

**United States Department of Agriculture  
Agricultural Marketing Service, Science & Technology  
Pesticide Data Program**

SOP No.: PDP-QC-01		Page 1 of 6
Title: Blanks and Spikes Required per Set		
Revision: 7	Replaces: 10/1/98	Effective: 06/01/02

**1. Purpose:**

To provide requirements for quality control (QC) samples to be analyzed with each set of USDA/AMS-Pesticide Data Program (PDP) samples.

**2. Scope:**

This standard operating procedure (SOP) shall be followed by all analytical laboratories conducting pesticide residue studies for PDP, including support laboratories conducting stability or other types of studies which may impact the program.

**3. Outline of Procedure:**

- 5.1 Blanks and Spikes Required per Set
  - 5.1.a Reagent Blank
  - 5.1.b Matrix Blank
  - 5.1.c Matrix Spike(s)
  - 5.1.d Process Control Spikes

**4. References:**

- USDA/AMS PDP Quality Assurance (QA)/Technical Meeting, April 9-11, 2002
  - Quality Assurance (QA) Committee Meeting, May 19-21, 1998
  - Quality Assurance (QA) Committee Meeting, July, 1996
  - GLP Committee Meeting, April 1992
  - FDA, Standard Operating Procedures for the Total Diet Study, KCM TD G2, revision 0, Quality Assurance, January, 1993
  - Federal Register, Volume 49, Number 209, "Rules and Regulations", October, 1984
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**5. Specific Procedures:**

These standard operating procedures represent minimum PDP requirements and are presented as general procedures. Each laboratory shall have written operating procedures which shall provide specific details concerning how the procedures have been implemented in that laboratory.

5.1 A sample set is a group of samples which are spiked individually with the designated process control(s), extracted on a single day along with the required QC samples, and analyzed with the applicable required QC samples. Each set shall not exceed 24 samples. Required QC samples per set consist of a reagent blank, matrix blank, and matrix spike(s).

Each laboratory is given the option of combining two or more small sets into a larger set (e.g., peaches month A + peaches month B or apples month A + peaches month A). If the larger set contains two commodities, then the set shall contain a matrix blank of each commodity and a matrix spike(s) in at least one of the commodities.

The matrix spike(s) shall contain at least the marker pesticides (refer to QC-13). All reported compounds (markers, required, and any other compound reported by that laboratory) shall be spiked at least twice per year for each commodity group. All components of sample sets shall be subject to the same analytical process as detailed in the method SOP's.

a. **Reagent Blank**

A reagent blank is intended to demonstrate glassware cleanliness and total system integrity. It shall be prepared by subjecting an amount of distilled water equivalent to that contained in an average sample to the entire analytical process. For consistency in the preparation of the reagent blank, it shall be assumed that an "average" (includes fresh, canned, or frozen) fruit or vegetable sample contains 80% water. If contamination or interferences in the retention time window of the pesticide of interest is present in excess of the calculated LOQ, corrective action must be taken and documented.

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b. Matrix Blank

A matrix blank is intended to demonstrate the behavior of a substrate within an analytical system. Ideally, a matrix blank should be void of any compounds of interest. A matrix blank may be a previously characterized sample of the same commodity. If a suitable sample is not available, a portion of one of the samples may be randomly selected and used as a matrix blank. If an incurred residue is found in the matrix blank which has been chosen from the sample set, determine if the same residue is incurred in the actual sample and is not present in other samples in the same set. If this condition cannot be met, corrective action must be taken, such as reviewing reagent blank information.

c. Matrix Spike(s)

A matrix spike is intended to reflect the behavior of a chemical in a substrate within an analytical system. The matrix spike indicates the behavior of the chemical for the entire sample set.

A second portion of the same material used for the matrix blank shall be used for the matrix spike(s). Spike prior to extraction at approximately 2 x LOQ with the PDP marker pesticides as specified in QC-13. Additional spikes may be added to satisfy the biannual spiking of each commodity group with all reported compounds, as part of a validation study under QC-7, or to familiarize a laboratory with pesticides that have not been previously analyzed. More than one matrix spike shall be required if necessary for all spiked compounds to be separated during the chromatographic process. If a laboratory has combined commodities within a set, then the PDP QA Form shall indicate which commodity was used for the matrix spikes. Results for all spiked compounds shall be reported to Manassas through normal RDE procedures.

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d. Process Control Spikes

A process control spike is intended to assure the integrity of a particular sample within an analytical system. Each sample set component, except the reagent and matrix blanks, shall be spiked with a process control at approximately 5 x LOQ. Refer to PDP-QC-13 for a list of process controls required for each type of analysis.

The process control shall be added before extraction. However, if the intent of the process control is to monitor the percent recovery of a clean-up step, or of a derivitization, then the process control shall be added to the extract before the clean-up or derivitization step.



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*05/15/02*

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