LABORATORY DOCUMENTATION GUIDANCE

MISSISSIPPI CANYON 252 (DEEPWATER HORIZON) NATURAL RESOURCE DAMAGE ASSESSMENT

Version 2.0

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Prepared for:

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VERSION 2.0 CHANGES FROM VERSION 1.0:

Page	Change
1	In Section 2.1 , replaced the second paragraph with following: If a work plan is not available or does not adequately describe the procedural steps for the requested laboratory work, a protocol or Standard Operating Procedure (SOP) must be prepared. The protocol may be similar to a "methods" section of a journal article, ensuring that adequate detail is provided to describe all steps performed. The protocol or SOP must be reviewed and available to all personnel performing the procedure, and must be submitted to the trustees so that this supporting procedural information can be reviewed and maintained along with the project data files. The requirement for written protocols or SOPs includes analytical, as well as auxiliary processes which may have an impact on quality and data usability. If analysis has already been conducted prior to the implementation of this guidance document, the protocol or SOP will reflect the actual operating procedures in place for the analysis.
1	In Section 2.1, at the beginning of the third paragraph, and in Item 1 of the list, added the words “protocol or”. And changed the word “should” to “must” in the first sentence. In Item 4 deleted repetitive language (‘clearly stated” and “in the SOP”).
2	Section 2.2, clarified language requiring laboratories to have contingencies in place to preserve samples and data.
2	Section 3.1 in the third paragraph, the next to last sentence the word “and” was added between the words ‘times’ and ‘the’.
2	Section 3.1 in third paragraph, in the last sentence the word “deviations” was replaced with “discrepancies”.
3	Section 4.1: In the last sentence the article “a” was replaced with “the” in the phrase “initials of person making the correction”.
5	Formatting updated to indicate the Summary section is Section 7.0.

1.0 INTRODUCTION

In support of the Natural Resource Damage Assessment (NRDA) for the Deepwater Horizon Oil Spill (DWHOS), an Analytical Quality Assurance Plan (AQAP, version 3.0, December 2011) and a Data Validation Plan (DV Plan, July 2010) have been developed to describe the analytical and quality assurance requirements for chemical analyses of oil spill related contaminants performed at laboratories identified in the AQAP. However, there are laboratories conducting other types of analyses outside the scope of the AQAP and DV Plan. This guide provides for analytical process and quality assurance documentation for laboratories performing such other analyses. Because there are many kinds of laboratories and analyses, this document is not tailored to specific sample types, analytical objectives, laboratory methods, QA/QC procedures, or validation criteria as are the AQAP and DV Plan. This guidance sets forth general recommendations and the ideas presented may be modified to fit individual situations.

2.0 WORK PLANS AND STANDARD OPERATING PROCEDURES (SOPS)

2.1 Generally, written work plan(s) which describe the overall study plan and the analytical needs should be provided to the laboratory by the associated Technical Working Group (TWG) or Principal Investigator (PI) requesting the laboratory work. The work plan should contain sufficient detail for the laboratory to be able to complete the requested analyses. Laboratory staff should be familiar with the plan as appropriate to their assigned tasks, and have access to a current copy as a reference. The laboratory should have a mechanism in place to provide the most current copy, including addenda, for all staff working on the analysis.

If a work plan is not available or does not adequately describe the procedural steps for the requested laboratory work, a protocol or Standard Operating Procedure (SOP) must be prepared. The protocol may be similar to a "methods" section of a journal article, ensuring that adequate detail is provided to describe all steps performed. The protocol or SOP must be reviewed and available to all personnel performing the procedure, and must be submitted to the trustees so that this supporting procedural information can be reviewed and maintained along with the project data files. The requirement for written protocols or SOPs includes analytical, as well as auxiliary processes which may have an impact on quality and data usability. If analysis has already been conducted prior to the implementation of this guidance document, the protocol or SOP will reflect the actual operating procedures in place for the analysis.

Protocols or SOPs must address the following information at a minimum:

1. The types or matrices of samples that can be processed using the procedure specified in the protocol or SOP;
2. Equipment and materials needed, including the quality grade of reagents specified;
3. Sufficient detail describing the analytical procedure to allow a knowledgeable person to repeat the procedure without relying on outside references;
4. Quality controls (i.e., actions that will assess accuracy and precision) with frequency and acceptance limits. Corrective actions for any quality control parameter that fails should also be included;
5. Reporting/detection limits, as appropriate; and
6. Method references, as appropriate.

Additional sections may be added to SOPs as needed and may include definitions, health and safety information, personnel qualifications and responsibilities, and data and records management requirements.

Additional information regarding SOPs can be found in Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, available at http://www.epa.gov/quality/qs-docs/g6-final.pdf. SOPs need not be in the format specified in the EPA document: the reference is provided for informational purposes. An example of an SOP is included at the end of this guidance document (**Attachment 1**).

2.2 Laboratories should have written contingencies in place for catastrophic failure of analytical and sample processing equipment, and sample storage refrigerators and freezers. Contingency planning should address failures due to mechanical and electrical causes as well as natural disasters, and should include specific actions and alternate storage options in order to preserve samples and data.

3.0 CUSTODY AND SAMPLE HANDLING

3.1 The integrity of each sample must be maintained through appropriate handling, preservation, and transportation techniques. Any indication that a sample has been subjected to tampering or physical alteration could disqualify it as evidence.

The sampler is responsible for the care and custody of the samples collected until they are transferred to the laboratory under chain of custody procedures. Situations where a sample is considered in "custody" include: it is in the lab's actual physical possession; the lab has stored it in a secured place (under lock) with restricted access; or the lab received the sample in a container secured with an official seal(s) such that the sample cannot be reached without breaking the seal(s).

Due to the evidentiary nature of samples collected during an injury assessment, possession must be traceable from the time the samples are collected until the data derived from the samples are introduced as evidence. The laboratory must continue to maintain Chain of Custody (COC) documentation showing continuous custody of samples beginning with sample collection and ending with sample archiving or disposal, i.e. cradle to grave. All samples must be retained by the laboratory, under COC documentation and in good condition, until a person legally authorized to direct such action authorizes either transfer of the samples to another facility or directs disposal of the samples. Chain of custody forms must accompany the samples at all times and the identification and presence or absence of all samples listed on the COC form must be verified by the receiving laboratory. Any COC discrepancies must be clearly documented at the time of sample receipt.

3.2 In addition, any preservation or storage conditions must be verified and documented by the person receiving the samples for the laboratory. Any discrepancies need to be resolved as quickly as possible and all resolutions must be documented. For example, if samples require a particular type of storage condition, such as refrigeration at 4°C ± 2°C, then the laboratory must retain documentation to show that this condition was satisfied both upon arrival at the laboratory

and throughout the period during which the sample is in the custody of the laboratory. This may require evidence of periodic temperature monitoring on affected cold-storage units and evidence that the thermometer used to check the temperature has been calibrated against a traceable source and that calibration has been routinely verified.

4.0 DATA AND DOCUMENTATION OF ANALYTICAL PROCESS

4.1 Complete data files must be maintained by the laboratories so that all results stated in the reports can be reproduced. These records, including any emails associated with the laboratory's work on the DWHOS must be exempt from any data retention procedures in place at the laboratory for periodic deletion. In addition, all samples and related materials relevant to the Deepwater Horizon oil spill must be retained indefinitely unless approval is given for their disposal in writing and by a person legally authorized to direct such action. The requirement that all material be retained indefinitely covers documents, e-mails, data and samples. A complete laboratory record will include documentation of the entire analytical process including auxiliary processes such as training, purchase of materials and equipment, data reduction and reporting, etc. Documentation must be complete, legible (in ink – no pencil allowed), and corrections to data must be proper (single line cross-out, initials of person making the correction, and date of correction plus a reason for the correction if it is not obvious).

4.2 As appropriate, laboratory procedures should address control of laboratory documents. The goals of document control are to:

- Ensure that all data and documents generated or obtained during the analysis will be accounted for when the investigation is completed; and
- Prevent premature or inadvertent disclosure of information.

Aspects of document control that should be assessed by the laboratory for inclusion in their procedures include:

- Version control for reports and other finalized documents (e.g., standard practice of adding a date at the end of each electronic file when it is saved);
- Document distribution and transmittal requirements (e.g., the Sample Confirmation file is transmitted electronically every Wednesday to Industrial Economics, Inc., etc.);
- Document inventory requirements (e.g., all versions of a document will be retained and back-up of files will occur electronically on a routine basis, emails associated with work on the DWHOS must be exempt from document retention protocols that might result in deletion);
- Procedures for corrections to documentation; and
- Document file organization (e.g., a special directory on each computer gathering data will be set-up and called DWHOS NRDA with sub-folders for specific jobs/tasks being performed).

Recordkeeping should include all of the steps in a process that can contribute to error or ambiguity and the entire process should be evaluated by the laboratory in considering the extent

of the documentation determined to be needed. Laboratories documenting information for each analysis, at a minimum, should include:

- Signature of the analyst;
- Date and time of analysis;
- All information used in calculations; and
- An indication of whether problems or anomalies occurred during analysis and what actions were taken, if any, to address the problems or anomalies.

When laboratory notebooks are used, personnel should enter data in a way that minimizes the ability to alter data after completing an analysis. Separate notebooks dedicated to DWHOS NRDA should be used for the purpose of documenting sample analyses. Further details on laboratory notebooks can be found at the following websites:

- http://www.iphandbook.org/handbook/ch08/p02/
- http://colinpurrington.com/tips/academic/labnotebooks

4.3 Supporting data (including electronic files) used to calculate results must be retained. In order to facilitate validation of data by a third party, the supporting materials should include information for all interim steps that contribute to result calculation, such as calibration and regression information, true values for standards, and quality control sample results with current, laboratory-derived control limits. Data and documentation must be archived by the laboratory in a manner that provides security, traceability and retrievability. Data must be archived indefinitely and authorization must be obtained from a person legally authorized to direct such action prior to destruction of any data or documents.

4.4 Integrity of electronic data must be maintained by ensuring that data are reliable and accurate through data verification (review) procedures, password-protection access, anti-virus protection, and documentation of data changes. Spreadsheets and other software developed in-house must be verified with documentation through manual calculations prior to use.

Protection against loss of electronic information or service must be ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, uninterruptible power supply (UPS), and maintaining older versions of software as revisions are implemented.

5.0 VERIFICATION AND VALIDATION

Laboratories and, if appropriate, the PI overseeing the analysis, need to complete a secondary review of their data to ensure proper reporting of results. This review process will vary based on the type of analysis being performed. The review of laboratory data should be documented. A convenient and efficient form of documentation is a checklist. The checklist is useful in providing consistency within the review process, and documentation of the review elements including errors found, corrective actions, reviewer(s), and date(s) of review.

Decisions about third party validation of data are the responsibility of the TWG. In order to

facilitate validation of data by a third party, the supporting materials should include information for all interim steps that contribute to result calculation, such as calibration and regression information, true values for standards, and quality control sample results with current, method- or laboratory-derived control limits. Data and documentation must be archived by the laboratory in a manner that provides security, traceability, and retrievability.

6.0 TRAINING RECORDS

At a minimum, laboratories should maintain current curriculum vitae for personnel performing analyses. If possible, training records and demonstration of capability/competence should also be maintained in the training files. For analyses that do not incorporate spiked samples as a form of quality control, an analyst's results from a performance evaluation type of sample may be used for training documentation. An example of a training record is included at the end of this guidance document (**Attachment 2**).

7.0 SUMMARY

There's a saying, 'If you didn't document it, then it didn't happen'. With that in mind, laboratory records should be able to answer the following questions:

- What did you do?
- Who did it when?
- How do you know the reliability of the result obtained and reported?

The purpose of good documentation practices is to clearly document what actions took place during the analytical process so that anyone, including an auditor, has documented evidence that you did exactly what you said you did. Keep in mind that the DWHOS analyses are being conducted in part for potential use in litigation, so the data need to be clearly documented for legal and preservation purposes. In addition, the intermediate steps in an analysis must be clearly documented and all materials and written communications associated with the analysis must be retained indefinitely until authorized personnel instruct the laboratory otherwise.

Attachment 1: Example of SOP for Total Suspended Solids, SM 2540D

LABORATORY NAME AND ADDRESS:

1.0 Scope and Application

1.1 This SOP is applicable to the determination of total suspended solids (TSS) using a gravimetric technique and is applicable to drinking, surface and saline waters, and domestic and industrial wastes.

1.2 The methods cover a practical range of 4 mg/L - 20,000 mg/L. As a practical matter, the final residue weight should be limited to about 200 mg.

2.0 Equipment and Supplies

- Analytical balance capable of weighing to 0.0001 g. The balance calibration is checked each day of use with three Class 1 weights that bracket the range of use and recorded on the TSS benchsheet. The verification must be within 0.1% or 0.5 mg, whichever is larger, of the certified value of the standard mass measured.
- Vacuum filtration apparatus with vacuum pump equipped with moisture trap.
- Glass fiber filter disks, 47 mm, without organic binder (Gelman Type A/E) or equivalent.
- Aluminum weighing dishes large enough to hold a 47 mm filter
- Graduated cylinders, assorted sizes
- Drying oven set at 103-105°C
- Forceps or tongs
- Desiccators providing sufficient space for storage of samples in process and containing a color indicator of moisture concentration or an instrumental indicator.

3.0 Reagents and Standards

3.1 De-ionized water.

3.2 Laboratory Control Sample (LCS) solution (100 mg/L TSS):
Place 1.0 g of sodium chloride and 0.1 g Celite into a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months. Alternatively, a commercially available LCS solution may be used. True value is 100 mg/L.

4.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Method	Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
TSS	Waters	HDPE	100 mLs	Cool 4 $\pm$ 2°C	7 Days	40 CFR Part 136.3

5.0 Quality Control

5.1 Batch Definition

A group of samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period.

5.2 Control Samples

Quality Controls	Frequency	Control Limit	Corrective Action
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit	Reanalysis
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	80-120% Recovery	Reanalysis
Duplicate Sample (DUP)	1 in 10 or fewer samples	within 5% of avg weight	Reanalysis if within holding time and sufficient sample is remaining

6.0 Procedure

- All samples should be maintained under Chain of Custody.
- Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filters holders must be done in a manner to ensure connection with the proper sample.
- Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the benchsheet.
- If samples are visibly oily, this should be noted on the benchsheet.
- If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the benchsheet and reporting limits must be adjusted appropriately.

6.1 Calibration

6.1.1 Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily or prior to sample analysis by checking the balance calibration. Analytical balance calibration must be performed daily (every 24 hours).

6.1.2 Oven temperature must be checked daily and recorded either on the benchsheet or in an oven temperature logbook.

6.2 Sample Preparation

6.2.1 Place the glass fiber filter discs, one at a time, on the membrane filter apparatus with wrinkled surface up.

6.2.2 While vacuum is applied, wash the disc with three successive (approximately) 20 mL volumes of distilled water.

6.2.3 Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.

6.2.4 Remove filter from membrane filter apparatus and place in a labeled, aluminum weighing dish and dry in an oven at 103-105 °C for one hour.

6.2.5 Remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

6.2.6 Weigh the cooled filter to the nearest 0.1 mg using an analytical balance. Record the weight and the dish identification number on the benchsheet.

6.3 Sample Analysis

6.3.1 Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water and begin suction.

Note: Handle the filters only with forceps.

6.3.2 Selection of Sample Volume

- For a 4.7 cm diameter filter, filter 100 - 500 mL of sample sufficient to yield between 10 mg and 200 mg of dried residue. If the weight of the captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue.

- If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

Note: If the sample appears high in TSS, start with a smaller sample volume.

6.3.3 Shake the sample vigorously and quickly aliquot the sample. It is important to pour out the sample immediately after shaking so that the solids do not have time to settle. A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Record the volume of sample filtered (to the nearest mL) on the benchsheet.

6.3.4 With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

6.3.5 Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

6.3.6 Carefully remove the filter from the filter support and transfer to an aluminum weighing dish. If the filter is torn or damaged during this process, the sample must be reanalyzed. Take care to keep the filter face-up during the transfer so that the residue does not fall off.

6.3.7 Dry the filter for at least one hour at 103-105 °C.

6.3.8 Use heat resistant gloves to remove the tray of dishes from the oven. Place in a desiccator and cool to room temperature.

6.3.9 Weigh the filters (to the nearest 0.1 mg), and record the weight on the benchsheet.

6.3.10 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until the weight change is less than 4% of the previous weight or weight difference is less than 0.5 mg, whichever is less. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

6.3.11 Calculate the results using the formula given in Section 7.3.1. Use the final weight achieved for calculating TSS.

7.0 Calculations / Data Reduction

- If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.
- If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

7.1 Accuracy

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

7.2 Precision (RPD)

$$\text{Sample Duplicate (DUP)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

7.3 Concentration

7.3.1 Total Suspended Solids = $\text{Total Suspended Solids, mg/L} = \dfrac{(A - B) \times 1000}{C}$

Where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

8.0 Method Performance

Method detection limits (MDL) are not determined for this procedure.

9.0 References / Cross-References

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

Attachment 2: Example Laboratory Training Record

Lab Name Here

INDIVIDUAL TRAINING RECORD

NAME: _____

ATTACH EVIDENCE OF COMPLETION OR SUPPLEMENTARY INFORMATION AS REQUIRED.

Procedure Certified:	Date Certified	Trainer or QA Initials
☐_____ Procedure or Work Instruction Name/No.:_____ Date of Observation or Review :_____ Observer/Trainer Initials: _____ Date of Observation or Review :_____ Observer/Trainer Initials: _____		
☐_____ Procedure or Work Instruction Name/No.:_____ Date of Observation or Review :_____ Observer/Trainer Initials: _____ Date of Observation or Review :_____ Observer/Trainer Initials: _____		
Other Instruments/Method		
☐_____ Procedure or Work Instruction Name/No.:_____ Date of Observation or Review :_____ Observer/Trainer Initials: _____ Date of Observation or Review :_____ Observer/Trainer Initials: _____		
☐_____ Procedure or Work Instruction Name/No.:_____ Date of Observation or Review :_____ Observer/Trainer Initials: _____ Date of Observation or Review :_____ Observer/Trainer Initials: _____		
Project Specific Training		
☐_____ Procedure or Work Instruction Name/No.:_____ Date of Observation or Review :_____ Observer/Trainer Initials: _____ Date of Observation or Review :_____ Observer/Trainer Initials: _____		