

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

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Title: Quality Control Criteria		
Revision: 11	Replaces: 07/01/07	Effective: 04/01/08

1. Purpose:

To provide standard quality control criteria for the USDA/AMS Pesticide Data Program (PDP).

2. Scope:

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

3. Outline of Procedures:

- 6.1 Process Control Criteria
 - 6.2 Fortification Recovery Criteria
 - 6.3 Evaluation of Recoveries
 - 6.4 Proficiency Testing Results Criteria
- Attachment 1 – Process Control and Spike Recovery Acceptability Flowchart

4. References:

- USDA/AMS PDP Quality Assurance/Technical Meeting, February 26-28, 2008, Crystal City, VA
 - USDA/AMS PDP Quality Assurance/Technical Meeting, March 20-22, 2007, Crystal City, VA
 - USDA/AMS Combined Microbiological Data Program/PDP Technical-Quality Assurance Meeting, March 27-31, 2006, Richmond, VA
 - USDA/AMS PDP Quality Assurance (QA)/Technical Meeting, April 9-11, 2002
 - USDA/AMS PDP Quality Assurance Meeting, May 18-20, 1999
 - USDA/AMS PDP QA Committee Meeting, June 10-11, 1997
 - USDA/AMS PDP QA Committee Meeting, July 9-11, 1996
 - USDA/AMS, EPA/OPP Meeting, May 3, 1993
 - USDA/AMS PDP Technical Meeting, March 2-3, 1993
 - USDA/AMS PDP QA Committee Meeting, April 27, 1992
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- Association of Official Analytical Chemists (AOAC), Quality Assurance Principles for Analytical Laboratories, 1991, pp. 91-94
- Taylor, Quality Assurance of Chemical Measurements, Taylor, 1989, pp. 15-39
- U.S. EPA, Standard Operating Procedures, 40 CFR part 160.81, August 17, 1989

5. Narrative:

There are two different criteria that may be used for evaluating process control and spike recoveries, “Absolute Range Criteria” and “Statistically Calculated Range Criteria.” The Absolute Range Criteria does not follow pure statistical theory but was set following a review and study of PDP interlaboratory QC process standard and spike recoveries data. A decision whether or not any given sample result should be re-run, re-extracted, etc. may take into account practical considerations such as cost of re-working the sample as well as the importance of particular analyte data to MPO (see analyte priority in applicable commodity-specific memorandum).

6. Specific Procedures:

This SOP represents minimum PDP requirements and is presented as a general guideline. Each laboratory shall have written procedures that provide specific details concerning how the procedure has been implemented in that laboratory.

6.1 Process Control Criteria

6.1.a. Each sample shall be spiked with a process control at approximately five times the Limit of Quantitation (LOQ) prior to the extraction step of the analytical procedure.

6.1.b Each laboratory shall decide whether to use the Absolute Range Criteria or the Statistically Calculated Range Criteria. A laboratory may choose different Range Criteria for different test types, but it is intended that a laboratory stay with the chosen criteria unless a letter of explanation is submitted by the Technical Program Manager (or designee) and approved by the laboratory QA officer.

6.1.c Absolute Range: Each process control recovery shall fall between 50-150% for all detection systems used to calculate sample data.

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6.1.d Statistically Calculated Range: The mean recovery for a sample set's process control shall be calculated by averaging. Each process control recovery shall fall within its acceptance recovery range which is the mean recovery \pm three standard deviations.

6.1.e Response To Failure To Meet Chosen Criteria Range:

6.1.e.1 When a process control falls outside the chosen range criteria, the Technical Program Manager (TPM) or designee must investigate the problem. Any one of the following options, or combination thereof, may be chosen by the TPM or designee as the course of action to be taken. All action(s) shall be documented.

6.1.e.1.a The original extract may be re-injected or re-aliquoted. If the process control recovery falls within the chosen range criteria, then the results from the re-injected or re-aliquoted extract shall be reported.

6.1.e.1.b The TPM or designee may further investigate the problem by examining control charts to determine whether there is a trend which may indicate a process problem. If a problem is found with the process, appropriate action shall be taken.

6.1.e.1.c The sample may be re-extracted from the frozen homogenate. Both the original and re-run results shall be reported. If neither of the extractions resulted in process control recoveries that are acceptable, then analyte data may need to be coded as "Unable to Analyze."

6.1.f Reporting Process Control Recoveries:

6.1.f.1 The value reported as "percent recovery" may be the original, re-injected, or re-aliquoted determination value [either value from primary detection system or averaged value (e.g., dual column results averaged)].

6.1.f.2 The value reported as "percent rerun" should be any applicable re-extraction (from homogenate) value [either value from primary detection system or averaged value (e.g., dual column results averaged)].

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6.2 Fortification Recovery Criteria

6.2.a A blank sample of the given matrix shall be fortified with PDP selected pesticides (see PDP-QC-13) at approximately two times the LOQ. If no blank matrix is available, purchased matrix may be used as a blank or a portion of one of the samples in that set may be randomly chosen as a blank. Additional pesticides may be added to the fortification mixture. However, the recoveries of these additional pesticides are not required to meet the fortification recovery criteria. Subtraction of incurred residues is allowed if the specified conditions are met.

6.2.b PDP spike pesticide recoveries shall fall between 50-150% or shall fall within three standard deviations of the mean recovery for that compound. The laboratory may elect to group recovery data across commodities, if justified.

6.2.c Response To Failure To Meet Criteria Range:

6.2.c.1 When a spiked pesticide recovery falls outside the range criteria, the TPM or designee must investigate the problem. Any one of the following options, or combination thereof, may be chosen by the TPM or designee as the course of action to be taken. All action(s) shall be documented.

6.2.c.1.a The original extract may be re-injected or re-aliquoted. If the spiked pesticide recovery falls within the range criteria, then the results from the re-injected extract shall be reported.

6.2.c.1.b The TPM or designee may further investigate the problem by examining control charts to determine whether there is a trend which may indicate a process problem. If a problem is found with the process, appropriate action shall be taken.

6.2.c.1.c The sample set may be re-extracted from the frozen homogenate. Both the original and re-run results shall be reported. If neither of the extractions resulted in fortification recoveries that are acceptable, then analyte data may need to be coded as "Unable to Analyze."

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6.2.d Reporting Fortification Recoveries:

6.2.d.1 “Fresh” spikes are matrix spikes fortified, extracted, and analyzed with that set of analytical samples. Fresh values reported may be the original, re-injected, re-aliquoted, or re-extracted (from homogenate) determination value. The results reported may be the value from primary detection system or the averaged value (e.g., dual column results averaged).

6.2.d.2 “Freezer” spikes are no longer required, except for initiation of special projects.

6.2.d.3 “Other” spikes are additional fortifications reported by the laboratory. The laboratory can request that MPO add a new spike type code as needed. Examples of “other spike” types are freezer, storage, failed fresh values, or “extra” QA spikes performed by the laboratory.

6.3 Evaluation of Recoveries

Laboratories shall use control charting or other appropriate statistical tools to evaluate recoveries and monitor trends over time.

6.4 Proficiency Testing (PT) Results Criteria

6.4.a Upon receipt of PDP PT results, the results shall be reviewed and if any corrective actions are initiated due to the results, MPO shall be informed within 30 days. Refer to SOP PDP-ADMIN-06B for notification details.

6.4.b Laboratories shall initiate corrective actions when results are considered unacceptable by the PT scheme provider (e.g., AOAC or FAPAS).

6.4.c For rounds administered by PDP (i.e., water or CDFA-issued commodity-specific rounds), unacceptable results shall be defined as those outside 50-150% recovery.

6.4.d PDP Fiscal Year (FY) PT program schedules are posted to the PDP Extranet site and are referenced in the applicable PDP Semi-Annual Program Plans.

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Revision 11 January 2008 Monitoring Programs Office

- Changed title to “Quality Control Criteria”
- Clarified criteria for evaluating process control and spike recoveries in Section 5
- Specified results may need to be coded as unable to analyze when both original and re-extracted recoveries are not acceptable in Sections 6.1.e.1.c and 6.2.c.1.c
- Added option of purchasing blank matrix to Section 6.2.a
- Removed requirement to use the last 20 data points for calculating the mean from Section 6.2.b
- Added evaluation of recoveries requirements to Section 6.3
- Added acceptability criteria for proficiency testing results to Section 6.4

Revision 10

- Modified subsection 6.2 to allow both absolute range and statistically calculated range for spike recoveries

Revision 9

- Added reference to 2006 Combined MDP/PDP Technical-Quality Assurance Meeting to subsection 4
- Changed “corrective action” to “appropriate action” in subsections 6.1.e.1.b and 6.2.e.1.b

Revision 8

- General update
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Process Control and Spike Recovery Acceptability Flowchart

