1. Purpose and Scope: Control samples consist of blanks (negative controls) and spikes (positive controls). Field personnel collect blank samples to demonstrate that sample collection, preparation and handling did not introduce contamination into the sample. Spiked samples are prepared to show that the samples are stable during collection, storage and shipment.

As a quality control check for the Program, samples of neat (undiluted) pesticide or the formulated product are routinely sampled to determine the percent of active ingredient present. Instructions on how to collect neat and formulated pesticides are also included in this SOP.

This SOP describes how to prepare spike and blank samples for water and dye cards and how to collect neat and formulated pesticide samples.

Program or pesticide-specific instructions on preparing control samples found in the Environmental Monitoring Plan (EMP) supersede the instructions contained in this SOP.

2. Water Controls:

2.1 Supplies Required: To request sampling supplies, contact the monitoring supplies coordinators, Lisa Mosser (305) 278 4902, or Richard King (305) 278 2905, Center for Plant Health Science and Technology, Miami, or email the Environmental Monitoring Supplies Checklist to lisa.k.mosser@usda.gov or richard.a.king@usda.gov.

2.1.1 distilled or deionized water (obtain locally)

2.1.2 appropriate sampling equipment (e.g. see SOP EM - 3, *Collection of Water samples* or SOP EM - 4, *Collection of Run-off Water Samples*)

2.1.3 spiking solution

2.1.4 APHIS Form 2060 (Environmental Monitoring Form)

2.2 Preparing a Blank Water Sample (negative control):

2.2.1 Using the same equipment as will be used to collect an environmental water sample, collect a ½ gallon sample of the distilled water. If the sample is to be taken from a stationary or flowing water body, or ground water, then the control should be collected by pouring a ½ gallon of the distilled water from the distilled water container into a sample container (cubitainer or 1 gallon glass jug). If a run-off water sample is to be collected, then pour a ½ gallon of the distilled water through a clean screen and funnel that will be used as part of an ARSE and into a sample container (cubitainer).

2.2.2 Check and record the pH of the negative control sample, if required adjust the pH and add buffer to stabilize the sample (i.e. for negative control samples of a program using malathion, adjust the pH to 5).

2.2.3 Seal the negative control sample container and handle it as you would any other water sample.

2.3 Preparing a Spiked Water Sample (positive control):

2.3.1 Collect an extra sample of the water being sampled using standard procedures. If possible collect the sample before any Program treatments (see the SOPs referenced in 2.1.2 above for directions on how to collect the sample).

2.3.2 Before freezing the extra sample, take it to a location sufficiently removed from where the environmental samples are being frozen to prevent contamination of the environmental samples.

2.3.3 Open the sample container, then open the vial provided by the lab containing the spiking solution and drop the vial, its contents and the vial's cap into the sample. Re-seal the sample, invert it three times gently and then freeze it along with the environmental samples.

3. Dye Card Controls:

3.1 Supplies Required: To request the supplies required to prepare or collect control samples, follow the procedure described in section 2.1 above.

3.1.1 dye cards

3.1.2 10 :L syringe

3.1.3 disposable gloves

3.1.4 spiking solution

3.1.5 CPHST Lab SOP for spiking the specific pesticide

3.1.6 paper clip (or alligator clip)

3.1.7 APHIS Form 2060 (Environmental Monitoring Form)

3.2 Preparing a Blank Dye Card Sample (negative control):

3.2.1 Before heading out for the day when dye cards are going to be collected, hang an unexposed dye card from the rear-view mirror of your vehicle using a bent paper clip or alligator clip. Leave the dye card in this position throughout the day until all dye card samples have been collected and are ready to transport back to the freezer.

3.2.2 After the last dye card sample has been collected for the day, collect the negative control dye card hanging from the rear-view mirror (see SOP EM - 1, *Collection of Dye Card Samples*).

3.3 Preparing a Spiked Dye Card Sample (positive control):

3.3.1 After collecting dye card samples, if the EMP calls for preparing a spiked dye card sample, then go to a location sufficiently removed from where the dye card samples are being frozen to prevent contamination of the dye card samples and conduct the spiking.

3.3.2 Follow the spiking procedure described in the SOP prepared by CPHST lab for the specific pesticide being sampled (the EMP will either have the appropriate SOP attached or describe which SOP to order from CPHST lab along with the supplies).

3.3.3 Once spiked, treat the positive control dye card like all of the other dye card samples.

4. Neat or Formulated Pesticide Samples:

4.1 Supplies Required: To request the supplies required to prepare or collect control samples, follow the procedure described in section 2.1 above.

4.1.1 sample bottle

4.1.2 disposable pipettes

4.2 Collecting a Neat or Formulated Pesticide Sample:

4.2.1 Conduct the sampling in a location and in a manner consistent with safe pesticide handling. Be sure to review the MSDS and label for cautions and safety instructions about the pesticide.

4.2.2 Neat pesticide stored in drums should be agitated before sampling.

4.2.3 Open the top of the drum and collect a sample several inches below the surface using the disposable pipette (Do NOT mouth pipette). Fill the sample container nearly full to minimize the air and seal the sample container. Dispose of the pipette according to State regulations.

4.2.3 Pesticide in bulk storage can be sampled by collecting a sample while it is being transferred to a sprayer as described in 4.2.2 above.

4.2.4 Pesticide formulations can be collected from the tank where the final mixing takes place. Follow the procedure described in 4.2.2 above or if there is a purge valve on the tank then a sample can be collected through that valve.

5. Documentation:

5.1 Complete an APHIS Form 2060 for each positive or negative control sample. Be sure to record whether it is a blank or a spike sample. For every spiked water sample, it is essential that it can be identified with the actual sample that was collected from the same spot at nearly the same time. This way, if there was residue of the pesticide of interest in the water, then the amount already in the water can be subtracted from the amount in the spiked sample so that the recovery can be calculated.

5.2 Once the APHIS Form 2060 is completed, retain the pink copy for your records and distribute the remaining copies as specified in the EMP.

6. Packaging and Shipping:

6.1 Package and ship positive and negative control samples as described in SOP EM - 17, *Packaging and Shipping of Samples.*

6.2 Package and ship neat and formulated pesticide samples **separate** from any other control or environmental samples. Use disposable corrugated boxes without dry ice. Neat and formulated pesticide samples can contaminate storage areas, shipping containers and other samples.