Chapter 2
Characteristics of PE Samples

2.1. Definitions of PE Samples.

A PE sample is a physical sample that is representative of a relevant matrix and contains one or more analytes of interest at concentration levels unknown to laboratories being evaluated. PE samples are used to evaluate the analytical and reporting performance of a laboratory under prescribed conditions against a given set of criteria.

2.2. Purposes of PE Samples.

The USACE Environmental QA Program uses PE samples to evaluate laboratory performance before a contract award and to continually monitor laboratory performance after a contract award. PE samples are not used as the sole tool for determining laboratory performance on sample analysis or data reporting, but as an integral element of the USACE Environmental QA Program. PE samples serve not only as a distinct and effective indicator for laboratory performance and data quality, but also as an efficient and effective tool to assist laboratories in improving data quality over time.

2.3. Types of PE Samples.

2.3.1. Fields of Testing. There are several ways to classify PE samples according to analytical parameters, target analytes, sample matrices, analytical methods, preparation techniques, method of applications, regulatory programs, etc. The USACE's PE samples are classified according to field of testing which are organized as "matrix-method-parameter/analyte" combinations (e.g., volatile organic compounds in water by Methods 5030B/8260B, lead in soil by Method 6010B.)

2.3.1.1. Matrix. The matrix of PE samples is generally classified as water or soil based on sample preparation methods. Most PE samples sent by the USACE are prepared with fortified reagent water or clean natural matrices that do not contain contaminants at detectable levels. Well characterized real-world matrices that contain analytes of interest are also used to prepare PE samples.

2.3.1.2. Method. The method of the matrix-method-parameter/analyte combinations indicates the sample preparation and analytical methods to be evaluated. PE samples are generally developed for evaluation of one or more preparation and analytical methods.

2.3.1.3. Parameter/Analyte. The parameter/analyte is usually a project-specified, method-listed, or program-regulated analytical parameter, target analyte, or group of target analytes.

2.3.2. Blindness. Based on the degree of blindness of the identity of PE samples, PE samples are classified as single or double blind.

2.3.2.1. Single Blind. A single blind PE sample is identified as a PE sample, but its composition is unknown to the laboratory. Compared with double blind PE samples, single blind PE samples are easier, less expensive to prepare and use, and are the most commonly used by the USACE Environmental QA Program.

2.3.2.2. Double Blind. Both the identification and composition of a double blind PE sample are unknown to the receiving laboratory. Double blind PE samples mimic field samples in analytes, concentrations, matrices, interferences, packaging, and related sample documentation. Double blind PE samples are indistinguishable from routine field samples so that a laboratory will not bias analytical performance or reports.

Which sample type is preferred? In certain situations, double blind PE samples are the better choice for assessing laboratories. However, single blind PE samples are the more practical and economical choice for the following reasons:

- The instability of water PE samples and the heterogeneity of soil PE samples are major concerns in double PE sample production and use.
- It is costly and difficult to obtain statistically valid acceptance criteria for project-specific double blind PE samples.
- Making PE samples indistinguishable from routine samples is a substantial obstacle.

2.3.3. Packaging. Based on sample preparation and packaging, PE samples can also be classified as full-volume or ampule.

2.3.3.1. Full-Volume. Full-volume PE samples mimic real-world field samples in composition, amount, and packaging. Because they are handled like real-world field samples in terms of log-in, storage, preparation, analysis, and reporting, they provide an evaluation of the entire operation of a laboratory from sample receiving to data reporting. However, it is more difficult and expensive to prepare and use full-volume PE samples due to their short shelf-lives. The majority of USACE's PE samples are full-volume PE samples that are freshly prepared prior to shipment.

2.3.3.2. Ampule. Ampule PE samples are concentrates sealed in glass ampules. Laboratories follow special instructions to dilute the concentrates prior to sample preparation and analysis. Because the concentrates are flame sealed, the shelf-lives of ampule PE samples are usually much longer than those of full-volume PE samples. A large quantity of ampule PE samples is prepared and distributed for interlaboratory round-robin studies. The USACE uses ampule PE samples for a few less stable PE

samples such as volatile organic compounds in soil and organophosphorus pesticides in water. Ampule samples can only be used as single blind PE samples.

2.4. Requirements of PE Samples.

Guidelines for PE sample composition and certification are described below.

2.4.1. Sample Composition. Ideally, PE sample analytes and matrices should be designed and selected based on certain key aspects of a specific project such as project DQO, site contaminants, analytical methods required, etc. Because of variations in DQO or site contaminants, total site-specific PE samples may be unavailable in a timely or cost-effective manner. It may be more practical to develop generic PE samples that resemble various actual field samples and provide a wide spectrum of challenges to all laboratories.

2.4.2. Sample Certification. Because PE sample results can be used to disqualify a laboratory from being awarded a contract or to reject the analytical data produced by a laboratory, the quality of PE samples should be scientifically valid and legally defensible. To ensure the quality of samples, the following objectives must be met:

- The samples must be carefully designed and prepared with traceability to national or international reference standards and with proper documentation for legal defensibility.
- The acceptance limits of PE samples should be based on the results of interlaboratory studies and/or published method performance information.
- PE samples from different production batches or designs must offer a consistent challenge. PE samples must be of high quality, well documented, homogeneous, stable, and affordable.