LABORATORY QC SAMPLES (WATER, SOIL)

1.0 PURPOSE

This section sets forth the standard operating procedure (SOP) for identifying the number and type of laboratory Quality Control (QC) samples that will be. Laboratory QC analyses serve as a check on the precision and accuracy of analytical methods and instrumentation and potential contamination that may occur during laboratory sample preparation and analyses. Laboratory QC analyses include but are not limited to blank, duplicate, surrogate, blank spike, laboratory control sample, and matrix spike/ matrix spike duplicate analyses. These laboratory QC analyses are discussed in general below.

2.0 SCOPE

This procedure applies to all laboratory analytical activities conducted. This procedure shall serve as professional guidance for AMEC personnel. It is not intended to obviate the need for professional judgment that may arise in unforeseen circumstances. Deviations from this procedure in the planning or the execution of activities must be approved by the Project Manager.

3.0 DEFINITIONS

3.1 PRECISION

A measure of the agreement, i.e., reproducibility, among individual measurements of the same property, under prescribed similar conditions. Precision is measured by relative percent difference (RPD).

3.2 RELATIVE PERCENT DIFFERENCE (RPD)

A measure of precision, which is based on the mean of two values from related analyses and is reported as an absolute value. It is calculated using the following formula:

$$RPD = | S-D | / [(S + D)/2] \times 100\%$$

where S = original sample result

D = duplicate sample result

3.3 Laboratory Blank

A clean sample provided by the laboratory that is analyzed to monitor contamination during laboratory analysis. Also called a method blank, reagent blank, or preparation blank. The same matrix must be used for the laboratory blank as for site samples.

3.4 Duplicate

A duplicate is a split sample that is analyzed to determine laboratory precision for a particular matrix.

3.5 Matrix Spike

A quality control sample where a known amount of analyte is added to a site sample, then analyzed, for the purpose of determining efficiency of recovery for that type of matrix.

3.6 Blank Spikes and Laboratory Control Samples

Blank spikes and laboratory control samples consist of reagent water or clean soil/sand that has been spiked with known amounts of specific analytes and is carried through the entire analytical procedure with the samples. The term blank spike is used in reference to organic analyses, whereas the laboratory control sample is used in reference to inorganic analyses; however, they are basically the same.

3.7 Surrogates

Surrogates are organic compounds that have similar characteristics and behavior as the target analytes but are either not naturally occurring (such as deuterated surrogates for gas chromatography/mass spectrometry (GC/MS) analyses) or are not expected to be naturally occurring in the analyzed samples. Surrogates are added to every blank, sample, matrix spike, matrix spike duplicate, and standard, and are used to evaluate analytical efficiency of the analytical method by measuring percent recovery. Surrogates are normally added prior to extraction to portions of samples that will be analyzed for all GC, GC/MS, and high-performance liquid chromatography (HPLC) methods.

3.8 Quality Control (QC) Levels

USEPA QC Level IV is appropriate to use for laboratory analysis for sites where cleanup decisions will be based on risk assessment; sites on or eligible for the National Priorities List (NPL) will also have laboratory analyses conducted at Level IV QC. Other QC levels may be appropriate for certain types of samples or analyses; criteria for selection of the appropriate QC level for individual projects and field work activities are discussed in AMEC SOP, *Data Validation Planning and Coordination*.

4.0 RESPONSIBILITIES

The Project QA Coordinators and the Laboratory Manager are responsible for identifying instances of non-compliance with this procedure and ensuring that future laboratory analytical activities are in compliance with it.

The Technical Director/QA is responsible for ensuring that sample analytical activities during all Projects are in compliance with this procedure.

5.0 PROCEDURES

Laboratory QC checks include all types of samples specified in the requested analytical methods, such as the analysis of laboratory blank, duplicate, and matrix spike samples. Types of QC samples are discussed in general below. The procedures presented below are minimum requirements; QC requirements of each analytical method must also be followed, and take precedence over this SOP.

5.1 Laboratory Blanks

Laboratory blank samples are analyzed to assess the degree to which laboratory contamination by reagent or method preparation may have affected sample analytical results. At a minimum, one laboratory blank will be analyzed per matrix per analytical method for each batch of at most 20 samples.

In evaluating the blank results, all blank data are reviewed to identify any compounds detected in the blanks. The laboratory shall be contacted to discuss detection of analytes in blank samples only in the event of unusual contamination, but not for common laboratory contaminants at low levels. The following compounds are considered to be common laboratory contaminants: acetone, methylene chloride, 2-butanone, and common

phthalate esters. The data for samples analyzed during the same time period as the blank is then evaluated to identify the presence of any contaminants found in the blanks. The presence of the blank contaminants found in associated samples is then evaluated to avoid potential misinterpretation of actual sample constituents. Briefly, as discussed in the data validation SOPs, any analyte detected in both the sample and the associated blank is qualified as not detected if the sample concentration is less than 5 times the blank concentration (5x rule). For common laboratory contaminants (methylene chloride, acetone, toluene, 2-butanone, and common phthalate esters), a 10x rule applies.

5.2 DUPLICATES

Laboratory duplicates are analyzed to evaluate the reproducibility, or precision, of the analytical procedures for a given sample. Results of duplicate analyses are reported as the RPD, which is calculated by dividing the absolute value of the difference in concentration between the duplicate and original sample analyses by the arithmetic mean of their concentrations and multiplying the result by 100. One duplicate sample is analyzed for each batch of at most 20 samples analyzed of similar matrix. Duplicate analyses are normally performed on sample portions analyzed for inorganic constituents. For organic analyses, duplicate analyses are performed on matrix spike samples (see Section 5.3 of this procedure).

5.3 MATRIX SPIKES/MATRIX SPIKE DUPLICATES

Matrix spike (MS) analyses are conducted by the laboratory to assess the accuracy of specific analytical methods and to provide information on the effect of the sample matrix on the analytical methodology. Spike analyses are performed by adding compounds of known concentration to a sample, an unspiked portion of which has previously been analyzed or is concurrently analyzed; spikes are representative target compounds for each analytical method performed. The spiked sample is reanalyzed and the original and the spiked sample results are compared. One matrix spike is analyzed for each batch of at most 20 samples of similar matrix. Since MS samples only provide information about the specific sample matrix used for the spike, MS analyses should be performed for each type of matrix collected.

For the matrix spike duplicate (MSD), a separate sample is separately spiked and analyzed. As discussed in Section 5.2, results of matrix spike duplicate analyses are

reported the RPD, which is calculated by dividing the difference in concentration between the matrix spike duplicate and the matrix spike sample analyses by the arithmetic mean of their concentrations. One matrix spike duplicate analysis is required for at most each 20 samples of similar matrix.

5.4 Blank Spikes, Surrogates, and Laboratory Control Samples

Blank spikes, surrogates, and laboratory control samples are used to demonstrate that the laboratory process for sample preparation and analysis is under control.

Analytes selected for spiking of blank spikes and laboratory control samples are usually the same compounds used to spike MS/MSD samples and are representative target compounds.

At least two pesticides should be used as surrogates when pesticide analyses are being performed, and one polychlorinated biphenyl (PCB) when PCBs are analyzed. For wet chemistry methods, a single spike of an appropriate control for each method may be used for laboratory control sample analyses (i.e., cyanide, a control standard of sodium cyanide from a source other than that used for calibration may be spiked into water samples and analyzed with the water samples). For metals, at least three metals typically analyzed by inductively coupled plasma (ICP) must be monitored, and each element analyzed by graphite furnace atomic absorption and cold-vapor atomic absorption needs to be monitored. Blank spikes and laboratory control samples should be analyzed at a frequency of 1 per batch of at most 20 samples analyzed of similar matrix. Surrogates are required to be analyzed with all samples analyzed for volatile organics, base/neutral-acid extractables, and pesticides/PCBs.

6.0 RECORDS

Records of laboratory QC samples analyzed during Project activities will be maintained on laboratory bench sheets, raw data sheets, in the laboratory computerized data system, and on QC summary forms as requested. These QC summary forms will be provided in the laboratory analytical reports and laboratory data packages transmitted for each Project.

7.0 HEALTH AND SAFETY

Applicable to laboratory personnel only.

8.0 REFERENCES

NEESA. 1988. Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program. NEESA 20.2-047B. June.

NFESC. 1996. Navy Installation Restoration Laboratory Quality Assurance Guide. February.

SOP, *Data Validation Planning and Coordination*

9.0 ATTACHMENTS

None.