STANDARD OPERATING PROCEDURE

SIX

QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

Modified from

U.S. Environmental Protection Agency Environmental Response Team

Response Engineering and Analytical Contract

Standard Operating Procedures

Quality Assurance/Quality Control Samples

SOP 2005, Rev. 1.0, 2/7/92

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe types of Quality Assurance/Quality Control (QA/QC) samples that are collected in the field, or prepared for or by the laboratory for soil and water matrices.

The following are standard operating procedures which, under certain circumstances, may need to be changed or varied. However, deviation from the following procedures should only take place as a last resort. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

The goal of including QA/QC samples with any sampling or analytical event is to be able to identify, measure, and control the sources of error that may be introduced from the time of sample-container preparation through sample analysis. Quality assurance samples are used as an assessment tool to determine if environmental data meet the quality criteria established for a specific application. Quality control samples are generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. Several types of samples may be used for establishing QA/QC. Any one sample may serve to establish QA, QC, or both. Sample types are discussed under the Procedure section of this SOP.

Several terms are used to quantify QA/QC. Accuracy is defined as the closeness or agreement between an observed value and an accepted reference value. Bias is defined as the deviation of a measured value from a reference value or a known spiked amount, and is determined by calculating percent recovery. Precision is a measure of the closeness of agreement among individual measurements. Precision is determined by calculating the relative standard deviation or the coefficient of variation for at least eight (8) matrix-spike samples.

2.0 POTENTIAL PROBLEMS

Quality assurance/quality control samples are collected and analyzed in addition to environmental samples to assist in identifying the origin of both field and laboratory contamination. In order to provide useful information, QA/QC samples must be taken, prepared and analyzed in the same manner as the environmental samples. Failure to do so may result in invalid QA/QC interpretations.

3.0 SAMPLING EQUIPMENT, CONTAINERS, PRESERVATION, AND STORAGE

The site specific equipment/apparatus required to collect QA/QC samples is the same as the equipment/apparatus required to collect the environmental samples. Refer to the relevant SOPs for sampling techniques to obtain lists of the equipment/apparatus required for sampling. The amount of sample to be collected, the proper sample container, chemical preservation and storage requirements are discussed in the sample storage, preservation, and handling SOP.

4.0 PROCEDURE

Quality assurance/quality control samples for soil and water matrices are discussed below. Each type of sample is defined and a preparation procedure is outlined. In addition, the minimum frequency of collection of these QA/QC samples is discussed.

4.1 Soil Quality Assurance/Quality Control Samples

4.1.1 Field replicates

Field replicates are field samples obtained from one location. They are homogenized with thorough hand mixing and divided into separate containers. They are treated as separate samples throughout the remaining sample handling and analytical processes.

These samples are used to assess total error (precision) associated with sample heterogeneity, sample methodology, and analytical procedures. This procedure is useful in determining total (sampling and analytical) error because it evaluates sample collection, sample preparation, and analytical procedures.

Field replicates may be especially important when determining precision for critical samples with contamination concentrations near or above the action level (action level refers to the minimum concentration necessary to require some type of remediation, monitoring, or enforcement).

A minimum of eight replicate samples are required in order for valid statistical analysis to be performed. Field replicates may be collected on a site specific basis and do not have to be collected at all sites investigated.

4.1.2 Collocated samples

Collocated samples are collected adjacent to the routine field sample to determine the variability of the soil and contaminant(s) within a small area at the site. Typically, collocated samples are collected about one-half to three (3) feet away from the routine field sample location.

Analytical results from collocated samples can be used to assess variation in the immediate sampling area. Due to the non-homogeneous nature of soil, collocated soil samples should not be used to assess variability across an entire site. Applicability and frequency of collocated samples should be determined on a site-specific basis.

4.1.3 Background samples

Background samples are collected from area(s), either onsite or off-site where there is little or no chance of contamination. Background samples are collected in an attempt to determine the natural composition of the soil (especially important in areas with high concentrations of naturally occurring metals) and are considered "clean" samples. They provide a basis for comparison of contaminant concentration levels with samples collected onsite. At least one background soil sample should be collected. More are warranted when site specific factors such as natural variability of local soil, multiple onsite contaminant source areas, or off-site facilities potentially contributing to soil contamination exist. Background samples may be collected in order to evaluate potential error associated with sampling design, sampling methodology, and analytical procedures.

Background samples may be used to determine bias and precision if at least eight replicates are spiked with the analyte of interest at a concentration equal to the action level, and then analyzed.

4.1.4 Field blanks

Field blanks are prepared in the field by filling the appropriate sample container with certified clean sand or soil. The blanks are then submitted to the laboratory for analysis. A field blank is primarily used to evaluate contamination error associated with field operations and shipping but may also be used to evaluate contamination error associated with laboratory procedures. Submit field blanks at a rate of one per day per site.

4.1.5 Trip blanks

Trip blanks are only required if volatile organics are a concern. They are prepared in the laboratory, taken to the field, and then brought back to the laboratory with the environmental samples and analyzed. Trip blanks consist of certified clean sand or soil handled, transported, and analyzed in the same manner as the other volatile-organic containers and samples acquired. Trip blanks are used to evaluate contamination error associated with sample handling and shipment or laboratory handling and analysis. The minimum frequency of trip blanks is one per container used to transport volatile-organic samples.

4.1.6 Performance-Evaluation samples

Performance-evaluation (PE) samples evaluate the overall bias of the analytical laboratory and detect any error in the analytical method used. These samples are usually prepared by a third party, using a quantity of analyte(s) which is known to the preparer but unknown to the laboratory. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. Laboratory procedural error is evaluated by the percentage of analyte identified (percent recovery) in the PE sample. When analyzed, the minimum frequency of PE samples is one per analyte of interest.

4.1.7 Matrix-Spike samples

Matrix spike and matrix spike duplicate samples (MS/MSDs) are environmental samples that are spiked in the laboratory or in the field with a known concentration of a target analyte(s) to verify percent recoveries. Matrix spike and matrix spike duplicate samples are primarily used to check sample matrix interferences. They can also be used to monitor laboratory performance. However, a data set of at least three or more results is necessary to statistically distinguish between laboratory performance and matrix interference. The minimum frequency of MS/MSDs should be 10 percent of the total number of samples being analyzed for the target analyte(s).

Matrix spike and matrix spike duplicate samples are also used to evaluate error due to laboratory bias and precision. One MS/MSD pair per target analyte should be analyzed and the average percent recovery should be calculated to assess bias. To assess precision, at least eight matrix spike replicates from the same sample should be analyzed and the standard deviation and coefficient of variation should be determined.

4.1.8 Split samples

Split samples are field samples obtained from one location. They are homogenized with thorough hand mixing and divided into separate containers. They are treated the same as field-replicate samples except that they are sent to an alternate laboratory for analysis. The purpose of the split sample is to use the alternate laboratory to verify the accuracy of the primary laboratory. The number of split samples sent to the alternate laboratory should be at least two per site.

4.2 Aqueous Quality Assurance/Quality Control Samples

4.2.1 Field replicates

Field replicates are field samples obtained from one location. They are divided into separate containers. They are treated as separate samples throughout the remaining sample handling and analytical processes.

These samples are used to assess total error (precision) associated with sample heterogeneity, sample methodology, and analytical procedures. This procedure is useful in determining total (sampling and analytical) error because it evaluates sample collection, sample preparation, and analytical procedures.

Field replicates may be especially important when determining precision for critical samples with contamination concentrations near or above the action level (action level refers to the minimum concentration necessary to require some type of remediation, monitoring, or enforcement).

A minimum of eight (8) replicate samples is required in order for valid statistical analysis to be performed. Field replicates may be collected on a site-specific basis and do not have to be collected at all sites investigated.

4.2.2 Background samples

Background samples are collected from area(s), either onsite or off-site where there is little or no chance of contamination. Background samples are collected in an attempt to determine the natural composition of the aqueous matrix and are considered "clean" samples. They provide a basis for comparison of contaminant concentration levels with samples collected onsite. At least one background sample should be collected; however, more are warranted when site specific factors such as multiple onsite contaminant source areas, or off-site facilities potentially contributing to contamination exist. Background samples may be collected in order to evaluate potential error associated with sampling design, sampling methodology, and analytical procedures.

Background samples may be used to determine bias and precision if at least eight replicates are spiked with the analyte of interest at a concentration equal to the action level and then analyzed.

4.2.3 Rinsate blanks

Rinsate blanks are samples obtained by running distilled/deionized water over decontaminated sampling equipment to test for residual contamination. The blank water is collected in sample containers for handling, shipment, and analysis. These samples are treated identical to the samples collected that day. A rinsate blank is used to assess cross contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank per type of sampling device per day.

4.2.4 Field blanks

Field blanks are prepared in the field by filling the appropriate sample container with distilled/deionized water and are then submitted to the laboratory for analysis. A field blank is primarily used to evaluate contamination error associated with field operations and shipping but

may also be used to evaluate contamination error associated with laboratory procedures. Submit field blanks at a rate of one per day per site.

4.2.5 Trip blanks

Trip blanks are only required if volatile organics are a concern. They are prepared in the laboratory, taken to the field, and then brought back to the laboratory with the environmental samples and analyzed. Trip blanks consist of distilled/deionized water handled, transported, and analyzed in the same manner as the other volatile-organic containers and samples acquired. Trip blanks are used to evaluate contamination error associated with sample handling and shipment or laboratory handling and analysis. The minimum frequency of trip blanks is one per container used to transport volatile-organic samples.

4.2.6 Performance-Evaluation samples

Performance-evaluation (PE) samples evaluate the overall bias of the analytical laboratory and detect any error in the analytical method used. These samples are usually prepared by a third party, using a quantity of analyte(s), which is known to the preparer but unknown to the laboratory. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. Laboratory procedural error is evaluated by the percentage of analyte identified (percent recovery) in the PE sample. When analyzed, the minimum frequency of PE samples is one per analyte of interest.

4.2.7 Matrix-Spike samples

Matrix-spike and matrix spike duplicate samples (MS/MSDs) are environmental samples that are spiked in the laboratory or in the field with a known concentration of a target analyte(s) to verify percent recoveries. Matrix-spike and matrix spike duplicate samples are primarily used to check sample matrix interferences. They can also be used to monitor laboratory performance. However, a data set of at least three or more results is necessary to statistically distinguish between laboratory performance and matrix interference. The minimum frequency of MS/MSDs should be 10 percent of the total number of samples being analyzed for the target analyte(s).

Matrix-spike and matrix spike duplicate samples are also used to evaluate error due to laboratory bias and precision. One MS/MSD pair per target analyte should be analyzed and the average percent recovery should be calculated to assess bias. To assess precision, at least eight matrix spike replicates from the same sample should be analyzed and the standard deviation and coefficient of variation should be determined.

4.2.8 Split samples

Split samples are field samples obtained from one location and divided into separate containers. They are treated the same as field-replicate samples except that they are sent to an alternate laboratory for analysis. The purpose of the split sample is to use the alternate laboratory to verify the accuracy of the primary laboratory. The number of split samples sent to the alternate laboratory should be at least two per site.

5.0 DATA VALIDATION

All data must be documented on field-data sheets or within site logbooks and on chain of custody forms. Results of the QA/QC samples will be evaluated by laboratory personnel and the project leader. This information will be utilized to qualify the environmental-sample results accordingly with the project's data-quality objectives.

6.0 ADDITIONAL INFORMATION SOURCES

U.S. Environmental Protection Agency Office of Emergency and Remedial Response, 1990, Quality assurance/quality control guidance for removal activities: EPA/540/G-90/004, Sampling QA/QC Plan and Data Validation Procedures Interim Final, April, 1990.

_____1991, Removal program representative sampling guidance; Volume 1 - Soil: OWSER Directive 9360.4-10 Interim Final, June