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SUPERCEDES: SOP #2005; Revision 1.1; 04/19/93; U.S. EPA Contract 68-03-3482.

1.0 SCOPE AND APPLICATION

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

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or other procedure limitations. 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 U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

QA samples are used as an assessment tool to determine if environmental data meet the quality criteria established for a specific application. QC 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. 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 bottle preparation through analysis.

Analytical results for these samples can be used to assess accuracy as well as cross contamination. Accuracy refers to the correctness of the concentration value and the qualitative certainty that the analyte is present. It is a combination of both bias (systematic error) and precision (random error). Bias is defined as the deviation of a measured value from a reference value or 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 coefficient of variation calculations.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

The amount of sample to be collected, and the proper sample container type (i.e., glass, plastic), chemical preservation, and storage requirements are dependent on the matrix being sampled and the parameter(s) of interest, and are discussed in ERT/REAC SOP #2003, Sample Storage, Preservation, and Handling, for the soil and water matrices. Sample preservation, containers, handling, and storage for air and waste samples are discussed in the specific SOPs for air and waste sampling techniques.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

QA/QC 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 prepared and analyzed appropriately.

5.0 EQUIPMENT/APPARATUS

With the exception of some types of blank and performance evaluation samples, the equipment/apparatus required to collect QA/QC samples is the same as the equipment/apparatus required to collect the environmental samples. This is determined on a site specific basis. Due to the wide variety of sampling

equipment available, refer to the specific SOPs for sampling techniques which include lists of the equipment/apparatus required for sampling. Sampling equipment/apparatus are generally not required for field, trip, or lot blanks or performance evaluation samples.

6.0 REAGENTS

Reagents may be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed and are summarized in ERT/REAC SOP #2003, Sample Storage, Preservation, and Handling. Decontamination solutions are specified in ERT/REAC SOP #2006, Sampling Equipment Decontamination.

7.0 PROCEDURE

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

7.1 Soil QA/QC Samples

7.1.1 Field Replicates

Field replicates are field samples obtained from one location, homogenized, 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 error (precision) associated with sample heterogeneity, sampling methodology and analytical procedures. Field replicates may be collected on a site-specific basis and may not be collected at all sites investigated.

Field replicates may be used when determining total error (precision) for critical samples with contamination concentrations at or near the action level. This procedure is useful in determining total (sampling and analytical) error because it evaluates sample collection, sample preparation, and analytical procedures. If error is to be determined, a minimum of eight replicate samples from a single sample location is required in order for a valid statistical analysis to be performed.

NOTE: The terms "field duplicate" or "duplicate sample" have been replaced by the term "field replicate".

7.1.2 Collocated Samples

Collocated samples are collected adjacent to the routine field sample to determine variability of the soil and contaminant(s) at the site within a small area. Typically, collocated samples are collected about one-half to three feet away from the routine field sample location. Analytical results from collocated samples can be used to assess site variation, but only in the immediate sampling area. Due to the non-homogenous nature of soil at sites, collocated samples should not be used to assess variability across a site and are not recommended for assessing error. Applicability and frequency of collocated samples should be determined on a site-specific basis.

7.1.3 Background Samples

Background samples are collected from area(s) either on or off site where there are little or no contaminants. 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 on site. At least one background soil sample should be collected; however, more may be warranted when site-specific factors such as natural variability of local soil, multiple on-site contaminant source areas, or off-site facilities potentially contributing to soil contamination exist. Background samples may be collected for all QA objectives, 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 spike with the analyte of interest at a concentration equal to the action level and then analyzed.

7.1.4 Rinsate Blanks

For the soil matrix, rinsate blanks are not required because the aqueous rinse does not simulate the cross-contamination mechanism that would occur.

7.1.5 Field Blanks

Field blanks are prepared in the field by filling the appropriate sample container with certified clean sand or soil 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 to meet QA2 and QA3 objectives.

7.1.6 Trip Blanks

Trip blanks are only required for volatile organics analysis and are prepared prior to going into the field. Trip blanks consist of certified clean sand or soil and are handled, transported, and analyzed in the same manner as the other volatile organic samples collected that day. Trip blanks are used to evaluate contamination error associated with sample handling and shipment, or laboratory handling and analysis. Utilize trip blanks to meet QA2 and QA3 objectives for volatile organic analyses only. The minimum frequency of trip blanks is one per container used to transport volatile organic samples.

7.1.7 Performance Evaluation Samples

Performance evaluation (PE) samples evaluate the overall accuracy of the analytical laboratory and detect any bias 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. These samples always undergo some type of certification analysis. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. Laboratory accuracy is evaluated by comparing the percentage of analyte identified in the PE sample (percent recovery) with the analytical results of the site samples. Even though they are not available for all analytes, PE samples are required to achieve QA3 objectives. Where PE samples are unavailable for an analyte of interest, QA2 is the highest QA objective achievable. When analyzed, the minimum frequency of PE samples is one per analyte of interest per matrix.

7.1.8 Matrix Spike Samples

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

MS/MSDs are also used to evaluate error due to laboratory bias and precision. One MS/MSD pair 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. Bias and precision calculations are optional for QA2 objectives and required to meet QA3 objectives.

7.2 Aqueous QA/QC Samples

7.2.1 Field Replicates

Field replicates are field samples obtained from one location 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 error (precision) associated with sample heterogeneity, sampling methodology and analytical procedures. Field replicates may be collected on a site-specific basis and may not be collected at all sites investigated.

Field replicates may be used when determining total error (precision) for critical samples with contamination concentrations at or near the action level. This procedure is useful in determining total (sampling and analytical) error because it evaluates sample collection, sample preparation, and analytical procedures. If error is to be determined, a minimum of eight replicate samples from a single sample location is required in order for a valid statistical analysis to be performed.

NOTE: The terms "field duplicate" or "duplicate samples" have been replaced by the term "field replicate".

7.2.2 Background Samples

Background samples are collected from area(s) either on or off site where there are little or no contaminants. Background samples 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 on site. At least one background sample should be collected; however, more may be warranted when site-specific factors such as multiple on-site contaminant source areas, or off-site facilities potentially contributing to contamination exist. Background samples may be collected for all QA objectives, 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.

7.2.3 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 in the same manner as the samples collected that day. A rinsate blank is used to assess cross-contamination brought about by improper decontamination procedures. Where non-dedicated sampling equipment is utilized, collect one rinsate blank per type of sampling device per day to meet QA2 and QA3 objectives.

7.2.4 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 to meet QA2 and QA3 objectives.

7.2.5 Trip Blanks

Trip blanks are only required for volatile organics analysis and are prepared prior to going into the field. Trip blanks consist of distilled/deionized water and are handled, transported, and analyzed in the same manner as the other volatile organic samples collected that day. Trip blanks are used to evaluate contamination error associated with sample handling and transport, or laboratory handling and analysis. Utilize trip blanks to meet QA2 and QA3 objectives for volatile organic analyses only. The minimum frequency of trip blanks is one per container used to transport volatile organic samples.

7.2.6 Performance Evaluation Samples

Performance evaluation (PE) samples evaluate the overall accuracy of the analytical laboratory and detect any bias 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. These samples always undergo some type of certification analysis. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. Laboratory accuracy is evaluated by comparing the percentage of analyte identified in the PE sample (percent recovery) with the analytical results of the site samples. Even though they are not available for all analytes, PE samples are required to achieve QA3 objectives. Where PE samples are unavailable for an analyte of interest, QA2 is the highest QA objective achievable. When analyzed, the minimum frequency of PE samples is one per analyte of interest per matrix.

7.2.7 Matrix Spike Samples

MS/MSDs are environmental samples that are spiked in the laboratory with a known concentration of a target analyte(s) to verify percent recoveries. MS/MSDs are primarily used to check sample matrix interferences. They can also be used to monitor laboratory performance. However, a dataset of at least three or more results is necessary to distinguish between laboratory performance and matrix interference. For ERT/REAC sampling events, the minimum frequency of MS/MSDs is 10% of the total number of samples being analyzed for the target analyte(s).

MS/MSDs are also used to evaluate error due to laboratory bias and precision. One MS/MSD pair should be analyzed and the average percent recovery should be calculated to assess bias. To asses 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. Bias and precision calculations are optional for QA2 objectives and required to meet QA3 objectives.

7.3 Air QA/QC Samples

7.3.1 Collocated Samples

Collocated samples are collected by placing two identical samplers next to each other and, either: (1) air is drawn from one source and split with a manifold; or (2) two pumps are set adjacent to each other and each collect a sample at the same flow rate. Depending upon the methods used to collect and analyze the samples, collocated samples can determine the variation due to both sampling error and precision in the analyses (e.g., using thermally desorbed adsorbent tubes), or to isolate the variation due to sampling error only (e.g., using solvent-extracted tubes and Summa canisters). The minimum frequency of collocated samples is 5% or one per sampling event for all QA objectives.

7.3.2 Field Blanks

Field blanks are samples that undergo the full handling and shipping process of an actual sample. Field blanks are designed to detect potential sample contamination that may occur during field operations or during shipment. The field blank is opened with the other sampling media, resealed and carried through the sampling process. The field blank must be associated with an actual sampling period. Submit field blanks at a rate of

5% of the total samples or a minimum of one per sampling event to meet QA2 and QA3 objectives.

7.3.3 Trip Blanks

Trip blanks detect whether samples are contaminated during shipping. It is typically used in conjunction with field blanks to isolate sources of sample contamination already noted in previous field blanks. The trip blank is prepared and added to the site samples after sampling has been completed, just prior to shipping samples for analysis. If the absorbent tubes were sealed from the manufacturer, their seals should be broken at this point. For absorbent tubes that have been recycled and resealed by the laboratory, there is no need to break these temporary seals prior to shipping. Canister trip blanks are evacuated containers that are shipped to and from the site with the canisters used for air sampling. A trip blank for an impinger-based sampling method consists of an aliquot of impinger reagent that is shipped back to the laboratory with the samples. Submit trip blanks at a rate of 5% of the total samples or a minimum of one per sampling event to meet QA2 and QA3 objectives.

7.3.4 Lot Blanks

A lot blank detects contamination producing false positive results strictly due to the sampling medium itself. It consists of a sample collector from the same lot as the sample collectors used during a particular day or time period. It comes from the manufacturer or laboratory with the seal intact. The lot blank is included with the samples when they are delivered to the laboratory. Whenever a set of canisters is cleaned by the laboratory for reuse, the previously most contaminated canister should be re-analyzed as a lot blank at least 24 hours later, in order to check the cleanliness of that lot of "cleaned" canisters. Whenever a new sampler system (e.g., Anderson stainless steel bellows pump) is initially received from the manufacturer or from a laboratory, a lot blank should be pulled off the system using humidified zero air or humidified nitrogen. In a similar manner, whenever a sampler system is cleaned, at a minimum, the sampler(s) that had generated the most contaminated canister sample(s) in the previous batch should be checked with humidified zero air. Submit lot blanks at a rate of 10% of the total samples or a minimum of one per sampling event per lot to meet QA1, QA2, and QA3 objectives.

7.3.5 Breakthrough Sample

Breakthrough samples detect false negative results and significant negative biases in the data. These problems can arise when compounds elute from the sampling media before the sampling run is completed. The two types of breakthrough samples are serial media samples and spiked media samples. To collect a serial media sample, a sampling train is set up with a primary sampling device and backed by a secondary sampling device. A spiked media breakthrough sample is obtained by pulling air through a sampling train that was either spiked in the field with a standard solution or was spiked in the laboratory prior to being shipped into the field. The spiked media breakthrough sample is always collected next to and concurrent with an upwind/background sample.

The breakthrough sample typically is used to determine whether the first sampling device has retained all of the compounds of concern. It should be collected in the first batch of samples. When mixed-bed adsorbent tubes are being used, serial media samples are not recommended. Instead, spike medium samples or distributed volume samples should be collected. Breakthrough samples are recommended to be collected to meet QA2 and QA3 objectives; however, the rate of collection is method dependent.

7.3.6 Performance Evaluation Sample

A PE sample evaluates the overall accuracy of the analytical laboratory and detects any bias in the analytical method being used. The PE sample contains a quantity of analyte(s) which is known to the sampling team but unknown to the laboratory. It is usually prepared by a third party and always undergoes some type of certification analysis. The analyte(s) used to prepare the PE sample is the same as the analyte(s) of concern. The laboratory's accuracy is evaluated by comparing the percentage of analyte identified in the PE sample with the analytical results of the site samples. PE samples are required to achieve QA3 objectives. Where they are unavailable for the analyte(s) of interest, QA2 is the highest QA objective achievable. When analyzed, the minimum frequency of PE samples is one per analyte of interest per matrix.

7.3.7 Blind Spike

A blind spike is a rarely used proficiency sample that is prepared and sent "blind" to a laboratory for the same analyses as the other samples. A blind spike is used when: (1) the desired frequency of check samples for the laboratory exceeds the number of available PE samples; (2) the background matrix of the PE does not truly reflect the background matrix of the samples (e.g., high summer-time humidity or the exhaust from soil vapor extraction or methane gas collection systems); or (3) many or all of the compounds of concern are not readily available in a PE sample. In the latter case, because of uncertainties in the stability and half-lives of "new" compounds in or on the sample media, the preparing laboratory must both certify the blind spikes which will be shipped to the field, and archive a few spike samples for re-certification analyses in the same time period as the actual sample analyses. A blind spike should be prepared by an individual who is proficient in its preparation. If used instead of PE samples, blind spikes are required to achieve QA3 objectives, and are optional for QA2 objectives. When analyzed, the minimum frequency of blind spikes is one per parameter.

Caution: Due to the large potential for errors, the difficulty of calculating the amount of spike needed, and the distribution of the spike compound throughout the sample, it is not recommended that blind spikes be used to evaluate labs. If used, the preparing laboratory must take all precautions to ensure accuracy and to reanalyze samples should there be any discrepancies.

7.4 Laboratory QA/QC Samples

7.4.1 Reagent/Method Blank

A reagent/method blank is a sample of the reagent used in sample analyses. Unlike field and trip blanks, a reagent/method blank is prepared in the laboratory and is designed to detect contamination that could arise from the reagents and laboratory equipment used in the analysis. This would include the reagents used in preparing impinger solutions and the reagents used in the extraction and cleanup of solvent extracted adsorbent media. Reagent/method blanks should be analyzed at a rate of one per sample batch per matrix.

7.4.2 Surrogate Spike

A surrogate spike is designed to detect potential quantitative errors in the actual analyses of each sample. The surrogate compounds, which are usually non-target compounds that elute throughout the analyses, are typically spiked into each sample prior to sample preparation. Surrogate spikes are also used to evaluate the method efficiency.

7.4.3 Matrix Spike (Air Matrix Only)

A matrix spike is designed to test the ability of the method to detect known concentrations of the target compounds. As a laboratory-prepared sample, it contains known concentrations of the target compounds which are spiked into a sample prior to analysis. The matrix spike results are used to verify retention times and percent recoveries in the extraction procedure and to determine the degree to which matrix interferences will affect the overall identification and quantification of the target compounds.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The following general QA procedures apply when preparing QC samples:

1. All data must be documented on Field Data Sheets or within site logbooks and on Chain of Custody forms.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

Results of the QA/QC samples will be evaluated for contamination. This information will be utilized to qualify the environmental samples results accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures.

12.0 REFERENCES

U.S. EPA Office of Emergency and Remedial Response. April, 1990. Quality Assurance/Quality Control Guidance for Removal Activities: Sampling QA/QC Plan and Data Validation Procedures. Interim Final. EPA/540/G-90/004.

U.S. EPA. Office of Emergency and Remedial Response. June, 1991. Removal Program Representative Sampling Guidance. Volume 1 - Soil. Interim Final. OWSER Directive 9360.4-10.

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