or spigot located just before a storage tank, spigots located inside the building or structure should be turned on to prevent any backflow from the storage tank to the sample tap or spigot. It is generally advisable to open several taps during the purge to ensure a rapid and complete exchange of water in the tanks.

2. Purge the system for at least 15 minutes when possible. During the purge period, obtain at least three sets of readings as follows: after purging for several minutes, measure the pH, specific conductivity, temperature and turbidity of the water. Continue to measure these parameters to assess for stabilization.

3. After three sets of readings have been obtained, samples may be collected. If stabilization has not occurred after the 15-minute purge period, it is at the discretion of the project leader to collect the sample or continue purging and monitoring the parameters. This would depend on the condition of the system and the specific objectives of the investigation.

5.3 Sample Preservation

After sample collection, all samples requiring preservation must be preserved as soon as practical. Consult the ASBLOQAM for the correct preservative for the particular analytes of interest. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

5.4 Special Sample Collection Procedures

5.4.1 Trace Organic Compounds and Metals

Special sample handling procedures should be instituted when trace contaminant samples are being collected. All sampling equipment which comes into contact with the water must be cleaned in accordance with the cleaning procedures described in SESD Operating Procedure for Field Equipment Cleaning and Decontamination, (SESDPROC-205).

5.4.2 Filtering

As a standard practice, potable water samples will not be filtered for routine analysis. Filtering will usually only be performed to determine the fraction of major ions and trace metals passing the filter and used for flow system analysis and for the purpose of geochemical speciation modeling. Filtration is not allowed to correct for improperly designed or constructed wells, inappropriate sampling methods, or poor sampling technique.

When samples are collected for routine analyses and are filtered, both filtered and non-filtered samples will be submitted for analyses. Samples for organic compounds analysis should not be filtered. Prior to filtration of the water sample for any reason other than geochemical speciation modeling, the following criteria must be demonstrated to justify the use of filtered samples for inorganic analysis:

1. The water samples were collected using sampling techniques in accordance with this section, and the water samples were analyzed in accordance with USEPA approved methods.

2. Efforts have been undertaken to minimize any persistent sample turbidity problems.

3. Turbidity measurements should be taken during purging and sampling to demonstrate stabilization or lack thereof. These measurements should be documented in the field notes. If the water sample appears to have either a chemically-induced elevated turbidity, such as would occur with precipitate

formation, or a naturally elevated colloid or fine, particulate-related turbidity, filtration will not be allowed.

If filtration is necessary for purposes of geochemical modeling or other **pre-approved** cases, the following procedures are suggested:

1. Accomplish in-line filtration through the use of disposable, high capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus. The high capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration. If a membrane filter is utilized, a minimum diameter of 142 mm is suggested.

2. Use a 5 µm pore-size filter for the purpose of determining the colloidal constituent concentrations. A 0.1 µm pore-size filter should be used to remove most non-dissolved particles.

3. Rinse the cartridge or barrel-type filter with 500 milliliters of the solute (potable water to be sampled) prior to collection of sample. If a membrane filter is used, rinse with 100 milliliters of solute prior to sample collection.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidal-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from the following: 1) Metals in Ground Water: Sampling Artifacts and Reproducibility; 2) Filtration of Ground Water Samples for Metals Analysis; and 3) Ground Water Sampling - A Workshop Summary

Bacterial Sampling

Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in the following: 1) Sampling for Organic Chemicals and Microorganisms in the Subsurface; 2) Handbook for Evaluating Water Bacteriological Laboratories; and 3) Microbiological Methods for Monitoring the Environment, Water and Wastes.

5.5 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect potable water samples shall be cleaned as outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination

(SESDPROC-205) and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

5.6 Auxiliary Data Collection

During potable water sample collection, it may be necessary to record additional sampling data, such as flow rates, etc. This information should be documented in the field records.

Appendix F – Soil Sampling
SESDPROC-300-R1

COPY

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Soil Sampling**

Effective Date: November 1, 2007 | **Number:** SESDPROC-300-R1

Authors

Name: Fred Sloan
Title: Environmental Engineer, Regional Expert
Signature: [signature] Date: 11/2/07

Approvals

Name: Antonio Quinones
Title: ~~Chief, Enforcement~~ and Investigations Branch
Signature: [signature] Date: 11/02/07

Name: Laura Ackerman
Title: Field Quality Manager, Science and Ecosystem Support Division
Signature: Laura Acker Date: 11/01/07

COPY

Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Field Quality Manager.

History	Effective Date
SESDPROC-300-R1, *Soil Sampling*, replaces SESDPROC-300-R0. **General** Corrected any typographical, grammatical and/or editorial errors. **Title Page** Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch. **Section 1.3** Updated information to reflect that the procedure is located on the H: drive of the LAN. Clarified Field Quality Manager (FQM) responsibilities. **Section 1.4** Updated referenced operating procedures due to changes in title names. Alphabetized and revised the referencing style for consistency. **Section 1.5.1** Corrected the title of the Safety, Health, and Environmental Management Program Procedures and Policy Manual. **Section 1.5.2, 4th bullet** Added references to the CFR and IATA's Dangerous Goods Regulations. **Section 2.7** Updated referenced operating procedures due to changes in title names.	November 1, 2007
SESDPROC-300-R0, Soil Sampling, Original Issue	February 05, 2007

TABLE OF CONTENTS

TABLES

Contents

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting soil samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling soil samples in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used to obtain a soil sample, the variant procedure will be documented in the field log book and subsequent investigation report, along with a description of the circumstances requiring its use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the H: drive of the SESD local area network. The Field Quality Manager (FQM) is responsible for ensuring the most recent version of the procedure is placed on the H: drive and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field X-Ray Fluorescence (XRF) Measurement, SESDPROC-107, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

United States Environmental Protection Agency (US EPA). 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting soil samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting soil samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could

alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.

- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader in the project files.

2 Special Sampling Considerations

2.1 Soil Samples for Volatile Organic Compounds (VOC) Analysis

If samples are to be analyzed for volatile organic compounds, they should be collected in a manner that minimizes disturbance of the sample. For example, when sampling with a bucket auger, the sample for VOC analysis should be collected directly from the auger bucket (preferred) or from minimally disturbed material immediately after an auger bucket is emptied into the pan. The sample shall be containerized by filling an En Core® Sampler or other Method 5035 compatible container. ***Samples for VOC analysis are not homogenized.*** Preservatives may be required for some samples with certain variations of Method 5035. Consult the method or the principal analytical chemist to determine if preservatives are necessary.

2.2 Soil Sampling (Method 5035)

The following sampling protocol is recommended for site investigators assessing the extent of volatile organic compounds (VOC's) in soils at a project site. Because of the large number of options available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method best applicable to the investigation can be made.

2.2.1 Equipment

Soil for VOC analyses may be retrieved using any of the SESD soil sampling methods described in Sections 3 through 8 of this procedure. Once the soil has been obtained, the En Core® Sampler, syringes, stainless steel spatula, standard 2-oz. soil VOC container, or pre-prepared 40 ml vials may be used/required for sub-sampling. The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the site or sampling investigation. The various sub-sampling methods are described below.

2.2.2 Sampling Methodology - Low Concentrations (<200 ug/kg)

When the total VOC concentration in the soil is expected to be less than 200 µg/kg, the samples may be collected directly with the En Core® Sampler or syringe. If using the syringes, the sample must be placed in the sample container (40 ml pre-prepared vial) immediately to reduce volatilization losses. The 40 ml vials should contain 10 ml of organic-free water for an un-preserved sample or approximately 10 ml of organic-free water and a preservative. It is recommended that the 40 ml vials be prepared and weighed by the laboratory (commercial

sources are available which supply preserved and tared vials). When sampling directly with the En Core® Sampler, the vial must be immediately capped and locked

A soil sample for VOC analysis may also be collected with conventional sampling equipment. A sample collected in this fashion must either be placed in the final sample container (En Core® Sampler or 40 ml pre-prepared vial) immediately or the sample may be immediately placed into an intermediate sample container with no head space. If an intermediate container (usually 2-oz. soil jar) is used, the sample must be transferred to the final sample container (En Core® Sampler or 40 ml pre-prepared vial) as soon as possible, not to exceed 30 minutes.

NOTE: After collection of the sample into either the En Core® Sampler or other container, the sample must immediately be stored in an ice chest and cooled.

Soil samples may be prepared for shipping and analysis as follows:

En Core® Sampler - the sample shall be capped, locked, and secured in a plastic bag.

Syringe - Add about 3.7 cc (approximately 5 grams) of sample material to 40-ml pre-prepared containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag. Note: When using the syringes, it is important that no air is allowed to become trapped behind the sample prior to extrusion, as this will adversely affect the sample.

Stainless Steel Laboratory Spatulas - Add between 4.5 and 5.5 grams (approximate) of sample material to 40 ml containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag.

2.2.3 Sampling Methodology - High Concentrations (>200 ug/kg)

Based upon the data quality objectives and the detection level requirements, this high level method may also be used. Specifically, the sample may be packed into a single 2-oz. glass container with a screw cap and septum seal. The sample container must be filled quickly and completely to eliminate head space. Soils\sediments containing high total VOC concentrations may also be collected as described in Section 2.2.2, Sampling Methodology - Low Concentrations, and preserved using 10 ml methanol.

2.2.4 Special Techniques and Considerations for Method 5035

Effervescence

If low concentration samples effervesce from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un-preserved as needed, or all samples must be collected unpreserved.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence (rapid formation of bubbles) then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic free water. An appropriate amount of organic free water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured.

Sample Size

While this method is an improvement over earlier ones, field investigators must be aware of an inherent limitation. Because of the extremely small sample size and the lack of sample mixing, sample representativeness for VOC's may be reduced compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Holding Times

Sample holding times are specified in the Analytical Support Branch *Laboratory Operations and Quality Assurance Manual* (ASBLOQAM), Most Recent Version. Field investigators should note that the holding time for an un-preserved VOC soil/sediment sample is 48 hours. Arrangements should be made to ship the soil/sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may preserve and/or analyze the sample within 48 hours of collection.

Percent Moisture

Samplers must ensure that the laboratory has sufficient material to determine percent moisture in the VOC soil/sediment sample to correct the analytical results to dry weight. If other analyses requiring percent moisture determination are

being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 oz.) for percent moisture determination will be required. The sample collected for Percent Moisture may also be used by the laboratory to check for preservative compatibility.

Safety

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all required safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The vials of methanol should be stored in a cooler with ice at all times.

Shipping

Methanol and sodium bisulfate are considered dangerous goods, therefore shipment of samples preserved with these materials by common carrier is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulations must be followed when shipping methanol and sodium bisulfate. Consult the above documents or the carrier for additional information. Shipment of the quantities of methanol and sodium bisulfate used for sample preservation falls under the exemption for small quantities.

The summary table on the following page lists the options available for compliance with SW846 Method 5035. The advantages and disadvantages are noted for each option. SESD's goal is to minimize the use of hazardous material (methanol and sodium bisulfate) and minimize the generation of hazardous waste during sample collection.

2.3 Dressing Soil Surfaces

Any time a vertical or near vertical surface is sampled, such as achieved when shovels or similar devices are used for subsurface sampling, the surface should be dressed (scraped) to remove smeared soil. This is necessary to minimize the effects of contaminant migration interferences due to smearing of material from other levels.

Table 1: Method 5035 Summary

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect 2 - 40 mL vials with ~5 grams of sample and 1 - 2 oz., glass w/septum lid for screening, % moisture and preservative compatibility	Screening conducted by lab	Presently a 48 hour holding time for unpreserved samples Sample containers must be tared
2	Collect 3 En Core® Samplers; and 1- 2 oz., glass w/septum lid for screening, % moisture and preservative compatibility	Lab conducts all preservation/preparation procedures	Presently a 48 hour holding time for preparation of samples
3	Collect 2 - 40 ml vials with 5 grams of sample and preserve w/methanol or sodium bisulfate and 1 - 2-oz., glass w/septum lid for screening, % moisture and preservative compatibility	High level VOC samples may be composited Longer holding time	Hazardous materials used in field Sample containers must be tared
4	Collect 1 - 2-oz., glass w/septum lid for analysis, % moisture and preservative compatibility	Lab conducts all preservation/preparation procedures	May have significant VOC loss

2.4 Special Precautions for Trace Contaminant Soil Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different sample is collected and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be collected, handled and stored separately.
- All background samples shall be segregated from obvious high concentration or waste samples. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.

- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205), for collection of samples for trace metals or organic compound analyses.

2.5 Sample Homogenization

1. If sub-sampling of the primary sample is to be performed in the laboratory, transfer the entire primary sample directly into an appropriate, labeled sample container(s). Proceed to step 5.

2. If sub-sampling the primary sample in the field or compositing multiple primary samples in the field, place the sample into a glass or stainless steel homogenization container and mix thoroughly. Each aliquot of a composite sample should be of the same approximate volume.

3. All soil samples must be thoroughly mixed to ensure that the sample is as representative as possible of the sample media. ***Samples for VOC analysis are not homogenized.*** The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

   - The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
   - Two quarters should then be mixed to form halves.
   - The two halves should be mixed to form a homogenous matrix.

   This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4. Place the sample into an appropriate, labeled container(s) by using the alternate shoveling method and secure the cap(s) tightly. The alternate shoveling method involves placing a spoonful of soil in each container in sequence and repeating until the containers are full or the sample volume has been exhausted. Threads on the container and lid should be cleaned to ensure a tight seal when closed.

5. Return any unused sample material back to the auger, drill or push hole from which the sample was collected.

2.6 Quality Control

If possible, a control sample should be collected from an area not affected by the possible contaminants of concern and submitted with the other samples. This control sample should be collected as close to the sampled area as possible and from the same soil type. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by sampling tools. SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) contains other procedures that may be applicable to soil sampling investigations.

2.7 Records

Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation, as described in the SESD Operating Procedure for Logbooks (SESDPROC-010) and the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

3 Manual Soil Sampling Methods

3.1 General

These methods are used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The most common interval is 0 to 6 inches, however the data quality objectives of the investigation may dictate another interval, such as 0 to 3 inches for risk assessment purposes. The shallow subsurface interval may be considered to extend from approximately 12-inches below ground surface to a site-specific depth at which sample collection using manual collection methods becomes impractical.

3.2 Spoons

Stainless steel spoons may be used for surface soil sampling to depths of approximately 6-inches below ground surface where conditions are generally soft and non-indurated and there is no problematic vegetative layer to penetrate.

3.2.1 Special Considerations When Using Spoons

- When using stainless steel spoons, consideration must be given to the procedure used to collect the volatile organic compound sample. If the soil being sampled is cohesive and holds its in situ texture in the spoon, the En Core® Sampler or syringe used to collect the sub-sample for Method 5035 should be plugged directly from the spoon. If, however, the soil is not cohesive and crumbles when removed from the ground surface for sampling, consideration should be given to plugging the sample for Method 5035 directly from the ground surface at a depth appropriate for the investigation Data Quality Objectives.
- When compositing, make sure that each composite location (aliquot) consist of equal volumes, i.e., same number of equal spoonfuls.
- If a thick, matted root zone is present at or near the surface, it should be removed before the sample is collected

3.3 Hand Augers

Hand augers may be used to advance boreholes and collect soil samples in the surface and shallow subsurface intervals. Typically, 4-inch stainless steel auger buckets with cutting heads are used. The bucket is advanced by simultaneously pushing and turning using an attached handle.

3.3.1 Surface Soil Sampling

When conducting surface soil sampling with hand augers, the auger buckets may be used with a handle alone or with a handle and extensions. The bucket is

advanced to the appropriate depth and the contents are transferred to the homogenization container for processing. Observe precautions for volatile organic compound sample collection found in Section 2.2.4, Special Techniques and Considerations for Method 5035.

3.3.2 Subsurface Soil Sampling

Hand augers are the most common equipment used to collect shallow subsurface soil samples. Auger holes are advanced one bucket at a time until the sample depth is achieved. When the sample depth is reached, the bucket used to advance the hole is removed and a clean bucket is attached. The clean auger bucket is then placed in the hole and filled with soil to make up the sample and removed.

The practical depth of investigation using a hand auger depends upon the soil properties and depth of investigation. In sand, augering is usually easily performed, but the depth of collection is limited to the depth at which the sand begins to flow or collapse. Hand augers may also be of limited use in tight clays or cemented sands. In these soil types, the greater the depth attempted, the more difficult it is to recover a sample due to increased friction and torqueing of the hand auger extensions. At some point these problems become so severe that power equipment must be used.

3.3.3 Special Considerations for Soil Sampling with the Hand Auger

- Because of the tendency for the auger bucket to scrape material from the sides of the auger hole while being extracted, the top several inches of soil in the auger bucket should be discarded prior to placing the bucket contents in the homogenization container for processing.
- Observe precautions for volatile organic compound sample collection found in Section 2.2.4, Special Techniques and Considerations for Method 5035. Collect the VOC sample directly from the auger bucket, if possible.
- Power augers, such as the Little Beaver®, and drill rigs may be used to advance boreholes to depths for subsurface soil sampling with the hand auger. They may not be used for sample collection. When power augers are used to advance a borehole to depth for sampling, care must be taken that exhaust fumes, gasoline and/or oil do not contaminate the borehole or area in the immediate vicinity of sampling.
- When a new borehole is advanced, the entire hand auger assembly must be replaced with a properly decontaminated hand auger assembly.

4 Direct Push Soil Sampling Methods

4.1 General

These methods are used primarily to collect shallow and deep subsurface soil samples. Three methods are available for use with either the Geoprobe® or the drill rig adapted with a hydraulic hammer. All methods involve the collection and retrieval of the soil sample within a thin-walled liner. The following sections describe each of the specific sampling methods that can be accomplished using direct push techniques, along with details specific to each method.

4.2 Large Bore® Soil Sampler

The Large Bore® (LB) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of depth-discrete subsurface soil samples. The sample barrel is approximately 30-inches (762 mm) long and has a 1.5-inch (38 mm) outside diameter. The LB® sampler is capable of recovering a discrete sample core 22 inches x 1.0 inch (559 mm x 25 mm) contained inside a removable liner. The resultant sample volume is a maximum of 283 ml.

After the LB® sample barrel is equipped with the cutting shoe and liner, the piston-rod point assembly is inserted, along with the drive head and piston stop assembly. The assembled sampler is driven to the desired sampling depth, at which time the piston stop pin is removed, freeing the push point. The LB® sampler is then pushed into the soil a distance equal to the length of the LB® sample barrel. The probe rod string, with the LB® sampler attached, is then removed from the subsurface. After retrieval, the LB® sampler is then removed from the probe rod string. The drive head is then removed to allow removal of the liner and soil sample.

4.3 Macro-Core® Soil Sampler

The Macro-Core® (MC) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of either continuous or depth-discrete subsurface soil samples. Although other lengths are available, the standard MC® sampler has an assembled length of approximately 52 inches (1321 mm) with an outside diameter of 2.2 inches (56 mm). The MC® sampler is capable of recovering a discrete sample core 45 inches x 1.5 inches (1143 mm x 38 mm) contained inside a removable liner. The resultant sample volume is a maximum of 1300 ml. The MC® sampler may be used in either an open-tube or closed-point configuration. Samples collected for chemical analyses must be collected with the closed-point configuration. If used for collection of soil for stratigraphic descriptions, the open-tubed configuration is acceptable.

4.4 Dual Tube Soil Sampling System

The Dual Tube 21 soil sampling system is a direct push system for collecting continuous core samples of unconsolidated materials from within a sealed outer casing of 2.125-inch (54 mm) OD probe rod. The samples are collected within a liner that is threaded onto the leading end of a string of 1.0-inch diameter probe rod. Collected samples have a volume of up to 800 ml in the form of a 1.125-inch x 48-inch (29 mm x 1219 mm) core. Use of this method allows for collection of continuous core inside a cased hole, minimizing or preventing cross-contamination between different intervals during sample collection. The outer casing is advanced, one core length at a time, with only the inner probe rod and core being removed and replaced between samples. If the sampling zone of interest begins at some depth below ground surface, a solid drive tip must be used to drive the dual tube assembly and core to its initial sample depth.

4.5 Special Considerations When Using Direct Push Sampling Methods

- *Liner Use and Material Selection* – Due to the mode of operation, the samples must be collected with a liner. Liners are available in the following materials: stainless steel, brass, cellulose acetate butyrate (CAB), PETG, polyvinyl chloride (PVC) and Teflon®. For the majority of environmental investigations conducted by EIB, either CAB or Teflon® liners are used. If samples are collected for organic compound analyses, Teflon® liners are required. CAB or PVC liners may be used if metals or other inorganic constituents are the object of the investigation.
- *Sample Orientation* – When the liners and associated sample are removed from the sample tubes, it is important to maintain the proper orientation of the sample. This is particularly important when multiple sample depths are collected from the same push. It is also important to maintain proper orientation to define precisely the depth at which an aliquot was collected. Maintaining proper orientation is typically accomplished using vinyl end caps. Convention is to place red caps on the top of the liner and black caps on the bottom to maintain proper sample orientation. Orientation can also be indicated by marking on the exterior of the liner with a permanent marker.
- *Core Catchers* – Occasionally the material being sampled lacks cohesiveness and is subject to crumbling and falling out of the sample liner. In cases such as these, the use of core catchers on the leading end of the sampler may help retain the sample until it is retrieved to the surface. Materials of construction for core catchers must be consistent with the type of liner used, i.e., if stainless steel liners are required, stainless steel core catchers must be used.
- *VOC Sample Collection* - Observe precautions for volatile organic compound sample collection found in Section 2.2.4, Special Techniques and Considerations for Method 5035.

5 Split Spoon/Drill Rig Methods

5.1 General

Split spoon sampling methods are used primarily to collect shallow and deep subsurface soil samples. All split spoon samplers, regardless of size, are basically split cylindrical barrels that are threaded on each end. The leading end is held together with a beveled threaded collar that functions as a cutting shoe. The other end is held together with a threaded collar that serves as the sub used to attach the spoon to the string of drill rod. Two basic methods are available for use, including the smaller diameter standard split spoon, driven with the drill rig safety hammer, and the larger diameter continuous split spoon, advanced inside and slightly ahead of the lead auger during hollow stem auger drilling. The following sections describe each of the specific sampling methods, along with details specific to each method.

5.2 Standard Split Spoon

A drill rig is used to advance a borehole to the target depth. The drill string is then removed and a standard split spoon is attached to a string of drill rod. Split spoons used for soil sampling must be constructed of stainless steel and are typically 2.0-inches OD (1.5-inches ID) and 18-inches to 24-inches in length. Other diameters and lengths are common and may be used if constructed of the proper material. After the spoon is attached to the string of drill rod it is lowered into the borehole. The drill rig safety hammer is then used to drive the split spoon into the soil at the bottom of the borehole. After the split spoon has been driven into the soil, filling the spoon, it is retrieved to the surface, where it is removed from the drill rod string and opened for sample acquisition.

5.3 Continuous Split Spoon

The continuous split spoon is a large diameter split spoon that is advanced into the soil column inside a hollow stem auger. Continuous split spoons are typically 3-inches to 5-inches in diameter and either 5-feet or 10-feet in length, although the 5-foot long samplers are most common. After the auger string has been advanced into the soil column a distance equal to the length of the sampler being used it is returned to the surface. The sampler is removed from inside the hollow stem auger and the threaded collars are removed. The split spoon is then opened for sampling.

5.4 Special Considerations When Using Split Spoon Sampling Methods

- Always discard the top several inches of material in the spoon before removing any portion for sampling. This material normally consists of borehole wall material that has sloughed off of the borehole wall after removal of the drill string prior to and during inserting the split spoon.
- Observe precautions for volatile organic compound sample collection found in Section 2.2.4, Special Techniques and Considerations for Method 5035.

6 Shelby Tube/Thin-Walled Sampling Methods

6.1 General

Shelby tubes, also referred to generically as thin-walled push tubes or Acker thin-walled samplers, are used to collect subsurface soil samples in cohesive soils and clays during drilling activities. In addition to samples for chemical analyses, Shelby tubes are also used to collect relatively undisturbed soil samples for geotechnical analyses, such as hydraulic conductivity and permeability, to support hydrogeologic characterizations at hazardous waste and other sites.

6.2 Shelby Tube Sampling Method

A typical Shelby tube is 30-inches in length and has a 3.0-inch OD (2.875 ID) and may be constructed of steel, stainless steel, galvanized steel, or brass. They also typically are attached to push heads that are constructed with a ball-check to aid in holding the contained sample during retrieval. If used for collecting samples for chemical analyses, it must be constructed of stainless steel. If used for collecting samples for standard geotechnical parameters, any material is acceptable.

To collect a sample, the tube is attached to a string of drill rod and is lowered into the borehole, where the sampler is then pressed into the undisturbed clay or silts by hydraulic force. After retrieval to the surface, the tube containing the sample is then removed from the sampler head. If samples for chemical analyses are needed, the soil contained inside the tube is then removed for sample acquisition. If the sample is collected for geotechnical parameters, the tube is typically capped, maintaining the sample in its relatively undisturbed state, and shipped to the appropriate geotechnical laboratory.

6.3 Special Considerations When Using Split Spoon Sampling Methods

Observe precautions for volatile organic compound sample collection found in Section 2.2.4, Special Techniques and Considerations for Method 5035.

7 Backhoe Sampling Method

7.1 General

Backhoes may be used in the collection of surface and shallow subsurface soil samples. The trenches created by excavation with a backhoe offer the capability of collecting samples from very specific intervals and allow visual correlation with vertically and horizontally adjacent material. If possible, the sample should be collected without entering the trench. Samples may be obtained from the trench wall or they may be obtained directly from the bucket at the surface. The following sections describe various techniques for safely collecting representative soil samples with the aid of a backhoe.

7.2 Scoop and Bracket Method

If a sample interval is targeted from the surface, it can be sampled using a stainless steel scoop and bracket. First a scoop and bracket are affixed to a length of conduit and is lowered into the backhoe pit. The first step is to take the scoop and scrape away the soil comprising the surface of the excavated wall. This material likely represents soil that has been smeared by the backhoe bucket from adjacent material. After the smeared material has been scraped off, the original stainless steel scoop is removed and a clean stainless steel scoop is placed on the bracket. The clean scoop can then be used to remove sufficient volume of soil from the excavation wall to make up the required sample volume.

7.3 Direct-From-Bucket Method

It is also possible to collect soil samples directly from the backhoe bucket at the surface. Some precision with respect to actual depth or location may be lost with this method but if the soil to be sampled is uniquely distinguishable from the adjacent or nearby soils, it may be possible to characterize the material as to location and depth. In order to ensure representativeness, it is also advisable to dress the surface to be sampled by scraping off any smeared material that may cross-contaminate the sample.

7.4 Special Considerations When Sampling with a Backhoe

- Do not physically enter backhoe excavations to collect a sample. Use either procedure 7.2, Scoop and Bracket Method, or procedure 7.3, Direct-From-Bucket Method to obtain soil for sampling.
- Smearing is an important issue when sampling with a backhoe. Measures must be taken, such as dressing the surfaces to be sampled (see Section 2.3), to mitigate problems with smearing.

- Paint, grease and rust must be removed and the bucket decontaminated prior to sample collection.
- Observe precautions for volatile organic compound sample collection found in Section 2.2.4, Special Techniques and Considerations for Method 5035.

Appendix G – Collection of Vegetation Samples
USDA APHIS SOP No. EM-07

SOP No. EM-07		**Page 1 of 4**
Collection of Vegetation Samples		
Revision: #5	**Replaces: 6/25/02 version**	**Effective: 2/22/10**

1. Purpose and Scope: Vegetation samples are collected to measure the amount of pesticide present on or in plants. Residue information can be used to evaluate risks to human health if the vegetation is edible, or to wildlife susceptible to exposure from contaminated plants. This SOP describes how to collect, handle, and store vegetation samples. Vegetation sampling and documentation instructions found in the Environmental Monitoring Plan (EMP) supersede instructions contained in this SOP.

2. Supplies Required: To request sampling equipment and other supplies to collect vegetation samples, contact the Laboratory Supplies Coordinator at the APHIS, Plant Protection and Quarantine, Center for Plant Health Science and Technology (CPHST Gulfport Lab) at (228) 323-5329 or 822-3106.

2.1 pruning shears or large scissors

2.2 mattock or pick (for collecting root or tubers)

2.3 garden rake or potato digger (for collecting submerged aquatic plants)

2.4 foil lined envelopes

2.5 strapping tape

2.6 12" x 12" resealable plastic bags

2.7 field log book

2.8 ice chest with wet, dry, or reusable ice packs (obtain locally)

2.9 environmental monitoring forms (APHIS Form 2060)

2.10 indelible marker

2.11 aluminum foil

2.12 disposable gloves

2.13 sanitary wipes

SOP No. EM-07		Page 2 of 4
Collection of Vegetation Samples		
Revision: #5	Replaces: 6/25/02 version	Effective: 2/22/10

3. Collecting Above-Ground Vegetation: This sample consists of either: leaves, grasses, fruits, grains, or seeds. Do not mix types of vegetation in a single sample. Collect samples from the portion of a plant most likely to have been (or to be) exposed to the pesticide.

3.1 While wearing disposable gloves, use the pruning shears or scissors to cut off the portion of the plant to be sampled.

3.2 Place the vegetation into a foil lined envelope. If a fruit is too large to fit into the foil envelope, then wrap it in aluminum foil and place it into a 12"x 12" resealable plastic bag.

3.3 Repeat steps 3.1 and 3.2 until the foil lined envelope is filled to about two inches from the top.

3.4 Fold over the top of the envelope twice and seal with strapping tape. Using the indelible marker, label the sealed envelope with a unique identifier or code such that the information matches the sample documentation. Also record on the envelope the type of vegetation sampled, sampling site, and date.

3.5 Place the sample into the ice chest to keep it chilled until it can be transported to a freezer for storage until shipping.

3.6 Clean and decontaminate the pruning shears or scissors between each sample collection using fresh wipes.

4. Collecting Roots or Tubers:

4.1 Contact the Environmental Compliance Team to discuss the procedure and purpose for the collection of subsurface plant parts.

4.2 Dig up roots or tubers with the pick. While wearing disposable gloves, shake off as much of the attached soil as possible.

4.3 Place the roots or tubers into a heavy foil envelope. If necessary, cut roots to lengths short enough to fit into the envelope.

4.4 Repeat steps 4.1 and 4.2 until the foil envelope is filled to about two inches from the top.

4.5 Fold over the top of the envelope twice and seal with strapping tape. Using the indelible marker, label the sealed envelope with a unique identifier or code such

that the information matches the sample documentation. Also record on the envelope the type of vegetation sampled, sampling site, and date.

4.6 Place the sample into the ice chest to keep it chilled until it can be transported to a freezer for storage until shipping.

4.7 Clean attached soil from the collection pick using water and then decontaminate with fresh wipes between each sample collection.

5. Collecting Submerged Aquatic Plants:

5.1 Contact the Environmental Compliance Team to discuss the procedure and purpose for the collection of submerged plant parts.

5.2 Using a potato digger or a garden rake, pull out submerged parts of aquatic plants. While wearing disposable gloves, shake the plants to remove excess water.

5.3 Place the plants into a heavy foil envelope. If necessary, cut plants into segments small enough to fit into the envelope.

5.4 Repeat steps 5.1 and 5.2 until the foil envelope is filled to about two inches from the top.

5.5 Fold over the top of the envelope twice and seal with strapping tape. Using the indelible marker, label the sealed envelope with a unique identifier or code such that the information matches the sample documentation. Also record on the envelope the type of vegetation sampled, sampling site, and date.

5.6 Place the sample into the ice chest to keep it chilled until it can be transported to a freezer for storage until shipping.

5.7 Clean extraneous plant material from the digger or rake and then decontaminate using fresh wipes.

6. Documentation. A thorough description of the type of vegetation collected is important because it affects how the residue data is interpreted.

6.1 Record all observations in the field log book (see SOP EM-12, *Using a Field Log Book*). Draw a site map, including an approximate scale and North arrow, showing the location of the sample collection site and its relation to the treatment site and any nearby sensitive sites. A topographical map or aerial photograph

annotated with the required information should be provided if possible, as well as photographs or a video of the sample collection site. Global positioning system (GPS) coordinates of the site should be included. Describe or identify the type or species of the plant collected (common or scientific names). Be sure to record the part of the plant collected (e.g. leaves, fruit, stems, seeds, roots), and the location on the plant from which the sample was taken (e.g. top, bottom, edge). Describe the height and density of any vegetation in the area between the treatment site and the sample collection site

6.2 Complete an APHIS Form 2060 for each vegetation sample.

6.3 Retain the pink copy of Form 2060 for your records and distribute the remaining copies as specified in the EMP.

7. Packaging and Shipping:

7.1 Package and ship the vegetation samples as described in SOP EM-17, *Packaging and Shipping of Samples.*

Appendix H – Corrective Action Form

Project Name and Number: _____

Sample Dates Involved: _____

Measurement Parameter:

_____

Acceptable Data Range:

_____

Problem Areas Requiring Corrective Action:

_____

Measures Required to Correct the Problem:

_____

Means of Detecting Problems and Verifying Correction:

_____

Initiators Name: _____ Date: _____

Project Officer: _____ Date: _____

QA Officer: _____Date:_____

Appendix I – Sample Alteration Form

Project Name and Number: _____

Material to be sampled: _____

Measurement Parameter:

_____

Standard Procedure for Field Collection & Laboratory Analysis (cite reference):

_____

Reason for Change in Field Procedure or Analysis Variation:

_____

Variation from Field or Analytical Procedure:

_____

Special Equipment, Materials or Personnel Required:

_____

Initiators Name: _____ Date: _____

Project Officer: _____ Date: _____

QA Officer: _____ Date: _____