



Standard Guide for Spiking into Aqueous Samples ¹

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1. Scope

1.1 This guide covers the general technique of “spiking” a broad range of materials into aqueous media. This guide will serve the analyst in preparing spiked samples for quality control purposes. Guidance is also provided to aid the analyst in calculating recoveries and interpreting results. It is the responsibility of the analyst to determine whether the procedures and materials described here are appropriate to the task at hand.

1.2 The procedures in this guide are focused on “matrix spike” preparation, analysis, and interpretation of results. The applicability of these procedures to the preparation of calibration standards, calibration check standards, laboratory control standards, reference materials, and other quality control materials by spiking is incidental. A sample (the matrix) is fortified (spiked) with the analyte of interest for a variety of analytical and quality control purposes. While the spiking of multiple sample portions is discussed, the method of standard additions is not covered.

1.3 This guide is intended for use in conjunction with the individual analytical test method that provides procedures for analysis of the analyte or component of interest. The test method is used to determine an analyte or component’s background level and, again after spiking, its now elevated level. Each test method typically provides procedures not only for samples, but also for calibration standards or analytical control solutions, or both. These procedures include preparation, handling, storage, preservation, and analysis techniques. These procedures are applicable by extension, using the analyst’s judgement on a case-by-case basis, to spiking solutions, and are not reiterated in this guide. See also Practice E 200 for preparation and storage information.

1.4 These procedures apply only to analytes that are soluble in water at the concentration of the spike plus any background material, or to analytes soluble in a solvent that is itself water-soluble. The system used in the later case must result in a homogeneous solution of analyte and sample. Meaningful recovery data cannot be obtained if an aqueous solution or homogenous suspension of the analyte of interest in the sample cannot be attained. These procedures may be applicable to

microbiological preparations if the homogeneity of the suspension can be adequately maintained throughout the course of the analysis, for example, by mechanical agitation or stirring.

1.5 Matrix spiking may be performed in the field or in the laboratory, depending on which part of the analytical process is to be tested. Field spiking tests the recovery of the overall process, including preservation and shipping of the sample. Laboratory spiking tests the laboratory process only. Spiking of sample extracts, concentrates, or dilutions will test only that portion of the process subsequent to addition of the spike.

1.6 The values stated in SI units are to be regarded as the standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1129 Terminology Relating to Water²
- D 1193 Specification for Reagent Water²
- D 3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents³
- D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water²
- D 4375 Terminology for Basic Statistics in Committee D-19 on Water²
- E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis²

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *matrix spike, n*—the quantity (mass) of a component (analyte) of interest that is added to a sample (matrix) in order to test the bias as measured by recovery (of that component under specific analytical conditions) and reported as percent recovery (*P*).

3.2.2 *spike, v*—the addition of a known amount of an analyte of known identity to a measured volume of a sample

¹ This guide is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.02 on General Specifications, Technical Resources, and Statistical Methods.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.02.