

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

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Title: Acceptability Criteria for Process Control and Fortification Recoveries		
Revision: 7	Replaces: 04/01/00	Effective: 06/01/02

1. Purpose:

To provide standard quality control criteria for each data package submitted to 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

4. References:

- 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
 - 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
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5. Narrative:

There are two different criteria that may be used for determining when a result should be re-run, re-extracted, etc. These criteria were established by taking into account some statistical concepts as well as practical considerations such as cost of re-working the sample. As a result, it should be noted that these criteria do not follow pure statistical theory. The first type of criteria mentioned relates to absolute limits. These are correlated to interlaboratory process standard and spike recoveries as verified by review of 1995 QC data. The second type of criteria relates to intralaboratory recoveries and tends to follow traditional concepts.

6. Specific Procedures to be Followed:

This standard operating procedure (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

- a. Each sample shall be spiked with a process control at approximately five times the Limit of Quantitation prior to the extraction step of the analytical procedure.
 - 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 a chosen criteria unless a letter of explanation is submitted and approved by the QA officer.
 - 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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- 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 " three standard deviations.
- e. **RESPONSE TO FAILURE TO MEET CHOSEN CRITERIA RANGE:**
1. When a process control falls outside the chosen range criteria, the Technical Program Manager or designee must investigate the problem. Any one of the following options, or combination thereof, may be chosen by the Technical Program Manager or designee as the course of action to be taken. All action(s) shall be documented.
 - 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.
 - b. The Technical Program Manager 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, corrective action shall be taken.
 - c. The sample may be re-extracted from the frozen homogenate. If the process control recovery falls within the chosen range criteria, then both original and re-run results shall be reported.
- f. **REPORTING PROCESS CONTROL RECOVERIES:**
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)].
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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)].

6.2 Fortification Recovery Criteria

- a. A blank sample of the given matrix shall be fortified with the PDP marker pesticides (see PDP-QC-13) at approximately two times the Limit of Quantitation (as calculated by PDP-QC-10). If no blank matrix is available, a portion of one of the samples may be randomly chosen as a blank matrix. 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.
- 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 a chosen criteria unless a letter of explanation is submitted and approved by the QA officer.
- c. **ABSOLUTE RANGE:**
 1. PDP marker pesticide recoveries shall fall between 50-150%.
- d. **STATISTICALLY CALCULATED RANGE:**
 1. The mean recovery for each PDP marker pesticide shall be calculated using a minimum of the last 20 data points acquired for each marker pesticide across all commodities. Each PDP marker pesticide shall fall within its acceptance recovery range which is the mean recovery " three standard deviations.



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e. **RESPONSE TO FAILURE TO MEET CHOSEN CRITERIA RANGE:**

1. When a marker pesticide recovery falls outside the chosen range criteria, the Technical Program Manager or designee must investigate the problem. Any one of the following options, or combination thereof, may be chosen by the Technical Program Manager or designee as the course of action to be taken. All action(s) shall be documented.
 - a. The original extract may be re-injected or re-aliquoted. If the marker pesticide recovery falls within the chosen range criteria, then the results from the re-injected extract shall be reported.
 - b. The Technical Program Manager 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, corrective action shall be taken.
 - c. The sample set may be re-extracted from the frozen homogenate. If the marker pesticide recovery falls within the chosen range criteria, then both original and rerun results shall be reported.

f. **REPORTING FORTIFICATION RECOVERIES:**

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).
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2. “Freezer” spikes are no longer required, except for initiation of special projects (e.g., poultry). This option was used for reporting field spikes for finished drinking water prior to October 1, 2001, when field spikes were discontinued. This option/button will not be included in the new, reengineered remote data entry (RDE) system (see subsection 6.2.f.3 for handling any required freezer spike data).

 3. “Other” spikes are additional fortifications reported by the laboratory. In the new RDE system, this option/button will be a one-to-many designation, allowing an unlimited number of “other” spikes. A look-up table in the new RDE system will hold “other” spike type codes/descriptions. 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.
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April 2002

PDP QA/Technical Meeting

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- Added reporting procedures for process control recoveries to subsection 6.1
 - Added reporting procedures for fortification recoveries to subsection 6.2

QC-04, Attachment 1
Flowchart for Process Control and Marker Recovery Acceptability

