

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

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Title: Injection Sequence		
Revision: 7	Replaces 07/01/07	Effective: 07/01/08

1. Purpose:

To provide standard procedures for the injection sequence to be used in the analysis of USDA/AMS Pesticide Data Program (PDP) samples.

2. Scope:

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

3. Outline of Procedures:

5.1 Injection sequence description

4. References:

- USDA/AMS PDP Quality Assurance/Technical Meeting, March 20-22, 2007, Crystal City, VA
- USDA/AMS PDP Quality Assurance (QA)/Technical Meeting, April 9-11, 2002
- 40 CFR 160.63, Maintenance and calibration of equipment

5. Specific Procedures to be Followed:

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.

5.1 Each laboratory shall develop an SOP detailing an appropriate injection sequence in order to ensure data integrity and uniform response across the sample set. "Uniform response" shall be construed as no greater than 20% relative percent difference (RPD) between calibration responses (refer to SOP PDP-DATA-03).

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5.2 Standards spanning the expected range of residue concentrations shall be included with every injection sequence for each compound analyzed. A suggested range is limit of quantitation to ten times the limit of quantitation.

5.3 Standards must be run at a minimum of the beginning and end of the data run to demonstrate calibration integrity. This may be accomplished via a single standard or a full set of calibration curve standards.

5.4 Each initial analytical run shall include the reagent blank, matrix blank, spikes, and samples. For additional runs (i.e., reinjects/dilutions) QC samples shall be run as necessary (i.e. reagent or matrix interference).

5.5 A non-extracted limit of detection (LOD) standard for each compound analyzed shall be run with each data set as a diagnostic tool (i.e., the laboratory is not required to calculate signal-to-noise ratio, but the peak must be observable). If the peak is not observable, the laboratory shall take the appropriate action (e.g., raise the LOD, re-inject the standard, etc.). For laboratories that use in-matrix calibration standards, the LOD Standard shall also be in-matrix. For laboratories that do not use in-matrix calibration standards, the LOD Standard shall be in the same solution as the calibration standards.

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Revision 7

May 2008

Monitoring Programs Office

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- Removed “”A calibration curve consisting of” from the beginning of the first sentence in Section 5.2

Revision 6

- Clarified calibration curves must be run with each sequence in subsection 5.2
- Added LOD Standard requirements to subsection 5.5

Revision 5

- Added requirements for calibration curves to subsection 5.2

Revision 4

- General update
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