STANDARD OPERATING PROCEDURE
PREPARATION OF BLIND MATRIX SPIKES

KEY WORDS

Quality Control, sample fortification, COC

APPROVALS

Original SOP signed by the following 4/18/05
APPROVED BY: _____ DATE: _____
John Sanders Ph.D.
Environmental Monitoring Branch Management

4/15/05
APPROVED BY: _____ DATE: _____
Randy Segawa
Environmental Monitoring Branch Senior Scientist

4/12/05
APPROVED BY: _____ DATE: _____
Cindy Garretson
Environmental Monitoring Branch Quality Assurance Officer

4/11/05
PREPARED BY: _____ DATE: _____
Carissa Ganapathy
Associate Environmental Research Scientist

Environmental Monitoring Branch organization and personnel, such as management, senior scientist, quality assurance officer, project leader, etc., are defined and discussed in SOP ADMN002.

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1.0 INTRODUCTION

1.1 Purpose

A blind spike is a blank matrix sample that has been spiked with analyte and then submitted to the lab disguised as a field sample. Blind spike submission is part of the Environmental Monitoring Branch QAQC program which is described in SOP QAQC001.00. The purpose of this SOP is to describe the preparation of paperwork and blind samples for the laboratory. The analytical lab does the actual spiking. An agreement was made between the Environmental Monitoring Branch and the laboratory, that the chemist analyzing regular samples must not be involved whatsoever with the making of blind spikes.

1.2 Definitions

1.2.1 **Spike or Fortification** are both terms used to describe the addition of analyte to sample matrix. For example 0.10 micrograms of malathion added to 1 liter of water is a 0.10 ppb spike or fortification.

2.0 MATERIALS

2.1 Sample matrix (like river water, well water, water with sediment added, soil, resin tubes etc.)

2.2 Sample container

2.3 Chain of custody (COC)

2.4 Sample labels

2.5 Permanent marker

2.6 Clear tape

3.0 PROCEDURES

3.1 Requesting Blind Spikes

Two to three weeks prior to a sampling event the laboratory liaison or Quality Assurance officer makes a written request for blind spikes and submits it to the laboratory.

3.1.1 The request must list the matrix, analyte, spike level and storage procedures.

3.1.2 The lab liaison gives the lab sample containers and matrix to make the blind spikes. The containers must match those used for field sampling. The matrix should be similar to the field-sampling matrix.

3.1.3 The lab chemist creates the blind spikes following laboratory standard operating procedures.

3.1.4 The chemist labels the blind spike container with the analyte(s), spike level(s), date spiked and the chemist's initials.

3.1.5 Spikes must be held under proper storage procedures similar to field samples. Storage procedures are usually listed in the study protocol.

3.1.6 The lab liaison obtains the spike samples prior to submitting field samples to the lab. The liaison stores the spike sample appropriately until submission.

3.2 Labeling and Paperwork

Prior to submitting field samples to the lab, the liaison or QA officer labels the blind spikes.

3.2.1 Remove the spiked sample(s) from storage. Tape a sample label (SOP QAQC005.00) onto the container. Hint: if the samples are stored in the walk-in refrigerator, tape the label onto the bottles while in the refrigerator to prevent condensation from forming on the bottles. The condensation prevents the tape from adhering. Otherwise, dry condensation off bottles for tape to stick.

3.2.2 If making multiple blind spikes, leave the chemist's label on each spiked sample until after all samples are labeled. This keeps samples from getting mixed up.

3.2.3 Prepare each COC to look like the sample is real. Use proper procedures outlined in SOP ADMN006.01 as for real samples. The best way to disguise the COC is to have project staff sign the COC or even fill out the whole COC with typical field information. If field sample COCs are dirty, folded, or otherwise messy it is wise to disguise blind spike COCs by roughing them up a bit.

3.2.4 One by one, remove the chemist's sticker on the blind spike container and place it on the pink copy of the COC. If computer generated COCs are used, make a copy on pink paper and place the label on it.

3.2.5 Remove all pink copies from the COCs. The QA officer or liaison files the pink copies. The pink copy acts as a matching receipt to the white COC that will go to the lab.

3.2.6 Sample bottles and jars lids need to be labeled with permanent marker like a real sample. For example, many bottles are labeled TR, OP, P, P_T etc.

3.2.7 A check-out sheet, which lists samples bound for the lab, should be prepared according to SOP QAQC003.02. The blind spike sample number should be mixed in with regular field sample numbers listed on the sheet. The blind spikes should not regularly be the last several sample numbers on the list.

3.2.8 Spiked water samples should be placed in six-packs and ice chests with the field samples. Air blind spike samples should be placed in plastic bags with field samples. It is best if samples are in a similar order as on the check-out sheet.

3.3 When results return from the lab

3.3.1 When lab results return, match up the pink blind spike COCs with the white COCs using the sample numbers.

3.3.2 The analytes detected (white COC) should match the analytes listed on the chemist's label placed on the pink COC. If not, there is an error and the laboratory supervisor must be called immediately.

3.3.3 Calculate the percent recovery by

(Amount detected ÷ amount spiked) X 100

3.3.4 The percent recovery should fall within the control limits set for that analyte (QAQC001.00). If the sample recovery is way beyond the control limits (4 to 6X the SD of the validation) or is not detected

then the liaison requests that both the chemist who made the spike and the analyzing chemist check their data and calculations. If a mistake is not found, then the back-up blind spike and/or extract need to be analyzed. The liaison or QA officer may request theses measures any time results exceed warning or control limits if necessary.

3.3.5 All blind spike results must be reported to the lab and the project leader.

4.0 STUDY-SPECIFIC DECISIONS

4.1 The following tips may help in the preparation of blind matrix spikes.

4.1.1 SOP QAQC001.00 should be consulted to determine the number of blind spikes for a particular study and the number should be listed in the study protocol QC section.

4.1.2 Generally blind spikes should be made no more than 2 to 3 days prior to extraction. Metabolites should not be spiked with parent compounds unless the half-life is several weeks.

4.1.3 Blind spikes made with analytes having short half-lives need to be spiked close to or on the day samples will be extracted.

4.1.4 Spike levels should be similar to levels that may be detected in the field study. Very high levels may not be possible if the standard precipitates in the matrix water and will not go into solution.

4.1.5 With two separate laboratories, one lab can create blind spikes that can be submitted by the lab liaison to another lab. Analytical standards may need to be swapped to determine differences in purchased standards.