Prepared for

HONEYWELL

WORKPLAN FOR DIOXIN/FURAN CHARACTERIZATION

LCP Chemicals Site, Operable Unit 3
Brunswick Georgia

Prepared by:

Environmental Planning Specialists, Inc.
900 Ashwood Parkway, Suite 350
Atlanta, Georgia 30338
Tel: 404-315-9113

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EPS

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900 Ashwood Parkway, Suite 350
Atlanta, GA 30338

Kirk Kessler, Principal

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Telephone (404) 315-9113 • Fax (404) 315-8509 • www.envplanning.com

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1 INTRODUCTION

1.1 Overview

This Workplan was prepared in response to request by the Environmental Protection Agency (EPA) to characterize upland soil at the LCP site, which is identified as Operable Unit #3 (OU-3), for polychlorinated dibenzo-*p*-dioxin and polychlorinated dibenzo-*p*-furan congeners (commonly referred to as dioxins and furans).

This Workplan describes the existing dioxin/furan data, proposes a conceptual model to guide the investigation of potential dioxin/furan impacts, and proposes characterization of these potential impacts using the incremental sampling methodology, or ISM.

On March 10, 2011, Honeywell and EPS met with EPA and the Georgia Environmental Protection Division (EPD) regarding this topic. The content and scope of the work contained in this Workplan incorporate the discussions and agreements reached with EPA and EPD during that meeting.

1.2 Sampling Considerations

"Dioxin" is a general term that refers to a group of ubiquitous and persistent chemicals arising from both natural and anthropogenic processes. Dioxins exist as members of two closely related chemical families, the chlorinated dibenzo-p-dioxins (CDDs) and the chlorinated dibenzofurans (CDFs). There are 210 different potential chemical configurations of these compounds that are individually referred to as congeners. Individual congeners of CDDs, and CDFs are identified based on the number and location of the chlorine atoms within the chemical structures.

Dioxins are generated as a by-product of a variety of natural and industrial processes including waste incineration, forest and brush fires, wood and coal combustion, diesel exhaust, herbicide production, pentachlorophenol production, and pulp and paper bleaching. Each of the processes that produce dioxins differ in important aspects that affect the spectrum of dioxin congeners produced (meaning that there is a "fingerprint" associated with dioxins/furans). Dioxins are relatively immobile and persistent in the environment. For example, high levels of dioxins formed through natural processes, have been reported in 40 million year old geological clays (Ferrario and Byrne, 2002). Their environmental stability is the result of several physical/chemical characteristics including extremely low water solubility, low volatility, and high binding affinity to soil organic carbon (Mackay et al., 1992).

Dioxins produce toxicity through a relatively well understood sequence of biological events that starts with binding of dioxin/furan congeners to a specific cellular protein. Of the hundreds of different dioxin congeners, there are only seven CDD congeners, and ten CDF congeners that have the significant ability to bind to this protein and induce toxicity. Of these congeners, 2,3,7,8-TCDD has the highest binding affinity and, as a result, is used as the reference congener when evaluating toxicity. Toxic equivalency factors (TEFs) have been developed to provide a method to estimate the relative toxicity of different dioxin congeners present in environmental samples, and are typically used to convert congener-specific data into TCDD toxic equivalents (TEQ) to facilitate risk assessment and regulatory determinations.

If historic site operations resulted in the generation of dioxins/furans, the primary media and areas of the site impacted would be surface soil in the former process areas, including the mercury cell building and anode handling areas. Two samples that were collected by EPA during the removal action in 1995 were in these process areas, and the samples were analyzed for individual dioxin/furan congeners. The locations of these samples are shown in Figure 1 and the results are summarized in Table 1. The TCDD TEQ results from these two samples are below the dioxin soil cleanup level of 5,000 to 20,000 ng/kg for commercial/industrial soil identified in EPA's OSWER Directive 9200.4-26 (EPA 1998). The results are also below the preliminary remediation goal (PRG) of 950 ng/kg for commercial industrial soil, released by EPA in 2009 (EPA 2009).

1.3 Incremental Sampling Methodology

ISM is a structured composite sampling and processing protocol that reduces data variability and provides a robust estimate of the mean concentration of an analyte or analytes in the area or volume of soil being sampled. Variability in measured analyte concentrations between discrete soil samples is due primarily to the particulate nature of soil and heterogeneity in the distribution of the analyte due to site-specific release and fate/transport mechanisms. The elements of ISM that control data variability are incorporated into the field collection of soil samples and the laboratory processing procedures. Thus, ISM is a two part process designed to obtain a single sample for analysis that has all analytes in the same proportion as an explicitly defined area/volume of soil, termed a "Decision Unit (DU)[1].

[1] Under some ISM plans, including this one, individual incremental samples may also be collected to represent smaller areas, termed "sampling units (SU)" within a larger DU. The results of the incremental samples for the SUs may be combined to estimate the concentration of an analyte in the DU.

2 SYSTEMATIC PLANNING FOR ISM

2.1 Overview

Systematic planning involves a series of steps that identify the objectives of the site investigation and establishes the type of information needed to determine if unacceptable levels of a contaminant exist at a site. Systematic planning should be component of any sample collection effort, but the fact that ISM samples are integrally tied to a particular DU makes this type of advanced planning particularly important for ISM investigations.

The most important element in systematic planning for an ISM investigation is to develop a scope such that there is an understanding that that the results of the investigation will provide an estimate of the average concentration of the analytes of interest in each SU or DU and that the estimate will be used to determine whether further activity is warranted.

A number of other questions specific to ISM sampling must also be addressed during systematic planning, including:

- What were the potential sources, release mechanisms, and fate/transport processes that could affect the distribution of contaminants of interest at the site?
- What are the number, location, dimensions, and rationale used in selecting DUs and SUs?
- How many increments will samples include?
- What is the targeted sample volume and approximate increment volume needed?
- How many and what type of replicates should be collected (e.g., DU replicate, SU replicate, field replicate, laboratory replicate, instrument replicate)?

2.2 Conceptual Site Model

No specific source of dioxins/furans has been identified at the site[2]. If there are dioxins/furans on the site as the result of past industrial operations on the site, the likely location would be in the area of the former cell building and other areas where the anodes were handled after removal from the cell building. Because the majority of these areas were addressed (i.e., excavated or covered) during the removal action that occurred between 1995 and 1997, there are no longer any significant "source areas" associated with the handling/processing of these graphite anodes. However, because Aroclor 1268 was present in the graphite anodes used at the LCP site, residual

[2] Honeywell notes that dioxins/furans occur naturally and have also been associated with the pulp and paper industry.

concentrations of this chemical should serve as a marker for locations where dioxins/furans might also be present at the highest concentrations. Figure 2 illustrates the spatial distribution of Aroclor 1268 concentrations in existing surface soil samples from across the site. Similar information on the spatial distribution of Aroclor 1260 and 1254 concentrations in surface soil is provided in Figures 3 and 4, respectively. Although these two Aroclors were not utilized in the chor-alkali process and their spatial distribution is not entirely consistent with that of Aroclor 1268, their presence provides additional areas that may warrant investigation for dioxins/furans.

2.3 Decision Units / Sampling Units

The proposed DUs for the ISM sampling correspond to the exposure units established in the OU3 HHBRA, which encompasses approximately 110 contiguous acres of upland area that was segregated into four exposure units. Two physical boundaries were used to create the HHBRA exposure units: the north-south oriented fence line that separates the primary operational areas on the west side of the site from administrative and light operational areas on the east side of the site; and the east-west oriented "B Street" (an asphalt-paved site road maintained throughout all of the operational history of the site). The sampling strategies for each DU described below are consistent with previous sampling and based on discussions with EPA and EPD.

2.3.1 Quadrant 1

Quadrant 1 occupies approximately 33.2 acres. Based on knowledge of historical facility operations, this quadrant was not impacted by the chlor-alkali process. Based on these considerations, a single DU that covers the entire quadrant is recommended. Figure 5a illustrates the proposed sampling grid for this DU. This sampling grid consists of 144 100×100 foot cells in order to accommodate the collection of 100 incremental samples across the DU while allowing for some discretion on the part of field staff who may not be able to sample from some of the grids due to dense vegetation or the presence of buildings or pavement. Three replicate samples of 100 increments each will be collected from this DU for a total of three samples from this quadrant.

2.3.2 Quadrant 2

Quadrant 2 occupies approximately 18.9 acres. Historical use of this portion of the site was mostly limited to administrative site functions. However, the northwestern edge of this quadrant abuts the footprint of the former mercury cell building and there is a small area in central portion of this DU that was previously investigated for PCB impacts. We are proposing three approximately equal-size SUs for this quadrant.

As shown on Figure 5b, the locations of two of these SUs are biased towards areas of highest residual Aroclor 1268 concentrations, while the third SU is located in a more densely vegetated portion of the quadrant where less sampling had been performed. These three sampling grids consist of 36 50×50 foot cells in order to accommodate the collection of 30 incremental samples

from each SU while allowing for some discretion in determining an exact sampling location due to dense vegetation or other physical constraints. Two replicate samples of 30 increments each will be collected from each SU for a total of six samples from this quadrant.

2.3.3 Quadrant 3

Quadrant 3 occupies approximately 22.5 acres. Significant portions of this quadrant were remediated during the removal action (see Figure 2). We are proposing three approximately equal-size SUs for this quadrant. As shown on Figure 5c, the locations of two of these SUs are biased to areas of highest residual Aroclor 1268 concentrations in the southern portion of the quadrant, while the third SU is located to the north to provide more spatial coverage across the quadrant. These three sampling grids consist of 48 50×50 foot cells in order to accommodate the collection of 30 incremental samples from each SU while allowing for some discretion in determining the exact sampling location due to dense vegetation or other physical constraints. Two replicate samples of 30 increments each will be collected from each SU for a total of six samples from this quadrant.

2.3.4 Quadrant 4

Quadrant 4 occupies approximately 45.9 acres. Historical uses of this portion of the site included the mercury cell building and anode loading area (along a former rail spur). Significant portions of this quadrant were remediated during the removal action (see Figure 2). We are proposing three similarly sized SUs for this quadrant. As shown on Figure 5d, the locations of all three of these SUs are biased to areas of highest residual Aroclor 1268 concentrations. These three sampling grids consist of between 48 to 72 50×50 foot cells in order to accommodate the collection of 30 incremental samples from each SU while allowing for appropriate discretion in determining the exact sampling grids due to dense vegetation or other physical constrains. Two replicate samples of 30 increments each will be collected from each SU for a total of six samples from this quadrant.

2.4 Number and Mass of Increments

For Quadrant 1, three replicates consisting of 100 incremental samples each will be collected over the entire DU. Each of the 100 increments should weigh approximately 15-20 g in order to achieve the target sample mass of approximately 1.5 kg for each multi-increment sample to be delivered to the analytical laboratory. For the remaining quadrants, two replicates consisting of 30 increments each will be collected in each SU. Each of the 30 increments should weigh approximately 50 g in order to achieve the target sample mass of approximately 1.5 kg for each multi-increment sample to be delivered to the analytical laboratory.

2.5 Sample Processing and Analysis

All incremental samples will be sent to Test America's West Sacramento for ISM processing and analysis. At that lab, each incremental sample will be processed according to Test America's internal standard operating procedure (SOP) for the processing for ISM samples. A copy of this SOP is provided in Appendix A.

After processing, each sample will be analyzed for dioxins and furans by EPA SW-846 Method 8290 (*Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans by High Resolution Gas Chromatography/High Resolution Mass Spectrometry*). A copy of Test America's data quality objective (DQO) summary for Method 8290 is provided in Appendix B.

This DQO summary indicates that Method 8290 provides detection limits for dioxin/furan congeners that result in a TCDD TEQ detection limit well below EPA's current PRGs for TCDD in residential and commercial/industrial soil (EPA 2009).

2.6 Data Evaluation

The dioxin/furan congener data from each ISM sample will be used to calculate TCDD TEQ based on the most recent TEFs from the World Health Organization (WHO 2005). For Quadrant 1, the results from the three replicate ISM samples will be averaged and compared with EPA's current PRG for dioxin in commercial/industrial soil (i.e., 950 ng/kg). If the average TCDD TEQ concentration is below this value, no further action will be necessary. Follow up sampling (likely to involve discrete grab-type sampling) may be necessary should the decision unit fail the data comparison.

For Quadrants 2, 3, and 4, the results for the two replicate ISM samples from each SU will be averaged and compared EPA's current PRG for dioxin in commercial/industrial soil. If the average TCDD TEQ result for each SU is below the PRG value, no further action will be necessary. If the results in one or more of the SUs exceed the PRG value but the variance among SUs is relatively low, it may be appropriate to average TCDD TEQ results between the three SUs in a quadrant for comparison with the PRG value. If the representative TCDD TEQ concentration(s) for a SU or DU exceeds the PRG value, the individual SU concentrations will be examined to evaluate spatial trends in the data and to develop recommendations for follow-on actions. In that event, Honeywell will submit a separate Workplan to EPA for review for these recommended follow-on actions.

3 FIELD PROCEDURES FOR SAMPLING

3.1 ISM Soil Sampling Procedures

The proposed locations of the SUs within each quadrant are shown in Figures 5a through 5d. The layout of each of these SUs and interior grids were established using ArcGIS 10. Upon commencing the ISM fieldwork, a portable global positioning unit (GPS) will be used to locate and stake the grid intersections within each SU (compass and tape methods are likely necessary to augment the use of the GPS in areas of density canopy). The portable GPS unit will also be available in the field to assist in the sample collection work (noting that the GPS unit uses Arcpad technology whereby map visualization of the ISM grids are provided directly on the device, with a location cursor that moves in real time with the unit).

A cordless drill with a one to two-inch diameter wood auger drill bit will be used to collect soil samples at each incremental sampling location. The drill will penetrate through a rigid material (e.g., a plastic disk) with a center cut hole slightly larger than the drill bit being used, and continue three inches into the soil. The center hole of the material will be pressed firmly to the soil surface so that the soil tailings flow through the hole and onto the material, rather than piling up under the material. This may be accomplished by laying two yardsticks across the material once it is in position with the sampler standing on the yardsticks to keep the material from moving. The tailings collected on the material will be placed in a one gallon Ziploc storage bag. All incremental soil samples from a single replicate within each SU/DU will be placed together in a single Ziploc bag.

All samples will be double bagged prior to transport. All samples will be stored in coolers and maintained at a temperature of 4°C during shipment to the laboratory. Samples will be shipped overnight for each day of sample collection. Because of the lengthy holding time for dioxin/furan samples, the incremental samples, plus one blind duplicate will be shipped Test America's West Sacramento laboratory together at the end of the sampling event.

3.2 Sample Equipment Decontamination

Within an individual SU, any soil or mud adhered to the drill bit after collecting an incremental sample will be wiped onto the ground prior to the collection of the next incremental sample. Between SUs, drill bits will be washed in a 5-gallon bucket filled with a solution of Liqui-nox, rinsed with deionized water, rinsed with 70% isopropal alcohol, and rinsed again with deionized water. Drill bits will be air dried and wrapped in aluminum foil. All field equipment will be decontaminated prior to being moved from the Site. These decontamination procedures are

consistent with the EPA Region IV Field Branches Quality System and Technical Procedures (USEPA, 2008).

3.3 Sample Documentation

3.3.1 Overview

Documents for recording sampling events will include a daily field activity log, field measurement logs, and photographs as appropriate. Sample information to be included on sample labels, custody seals, and chain-of-custody forms is described below.

3.3.2 Sample Identification and Documentation

After sample collection, all sample containers will be labeled with an identification number that uniquely identifies the sample. The samples will be identified with a unique alpha-numeric identification that follows the format "YYDDD-Z" where:

- YY is the year the sample was taken;
- DDD is the Julian date of sample collection;
- X is the Decision Unit designation; and
- Z is the Sampling Unit designation.

Each sample container will have a sample label. The sample identification number will be logged in the field log book, along with the following information about the sampling event:

- Sampling personnel;
- Date and time of collection;
- Observations on ambient conditions;
- Decision Unit / Sampling Unit designations;
- Method of sampling; and
- Intended sample processing methods and analyses.

3.3.3 Sample Labels

Each sample container (Ziploc storage bag) will be labeled with the following information: unique sample number, date, time, project name and/or number, and sampler's initials. Indelible ink will be used to record information on the sample label.

3.3.4 Custody Seals

Custody seals will be used when a sample shipment is picked up by the laboratory or sent to the laboratory by overnight courier. Signed and dated custody seals will be attached to the top of the shipping container in such a way that it is necessary to break the seal to open the container. Custody seals ensure that any tampering during transportation will be detected by the receiving laboratory.

3.3.5 Chain-of-Custody Forms

Chain-of-custody forms provide the documentation to trace sample possession from the time of sample collection until receipt by the laboratory. One chain-of-custody form will be filled out for each cooler or shipping container and will list all the samples contained in the cooler or container. One copy of the completed form will be placed in a plastic bag and taped to the inside lid of the shipping container and one copy will be kept with the project files.

3.4 Field Activity Logs

3.4.1 Introduction

A field logbook will be maintained to record the details of field investigation activities and field data. This logbook will be bound and will have sequentially numbered pages. Entries will be written in indelible ink and will be initialed and dated by the field personnel recording the information. Several types of field activity logs will be maintained, including site health and safety logs, equipment calibration logs, and field sampling logs.

3.4.2 Field Sampling Logs

In addition to the descriptions of field investigation activities and field data recorded in the field log book, details of sampling information may be provided on field sampling logs. Field sampling logs will generally include the following information:

- date and weather;
- personnel;
- time and description of investigative activities;
- sample medium and type (i.e., grab, composite, ISM, duplicate, etc.);
- sample collection technique(s);
- sample containers, analyses, and preservatives;
- sample number, location, and depth;
- sampling times; and

- pertinent field observations.

3.4.3 Corrections to Documentation

All documents will be completed in permanent, waterproof ink. None of the field documents are to be destroyed or thrown away, even if they are damaged or contain inaccuracies that require a replacement document. Corrections will be made by crossing out mistakes with a single line and then dating and initialing the correction. The use of correction fluid is not permissible. The documents used during the field investigation will remain on-site in the field office during the field effort.

4 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

4.1 Control Parameters

QA/QC involves the collection of field QC samples, as well as control of field operations, sampling, and measurements as described below.

4.2 Field QC Samples

One field duplicate QC sample will be collected for every 20 environmental samples. Because this Workplan calls for a total of 21 total ISM samples, one filed duplicate will be collected during the ISM.

Because the Workplan already calls for at least two replicates within each SU, the field duplicate for this sampling event will be a split from one of the multi-increment samples collected in a SU. This split sample will be labeled so that the laboratory cannot distinguish it as a field duplicate. Thus, the field duplicate will provide a check on the variability arising from laboratory processing and subsampling activities. No trip blanks or equipment rinsate blanks will be collected or analyzed. Trip blanks are used to evaluate contamination related to sample packaging or handling during the shipping process and are typically used only for volatile analytes. Equipment rinsate blanks are used to evaluate the effectiveness of equipment decontamination between samples. Because ISM requires less sampling handling equipment and results fewer total samples submitted to the laboratory, there are fewer opportunities for cross-contamination. As described in Section 3.2, the drill bits used for the ISM sample collection will be thoroughly decontaminated between each multi-increment sample.

4.3 Field Operations

Control of field operations and sampling methods will be established through by ensuring that each field team member is familiar with the provisions of the Workplan, and HASP. Also, the EPS Project Manager will ensure that each field team member is familiar with the Workplan prior to implementation of field activities. The EPS Project Manager will also provide a QA review of field activities at the beginning of the sampling event to ensure that all procedures are followed and at least one additional time during the execution of this project for each sampling team through on-site monitoring of representative field activities. The Project Manager will regularly check field notebooks and forms.

5 DATA QUALITY EVALUATION AND REPORTING

5.1 Data Quality Evaluation

EPS will store the data in an MS Access normalized relational database. A database is defined as a large collection of data organized especially for rapid search and retrieval. Data are organized into standardized, structured tables that are specifically related to one another. MS Access is an industry-standard relational application for small to medium databases.

Before data is added to the database, it undergoes a validation process. In the case of hand written notes and hard copies, records are manually entered into an electronic spreadsheet, checked twice by two different people. Electronic records are then imported into a separate database (Build database) where several queries are used to perform additional data validation. In order to maintain internal consistency, each parameter is spell checked to ensure proper encoding, each Sample ID and date pair is evaluated to prevent duplicate entries, and all data are checked for proper units, methods, and matrix types.

The database is designed for use by two classes of users: the Database Manager (DM) and the End-User. A DM designs and maintains the structure of the database, appropriately prepares data for entry (outside of Access), correctly executes validation tests within Access during data entry, and informs end-users of any limitations to the dataset. An End-User queries data for day-to-day work (analysis, reports, thought experiments, etc.) and links data to outside applications (GIS, outside databases). There is one DM and any number of End-Users.

The database is not simply one database, but rather a collection of three separate databases: Build, Master, and Main. The Build database links directly to the Master database and is used exclusively by the DM to validate, format, and finally enter data into the Master database. The Master database stores all the data and is managed only by the DM. The Main database is an exact replicate of the Master database that is linked to by End-Users for day-to-day work. When changes are made to the Master database it is copied over to the Main database. This procedure, known as "compacting", ensures that the Main database always has the most up to date records, and that there is separation between the original records and those used on a daily basis.

The work necessary to validate raw data is performed in queries. A query in its basic form allows the user to select fields for a table or multiple tables. Queries can also perform statistical calculations, replace values, add and remove records, create and delete tables. Because of the

heterogeneity of the raw data, DMs modify queries and update key fields in order to maintain proper encoding. The following is a step by step process used to "clean" raw data:

- Raw data are imported into a temporary table that has the same structure as the Master database's Data table.
- Each set of raw data is assigned a batch number in order to track its addition.
- Raw data are checked for duplicate records. If duplicate records exist, they are assigned the proper Dup code. The database is designed to store all duplicate records that often are the result of multiple analysis methods and lab replicates. Original values are given a Dup code of 0. Duplicate records are given values that are the sum their duplicate characteristics. Characteristic codes are listed below:

  1 - Duplicate sample sent to the same lab (often with a different Sample ID)
  2 - Split sample sent to different lab: generally with the same Sample ID
  4 - A duplicate analysis by the same lab generally by another method
  8 – A duplicate due to reporting both the diluted and undiluted result
  16 – Miscellaneous
- The analyte names are checked for spelling to ensure proper encoding.
- Units and Methods are checked to ensure proper encoding.
- Missing values are checked in order to prevent errors of omission.
- Sample ID / Date pairs are checked.
- Sample IDs in the raw data are cross-checked with existing locations. New locations are added when necessary.
- All raw records are checked against the Master database's Data table to prevent duplicate entries.
- "Clean" data are added to the Master database.
- All temporary tables are deleted.

Note that all data are actually entered into the database. "Clean" data are to be used without qualification, whereas other data flagged during the data review process are to be used with appropriate professional judgment. Instead of being thrown out, all data is categorized to allow database End-Users flexibility in analyzing data: Records are given Dup codes, data quality flags, matrix codes, area designations, etc. Because the database is a living database, DMs often have to modify table structures and add keys to key tables to input new sources of data in order to categorize additional records. These modifications do not change existing records, but instead build upon them.

5.2 Data Reporting

Data deliverables from the analytical laboratory will consist of the following items:

- Case Narrative;
- Laboratory Final Reports;
- Surrogate Recovery Summary;
- Matrix Spike/Matrix Spike Duplicate Recovery Summary;
- Method Blank Summary;
- Laboratory Control Sample (LCS) Recovery Summary;
- Initial Calibration Summary Gas Chromatograph (GC) Method Printout;
- Continuing Calibration Summary;
- Analytical Sequence Printout;
- Chromatographs and Quantification Reports for all Samples, Standards, and QC Samples;
- Copies of Extraction Log Pages; and
- Copies of Chain-of-Custody Document.

For consistency and ease of review, the data deliverables will be organized in the same manner. The arrangement will be as follows:

- Sample Narrative;
- Final Reports;
- QC Summary Information;
- Analytical Sequence Printout(s);
- Sample Raw Data (arranged by sample number);
- Instrument Calibration Data (in chronological order);
- Raw QC Data;
- Blanks;
- LCS;
- Matrix Spike/ Matrix Spike Duplicate (MS/MSD);
- Extraction Logbook Pages; and
- Chain-of-Custody Documents.

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TABLES

Table 1. Results of 1995 Samples for Dioxins/Furans

		113036 (Process South)			113037 (Cell Building)		
Analyte	Congener-Specific TEF (unitless)	Surface Soil Concentration (ng/kg)		TCDD-TEQ Concentration (ng/kg)	Surface Soil Concentration (ng/kg)		TCDD-TEQ Concentration (ng/kg)
2,3,7,8-TCDD	1	1.5		1.5	2.2		2.2
1,2,3,7,8-PeCDD	1	2.8	U	1.4	0.8	U	0.4
1,2,3,4,7,8-HxCDD	0.1	13		1.3	5		0.5
1,2,3,6,7,8-HxCDD	0.1	6.8		0.68	5.2		0.52
1,2,3,7,8,9-HxCDD	0.1	5		0.5	1.8		0.18
1,2,3,4,6,7,8-HpCDD	0.01	110		1.1	56		0.56
OCDD	0.0003	370		0.111	380		0.114
	Total CDD-TEQ			**6.6**			**4.5**
2,3,7,8-TCDF	0.1	53		5.3	99		9.9
1,2,3,7,8-PeCDF	0.03	130		3.9	190		5.7
2,3,4,7,8-PeCDF	0.3	340		102	110		33
1,2,3,4,7,8-HxCDF	0.1	3400		340	1200		120
1,2,3,6,7,8-HxCDF	0.1	440		44	280		28
1,2,3,7,8,9-HxCDF	0.1	71		7.1	99		9.9
2,3,4,6,7,8-HxCDF	0.1	1300		130	120		12
1,2,3,4,6,7,8-HpCDF	0.01	12000		120	1700		17
1,2,3,4,7,8,9-HpCDF	0.01	340		3.4	340		3.4
OCDF	0.0003	5900		1.77	4100		1.23
	Total CDF-TEQ			**757.5**			**240.1**
	Total TEQ			**764.1**			**244.6**

U - Result is below reported detection limit. 1/2 of the reported detection limit used in TEQ calculation.

FIGURES

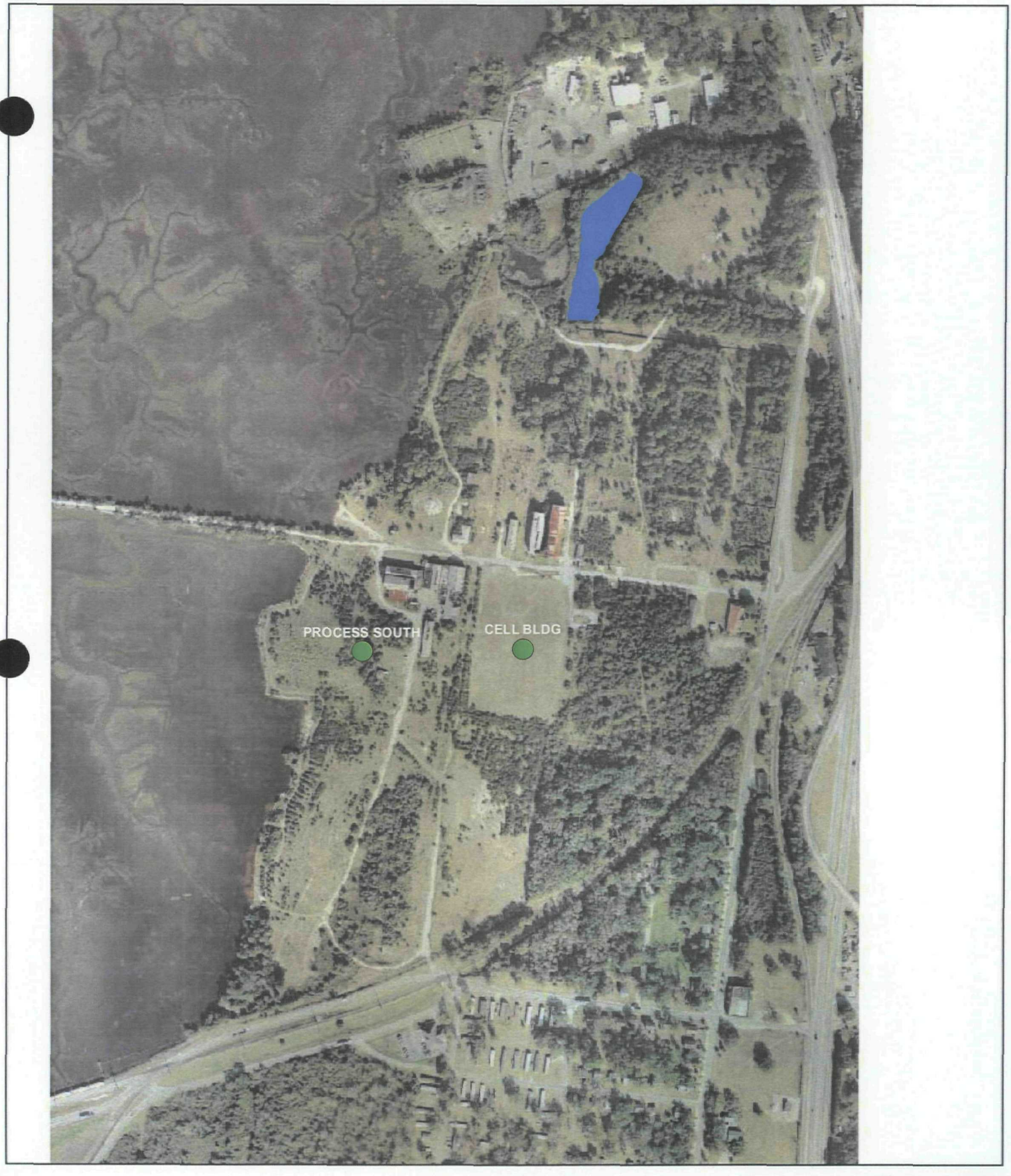

Figure 1. Locations of EPA Upland Soil Samples for Dioxin/Furans

Figure 2. Post Removal Aroclor 1268 in Surface Soil

Figure 3. Post Removal Aroclor 1260 in Surface Soil

Figure 4. Post Removal Aroclor 1254 in Surface Soil

Figure 5a. Proposed ISM Sampling Units - Quadrant 1

Figure 5b. Proposed ISM Sampling Units - Quadrant 2

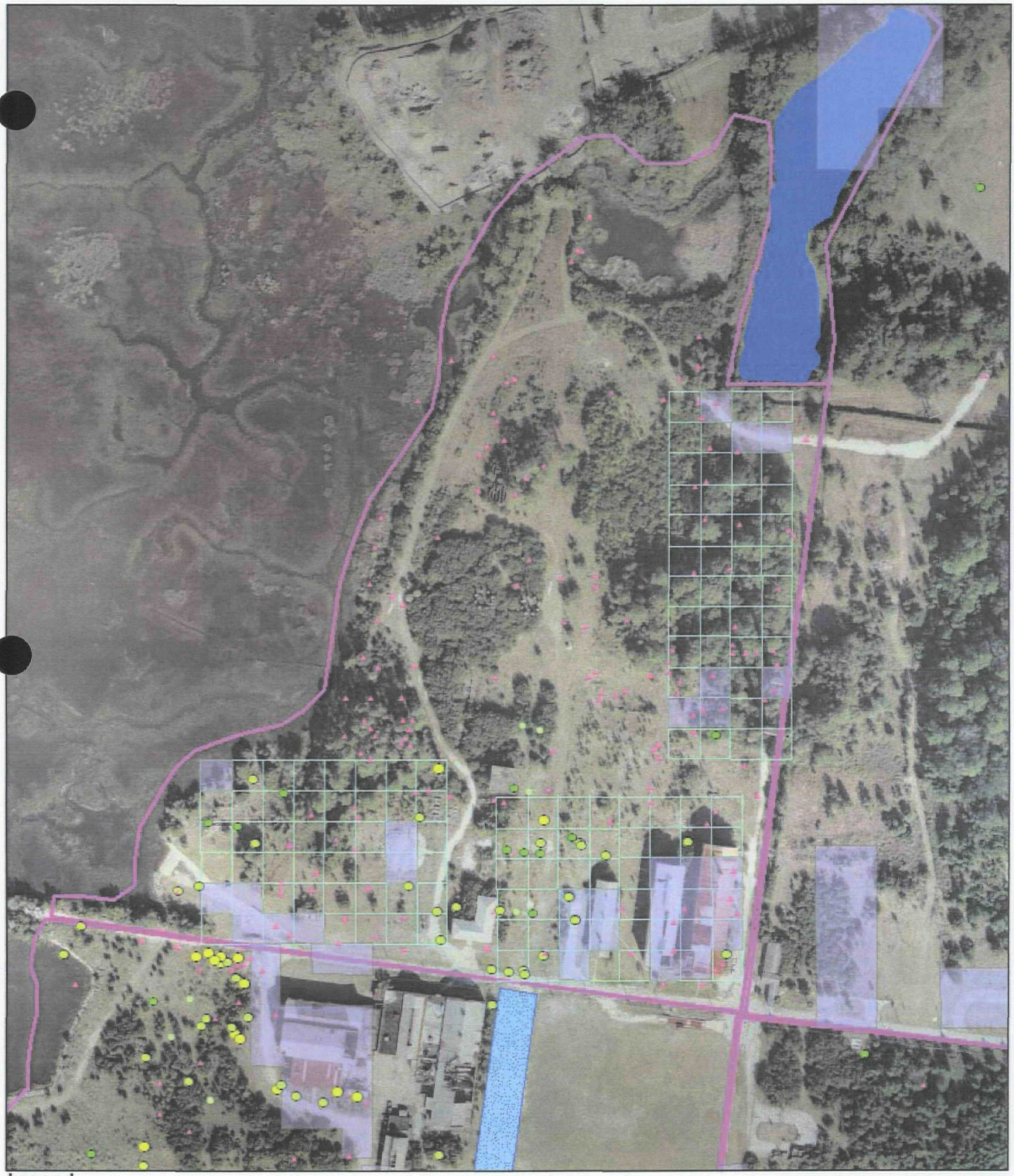

Figure 5c. Proposed ISM Sampling Units - Quadrant 3

Figure 5d. Proposed ISM Sampling Units - Quadrant 4

APPENDIX A

Title: Multi-Incremental Subsampling of Soils and Sediments

Approvals (Signature/Date):

Patrick Rainey — Date 2/17/09
Technical Manager

Joe Schairer — Date 2/18/09
Health & Safety Manager / Coordinator

Douglas Weir — Date 2/24/09
Quality Assurance Manager

Karla Buechler — Date 2/24/09
Laboratory Director

1. SCOPE AND APPLICATION

1.1. The purpose of this procedure is to obtain sub-samples from client provided samples which represent the concentration of material in the entire parent sample.

1.2. This SOP describes the procedures for laboratory staff to follow during the preparation of samples for the multi-incremental sampling procedure. These are guidelines for the preparation and subsampling of samples to be analyzed for routine organic and inorganic analyses.

1.3. The multi-incremental subsampling procedures are not applicable to volatile soil samples collected in Encore® samplers for Method 5035. These are discrete samples and the entire sample is used for analysis.

2. SUMMARY OF METHOD

2.1. Samples received from the field may require processing including drying, removal of extraneous material, and sieving to be performed for different analyses so that a representative concentration can be determined. An entire client sample is first processed and the sample is then sub-sampled using a multi-incremental sampling approach.

2.2. Care should be taken to ensure that these subsamples are representative of the component samples and are properly prepared and stored in accordance with the appropriate method of analysis.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

4. INTERFERENCES

4.1. Interferences can occur when using scoops or spatulas. All scoops or spatulas should be used for only one sample, and then disposed, or thoroughly cleaned between samples. Material that may be acceptable for one analysis may cause contamination for another analysis. All plastic should be avoided if organic parameters are requested.

4.2. Volatile analytes may be lost during subsampling from non-Encore containers. Subsampling for volatile analyses should be done from a previously unopened

container or the end of tube (where possible), and subsampling should be done as quickly as possible to avoid analyte loss.

4.3. Volatile and light semi-volatile analytes may be lost during the sample drying and grinding procedures. Consult the appropriate analytical SOP and QAS for guidance on the required drying and grinding procedure for the samples.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Extensive homogenization, subsampling, and/or compositing of soil/solid/waste or liquid samples presents an extreme risk of repetitive motion injuries for the individual performing the operation. No single employee will homogenize, sub-sample, or composite these types of samples for longer than one hour continuously without taking a five-minute break away from this type of work and stretching his/her hands, wrists and arms. If the manager/supervisor and the employee involved identify at the start of the process that the work will take longer than one hour, the employee should take mini-breaks of 2-3 minutes every 25-30 minutes. If there is extensive homogenization, subsampling, and/or compositing that must be performed, or if it is extremely time sensitive, managers/supervisors must assign additional personnel to the effort, or rotate different staff members through the job in order to prevent injury to any employee.

5.1.2. If sediment/soil samples have been frozen in glass jars, the freezing process may have cracked the jars when the sample expanded while freezing. After the samples have thawed, wear cut protective gloves while handling the jars until it can be confirmed that they have not cracked.

5.1.3. Any alternative procedures requested by a client must be reviewed by EH&S before they are put into practice.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide sufficient

protection when handling closed sample containers and most typical samples. Unusual or heavily contaminated samples must be evaluated to determine if there are any hazards for which a particular type of glove will not be appropriate.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable; therefore all samples must be opened, transferred, sub-sampled and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, moving heavy shipping containers, unloading shipping containers, manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

There are no materials used in this method, which have a serious or significant hazard rating.

6. EQUIPMENT AND SUPPLIES

6.1. Metal trays (or other appropriate material).

6.2. Aluminum foil or other inert tray cover material.

6.3. Sieves, various sizes, including 2mm (#10 sieve).

6.4. Stainless steel or disposable wooden spatulas (flat-ended and rounded).

6.5. Analytical balance.

6.6. Mortar and pestle (manual and automated).

6.7. Spoons, various sizes and materials.

6.8. Sample containers, various sizes, glass and poly.

6.9. Fume hood.

Note: *Some samples such as biological tissues and pulp and paper products may require pre-preparation before subsampling or compositing. See the appropriate SOP for matrix specific procedures.*

7. REAGENTS AND STANDARDS

Reagents used for rinsing equipment are indicated in method SOPs.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

All component, subsamples, and composites will be stored in compliance with the analytical methods under which they will be analyzed.

9. QUALITY CONTROL

Samples used for Matrix Spike and Matrix Spike Duplicates should be homogenized and subsampled using the same procedure as all batch samples.

10. CALIBRATION

10.1. Balances used for subsampling or compositing for analysis and preparation should be calibrated as per SOP WS-QA-0041.

10.2. Balances used for non-analytical subsampling, i.e., for trans-shipment, do not require calibration, as the weight is a rough value only.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. This method is dependent on the client/project provided Data Quality Objectives. Depending on the nature of the project, samples may need to be dried, extraneous material may need to be removed and replicates may need to be run per sample or per batch of samples. It is important that the analyst confirm with the Project Manager prior to performing this procedure. Any project specific changes or modifications to the procedure should be noted in the form of a client specific amendment to the SOP or in the Quality Assurance Summary (QAS). The procedures documented below

incorporate the commonly performed procedure. There are two primary procedures to consider, sample prep and sub-sampling.

11.3. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

Note: *Drying, sieving or subsampling for volatile analyses may lead to the loss of analytes or contamination from common laboratory solvents. Any subsampling procedures for volatiles should be performed in a solvent-free area and the mixing should be minimized to reduce the loss of volatile analytes. The potential for loss of volatile analytes should be discussed with the client before initiation of the program.*

11.4. Sample Preparation

11.4.1. Soils, solids or wastes: If only the solid material is to be tested, decant off the freestanding liquid. If the liquid and solid components are to be analyzed together, mix the sample. If samples do not require drying, remove and discard any large sticks, rocks or other materials that cannot be homogenized, unless specifically requested otherwise. Document (via NCM or benchsheet) the removal of liquid and foreign matter from the sample and proceed with subsampling. Perform the subsampling as quickly as possible to reduce the loss of sample moisture during the process.

11.4.2. Sediment samples: Remove the samples from the freezer and allow them to thaw for a minimum of two hours before homogenizing. Mix all free-standing water into the sediments unless otherwise specified. If samples do not require drying, remove and discard any large sticks, rocks or other materials that cannot be homogenized, unless specifically requested otherwise. Document (via NCM or benchsheet) the removal of liquid and foreign matter from the sample and proceed with subsampling. Perform the subsampling as quickly as possible to reduce the loss of sample moisture during the process.

WARNING: If sediment/soil sample have bbeen frozen in glass jars, the freezing process may have cracked the jars when the sample expanded while freezing. Wear cut protective gloves while handling the jars until it can be confirmed that they have not cracked.

11.4.3. When drying is needed, spread the entire sample evenly on a tray coated with aluminum foil or other inert material that is free from any analytes of interest or intereferences. Perform the processing in a fume hood, or in a well ventilated area to minimize exposure to dust. Moist samples should be placed on bakers racks or another location which allows for proper ventilation, and allowed to air-dry for a minimum of 24 hours.

11.4.4. When the samples have become dry, remove any obvious organic materials such as leaves and twigs. Carefully sieve the sample from the metal tray

using a 2mm sieve, or the appropriate size as designated in the method SOP or QAS. Break up any soil aggregate material by crushing against the screen with a clean object such as a mortar or the round side of a spoon. Employ disaggregation techniques if needed. In some cases a smaller mesh size sieve may need to be used, following 2mm sieve. Clients may ask to weigh the individual portions for particle size analysis. Record all observations on the appropriate laboratory benchsheet. Be certain to adequately decontaminate sieves, mortars, scoops etc. by wiping with clean towels and brushing as needed, followed by washing with soap and water and rinsing with the appropriate solvent or reagent as outlined in the appropriate analytical procedure.

11.4.5. Transfer the materials with particle size greater than the designated particle size (sieve mesh) back into the original sample container. The portion of the sample that has been prepared for grinding or sub-sampling is now ready for processing. Refer to the grinding procedure for specific information on the appropriate grinding procedure.

11.4.6. Label the appropriate sized sample container for the subsample with the laboratory sample ID.

11.5. Subsampling for Soils/Solids and Sediments

11.5.1. Multi-Incremental: Evenly distribute prepared materials onto a tray covered with aluminum foil, butcher paper or other inert cover. The sample layer should have a depth of approximately one-half inch or less to allow for sampling throughout the entire depth of the sample layer.

11.5.2. For inorganic metals digestion, use a smaller scoop to sub-sample an aliquot size of approximately 1 g or more (0.6 g are typically used for mercury analysis) and store in a pre-cleaned container. For organic extraction, use a flat-ended metal scoop to obtain an aliquot size of at least 10 to 30 g or larger, dependent upon the analytical method, into a glass jar.

11.5.3. Perform the sub-sampling by taking random scoops from 30 locations on the tray. The scoop should be taken evenly from the top to the bottom of the soil. Each scoop should represent approximately 1/30th of the desired target mass. This may be very difficult for analyses requiring only a small sample size. A larger than required sample may be collected in this manner and a subsample removed after homogenization. Practice on a few scoops before proceeding on the entire sample by measuring the weight of one scoop of soil. This may be done less frequently with knowledge of the type of soil. Once the desired amount is achieved, take scoops to represent the entire area of the tray in a random fashion. Record the final weight which should be approximately 1 g for inorganics analyses and 10 to 30 g for organic analyses. If the final

amount is less than the required amount, take a few more scoops. If the amount is much greater than the desired amount, it may be necessary to further homogenize the incremental-subsample and remove an appropriate sized sample for the analysis.

Note- Sediment samples may require the extraction of a larger sample size to correct for moisture content.

11.5.4. Store the sub-samples in the proper location for the test being conducted until they are ready for further preparation

12. CALCULATIONS/DATA REDUCTION

This section is not applicable to this procedure.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP SAC-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Contaminated disposable materials such as plastic vials, pipettes, empty sample containers, unused/excess sample matrix and disposable spatulas. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. STL White Paper "Representative Sub-sampling Techniques for Inorganic Analytes", February 23, 2004.

16.2. "Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples," USEPA, November 2003.

16.3. "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities" ASTM D 6323-98 (Reapproved 2003)

16.4. EPA SW-846, Method 8330B. Nitroaromatics, Nitramines, and Nitrate esters By High performance Liquid Chromatography (HPLC), Revision 2, October 2006.

17. METHOD MODIFICATIONS

17.1. There are no deviations from the method.

18. ATTACHMENTS

18.1. No attachments are present.

19. REVISION HISTORY

19.1. WS-QA-0028, Revision 3, Effective 03/01/2009

19.1.1. Editorial changes.

19.2. WS-QA-0028, Revision 2, Effective 9/25/07

19.2.1. This SOP format was updated to TestAmerica format.

19.3. WS-QA-0028, Revision 1, Effective 7/23/07

19.3.1. This is the first edition of this SOP.

EPS

APPENDIX B

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

METHOD SUMMARY & DATA QUALITY OBJECTIVES

TestAmerica West Sacramento
SW8290: Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography / High Resolution Mass Spectrometry (HRGC/HRMS)

This method provides instrument and extraction procedures for the detection and quantitation of PCDDs (tetra through octa-chlorinated homologues) and PCDFs (tetra through octa-chlorinated homologues) in a variety of sample matrices in part-per-trillion (ppt) to part-per-quadrillion (ppq) concentrations.

Method SW8290 is used to detect dioxins and furans in variety of matrices and uses additional quality controls to allow more sophisticated determinations of detection limits and target analyte concentrations than other routine GC and GC/MS methods.

Method SW8290 requires that isotopically labeled analogs of target analytes be spiked into each sample before extraction, and uses nine ^{13}C labeled analogs, one furan and one dioxin at each chlorination level. 13C-OCDF is not used as an internal standard due to its potential interference with OCDD and 13C-1,2,3,7,8,9-HxCDD is used as a recovery standard. By adding a known amount of labeled compounds to every sample prior to extraction, correction for recovery of the target analytes can be made because the target analytes and their labeled analog exhibit similar effects upon extraction, cleanup, concentration, and gas chromatography. Target analytes are quantitated relative to the labeled analog and therefore their calculated concentration compensates for extraction and cleanup efficiencies.

A batch specific LCS (Laboratory Control Sample) is not required by Method 8290, however, TestAmerica West Sacramento still analyzes an LCS at a frequency of 1 per batch of 20 samples as an ongoing system and standard check. The target analyte concentrations for the LCS are given in Table 2. Sample matrix spikes and/or spike duplicates are performed only at client request. The spike concentrations are nominal values based on a full volume sample preparation (1000 mls for liquids and 10 grams for solids). If less than a full volume of sample is prepared due to sample matrix, sample availability, or method requirements, the spike amount will remain constant and therefore the spike concentrations will vary. See Table 2 through Table 4 for specific QC control and corrective action measures.

Detection Limits and Reporting Limits:

TestAmerica West Sacramento's Method SW8290 provides customizable options to report detection limits and/or reporting limits.

- Reporting Limit (RL) - When target analytes meet method identification criteria and are free of interferences, they are reported down to the lowest calibration standard concentration (see reporting limits in Table 1). Data can be reported to the RL without the use of qualification if required.
- Estimated Detection Limit (EDL) - For each analyte not detected, an EDL can be reported. The sample specific EDL is an estimate of the concentration of a given analyte that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, etc. Because of the toxicological significance of dioxins, the EDL value can be reported for non-detected chemicals rather than reporting the reporting limit (RL). Any analyte with a peak greater than 2.5 times the noise and meets all qualitative requirements but less than the RL would be reported with a "J" flag.
- Method Detection Limit (MDL) – Qualitatively confirmed analytes are reported as "estimated" down to the statistically derived MDL to denote the less certain quantitation and the value is qualified with a "J" flag. Any peak with a calculated concentration below the MDL is reported as "not detected" with no further qualification.

Second column confirmation will be performed only for 2,3,7,8-TCDF positives as per the convention selected..

Toxicity Equivalence Factors (TEFs)

As per client request, the 2,3,7,8-TCDD toxicity equivalence can be calculated in accordance with the procedures given in one of three different formats:

- TEF values cited in the U.S. Environmental Protection Agency, (1989) "Interim procedures for estimating risks associated with exposures to mixtures of chlorinated dibenzo-p-dioxins and –dibenzofurans (CDDs and CDFs) and 1989 update. U.S. Environmental Protection Agency, Risk Assessment forum, Washington DC; (EPA 625/3-89/016)."
- "WHO TEFs for human risk assessment based on the conclusions of the World Health Organization meeting in Stockholm, Sweden, 15-18, June 1997 (Van den Berg et aL, 1998)."
- "WHO TEFs for human risk assessment based on the conclusions of the World Health Organization meeting in Geneva, Switzerland, June 2005."

TEFs are assigned to each 2,3,7,8-substituted PCDDs/PCDFs in order to relate their toxicity to that of 2,3,7,8-TCDD. See Table 6 for the factors used to calculate TEFs. Note that EDL and detection limit values are not normally included in the TEQ adjusted concentration.

Uniform Federal Policy for Quality Assurance Project Plan (UFP-QAPP) Worksheets

UFP – QAPP Worksheets for Method 8290 pre-filled in with laboratory specific information are available upon request. Available tables include:

- Table 12 – Measurement Performance Criteria Table (Field QC and Laboratory QC Samples).
- Table 15 – Reference Limits and Evaluation Table
- Table 19 – Analytical SOP Requirements Table
- Table 23 – Analytical SOP References Table
- Table 24 – Analytical Instrument Calibration Table
- Table 25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
- Table 28 – Laboratory QC Samples Table
- Table 30 – Analytical Services Table

All tables are available in Microsoft Excel format for easy import into your proposal. Please ask your Project Manager for details.

TABLE 1 REPORTING LIMITS (RLs)
Based on Lower Calibration Limits
Method 8290 – TestAmerica

Lower detection limits are achievable using the estimated detection limit option.

Analyte	Water[1] (pg/L) RL	Soil/Sediment/Tissue[2] (pg/g) RL	Waste[3] (pg/g) RL
Dioxins			
2,3,7,8-TCDD	10	1.0	100
1,2,3,7,8-PeCDD	50	5.0	500
1,2,3,4,7,8-HxCDD	50	5.0	500
1,2,3,6,7,8-HxCDD	50	5.0	500
1,2,3,7,8,9-HxCDD	50	5.0	500
1,2,3,4,6,7,8-HpCDD	50	5.0	500
OCDD	100	10	1000
Furans			
2,3,7,8-TCDF	10	1.0	100
1,2,3,7,8-PeCDF	50	5.0	500
2,3,4,7,8-PeCDF	50	5.0	500
1,2,3,4,7,8-HxCDF	50	5.0	500
1,2,3,6,7,8-HxCDF	50	5.0	500
1,2,3,7,8,9-HxCDF	50	5.0	500
2,3,4,6,7,8-HxCDF	50	5.0	500
1,2,3,4,6,7,8-HpCDF	50	5.0	500
1,2,3,4,7,8,9-HpCDF	50	5.0	500
OCDF	100	10	1000

Note: "Totals" values are available upon client request.

[1] Based upon a 1.0 liter sample aliquot. Sensitivity of the method depends on the level of interferences rather than instrumental limitations.

[2] Based upon a 10.0 gram sample aliquot. Maximum RL for samples "as received". Correction for moisture content may raise reporting limits above these levels.

[3] Based upon a 0.1 gram sample aliquot. Maximum RL for samples "as received". Correction for moisture content may raise reporting limits above these levels. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

TABLE 2 CONTROL LIMITS FOR LABORATORY CONTROL SAMPLES (LCS), MATRIX SPIKES and MATRIX SPIKE DUPLICATES
Method 8290 – TestAmerica

Target Compound	LCS/MS/MSD Control Limits (Soil/Sediment)				LCS/MS/MSD Control Limits (Water)			
	AMT (pg/g)	Lower Control Limit	Upper Control Limit	RPD	AMT (pg/L)	Lower Control Limit	Upper Control Limit	RPD
Dioxins								
2,3,7,8-TCDD	20	77	133	20	200	71	128	20
1,2,3,7,8-PeCDD	100	74	145	20	1000	74	139	20
1,2,3,4,7,8-HxCDD	100	68	146	20	1000	65	144	20
1,2,3,6,7,8-HxCDD	100	79	141	20	1000	73	142	20
1,2,3,7,8,9-HxCDD	100	68	139	20	1000	60	147	20
1,2,3,4,6,7,8-HpCDD	100	74	147	20	1000	79	137	20
OCDD	200	75	153	20	2000	71	147	20
Furans								
2,3,7,8-TCDF	20	80	146	20	200	75	142	20
1,2,3,7,8-PeCDF	100	84	143	20	1000	80	140	20
2,3,4,7,8-PeCDF	100	76	157	20	1000	71	144	20
1,2,3,4,7,8-HxCDF	100	78	141	20	1000	64	149	20
1,2,3,6,7,8-HxCDF	100	78	144	20	1000	56	161	20
2,3,4,6,7,8-HxCDF	100	73	157	20	1000	60	169	20
1,2,3,7,8,9-HxCDF	100	70	144	20	1000	53	163	20
1,2,3,4,6,7,8-HpCDF	100	79	143	20	1000	78	141	20
1,2,3,4,7,8,9-HpCDF	100	79	150	20	1000	80	146	20
OCDF	200	70	158	20	2000	76	147	20

Note:
Native compound limits are TestAmerica West Sacramento historical limits and are subject to change.
RPD limits are currently set to the method default of 20%.
Tissue and waste control limits are available upon request.

TABLE 3 CONTROL LIMITS FOR INTERNAL STANDARDS
Method 8290 – TestAmerica

Internal Standard Compound	Internal Standards Control Limits (Soil/Sediment)			Internal Standards Control Limits (Water)		
	AMT (pg/g)	Lower Control Limit	Upper Control Limit	AMT (pg/L)	Lower Control Limit	Upper Control Limit
Dioxins						
13C-2,3,7,8-TCDD	200	40	135	2000	40	135
13C-1,2,3,7,8-PeCDD	200	40	135	2000	40	135
13C-1,2,3,6,7,8-HxCDD	200	40	135	2000	40	135
13C-1,2,3,4,6,7,8-HpCDD	200	40	135	2000	40	135
13C-OCDD	400	40	135	4000	40	135
Furans						
13C-2,3,7,8-TCDF	200	40	135	2000	40	135
13C-1,2,3,7,8-PeCDF	200	40	135	2000	40	135
13C-1,2,3,4,7,8-HxCDF	200	40	135	2000	40	135
13C-1,2,3,4,6,7,8-HpCDF	200	40	135	2000	40	135

Note:
Method default control limits. Signal-to-noise is also evaluated for data acceptability. These labeled analytes are spiked into all samples. Tissue and waste control limits are available upon request.

TABLE 4 SUMMARY OF CALIBRATION PROCEDURES
Method 8290 – TestAmerica

Calibration	Frequency	Acceptance Criteria	Corrective Action
Tune using PFK.	Prior to sample analysis and at the end of the analytical sequence (no time limit for the ending PFK analysis).	Resolving power ≥10,000 at m/z=304.9824 & m/z=380.9760 ± 5 ppm of expected mass.	1) Retune instrument. 2) Reanalyze PFK. 3) End resolution acceptable "as is" – assess data for impact if resolution is less than 10,000 and narrate or reinject as necessary.
Column Performance Check Solution (CPSM). Solution includes the Window Defining Mix.	Prior to 12 hrs of sample analysis.	Used to set retention times of first and last eluters. CPSM must have ≤25% valley resolution for 2,3,7,8-TCDD	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.
(5 point ICAL) Multipoint calibration.	Initially and as required.	1) I.S. = %RSD<30% 2) Natives = %RSD<20% 3) Retention time must be within -1 to +3 seconds of labeled I.S. or 0.005 RRT units. 4) Ion ratios within Table 5 limits, and I.S. S/N ≥10:1 and Natives S/N ≥2.5:1	1) Evaluate system. 2) Recalibrate. 3) If all criteria are met except #4 (ratio), evaluate impact, narrate and report if no impact is found.
Daily Continuing Calibration Verification standard (CCV).	Once per 12 hours, prior to sample analysis and at the end of the analytical sequence (no time limit for the ending CCV).	1) %D of I.S. ≤30% from avg. RRF (ICAL). (Ending %D of I.S. ≤35% from avg. RRF). 2) %D of natives ≤20% from avg. RRF (ICAL). (Ending %D of natives ≤25% from avg. RRF). 3) Retention time must be within -1 to +3 seconds of labeled I.S. or 0.005 RRT units. 4) Ion ratios within Table 5 limits, and I.S. S/N ≥10:1 and Natives S/N ≥2.5:1 5)	1) Evaluate system. 2) Evaluate data for usability. 3) Reanalyze (CCAL). 4) Recalibrate (ICAL) as necessary.

TABLE 4 SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES
Method 8290 – TestAmerica

QC Element	Frequency	Acceptance Criteria	Corrective Action
Internal Standards	Every sample, method blank, and LCS.	1) Internal standard recovery within limits stated in Table 2.	1) Check chromatography for interferences. If found, flag data. 2) Check S/N. If < 10:1, re-extract sample. 3) If S/N > 10:1, evaluate data usability, flag, narrate and report. 4) Check instrument and re-analyze the extract if a problem is found and corrected. 5) Re-extract and re-analyze adversely affected samples.
Method blank	1 per analytical batch, not to exceed 20 field samples per matrix.	No target analyte concentrations above the reporting limit (RL). Exception: OCDD concentration in the method blank is allowed to be 5X the RL without narration. Note "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for positive totals in the method blank.	1) Re-analyze method blank if instrument carryover is suspected. 2) If still exceeds and analyte concentration in sample < RL or > 10X blank concentration, narrate and report results. 3) If "J" qualified positives are in the method blank or OCDD < 5X the RL, then no corrective action is necessary. Flag and report 4) If non-compliant and analyte concentration in sample is between RL and 10X blank concentration, re-extract and re-analyze affected samples.
Laboratory Control Sample	1 per analytical batch, not to exceed 20 field samples per matrix.	Refer to Table 2.	1) Review Internal Standards, as above. 2) Evaluate data for usability. 3) If sample results are ND and RL are met, no action is required – narrate and report. 4) If samples have positives > RL, re-extract and re-analyze affected samples for analytes outside the acceptance criteria.
Duplicates	As per client request.	Refer to Table 2 and Table 3.	1) Review data for usability. 2) Narrate outliers.
Matrix Spike	As per client request.	Refer to Table 2 and Table 3.	3) Review data for usability. 4) Narrate outliers.
Matrix Spike Duplicate	As per client request.	Refer to Table 2 and Table 3.	1) Review data for usability. 2) Narrate outliers.

TABLE 5 CRITERIA FOR ISOTOPIC RATIO MEASUREMENT FOR PCDDs AND PCDFs
Method 8290 – TestAmerica

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits (± 15%)
4	M/(M+2)	0.77	0.65-0.89
5	(M+2)/(M+4)	1.55	1.32-1.78
6	(M+2)/(M+4)	1.24	1.05-1.43
6[a]	M/(M+2)	0.51	0.43-0.59
7[b]	M/(M+2)	0.44	0.37-0.51
7	(M+2)/(M+4)	1.04	0.88-1.20
8	(M+2)/(M+4)	0.89	0.76-1.02

[a] Used only for ^{13}C-HxCDF (Internal Standard)
[b] Used only for ^{13}C-HpCDF (Internal Standard)

TABLE 6 PCDDs/PCDFs TOXICITY EQUIVALENCE FACTORS (TEF)
Method 8290 – TestAmerica

Analyte	TEF March 1989 (EPA 62/5-89/016)	TEF June 1998 WHO	TEF June 2005 WHO
Dioxins			
2,3,7,8-TCDD	1.0	1.0	1.0
1,2,3,7,8-PeCDD	0.5	1.0	1.0
1,2,3,4,7,8-HxCDD	0.1	0.1	0.1
1,2,3,6,7,8-HxCDD	0.1	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.1	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.01	0.01
OCDD	0.001	0.0001	0.0003
Furans			
2,3,7,8-TCDF	0.1	0.1	0.1
1,2,3,7,8-PeCDF	0.05	0.05	0.03
2,3,4,7,8-PeCDF	0.5	0.5	0.3
1,2,3,4,7,8-HxCDF	0.1	0.1	0.1
1,2,3,6,7,8-HxCDF	0.1	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1	0.1
1,2,3,4,6,7,8-HpCDF	0.01	0.01	0.01
1,2,3,4,7,8,9-HpCDF	0.01	0.01	0.01
OCDF	0.001	0.0001	0.0003

TABLE 8 PCDDs/PCDFs HOLDING TIMES AND CONTAINERS
Method 8290 – TestAmerica

Method	Extraction Holding Time	**Containers** (no preservative other than 4°C)
8290	30 Days for soil and water	4 oz jar for soil; 2x 1 Liter amber for water