

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

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|---|--------------------|---------------------|
| SOP No.: PDP-STD-03                                   |                    | Page 1 of 5         |
| Title: Analytical Reference Standards Quality Control |                    |                     |
| Revision: 6   | Replaces: 06/01/02 | Effective: 07/01/04 |

**1. Purpose:**

To provide standard procedures for quality control for analytical standards used in the USDA/AMS Pesticide Data Program (PDP).

**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 Checking standards
- 5.2 Working dilutions/mixed standards
- 5.3 Standard characterization - detector profiles

**4. References:**

- USDA/AMS PDP Quality Assurance (QA)/Technical Meeting, April 9-11, 2002
- USDA/AMS PDP Quality Assurance Meeting, April 4-5, 2000
- 40 CFR 160.105, Test, control and reference substance characterization
- 40 CFR 160.107, Test, control, and reference substance handling
- 40 CFR 160.83, Reagent and solutions
- 40 CFR 160.113, Mixtures of substances with carriers

**5. 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.

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5.1 Standard checking

Stock solutions of neat pesticide standards not previously prepared or not currently in use in the individual laboratory shall be prepared in duplicate and the two standards compared to each other. Responses for standards of comparable concentrations must match within 10% relative percent difference (RPD):

$$\text{RPD} = \frac{*\text{RF}_1 - \text{RF}_2*}{[(\text{RF}_1 + \text{RF}_2)/2]} \times 100,$$

where  $\text{RF}_1$  is the response factor<sup>1</sup> of the first analytical standard and  $\text{RF}_2$  is the response factor of the second standard. If standards do not match, a third standard shall be made and compared. This process shall be continued until two matching standards are prepared.

New stock solutions which are prepared from neat pesticides currently used in the laboratory shall be compared to the old stock solution. The two standards must match within 10% RPD (see above). If the two standards do not match, the problem must be identified and solved before the standard is used for quantitation. A suggested approach is to make new dilutions of both the old and new standards to check for dilution errors. If no dilution errors are found, a second stock dilution should be made to determine whether an error was made in the original preparation from neat material. If these two stocks match, then the standard may be used. If they do not match, a third stock solution should be made. Whenever possible, duplicate injections shall be used.

Documentation of the standard checking process shall be kept through appropriate logbooks or electronic logs. Chromatograms of all standards shall be kept indicating the standard comparisons of old and new standards and the calculated difference.

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<sup>1</sup> Area or height of each standard divided by the concentration of that standard

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5.2 Working dilutions/mixed standards

Working dilutions and mixed standards shall be checked to ensure integrity of the solutions. These solutions should be made as frequently as necessary to ensure that concentrations do not change and/or individual pesticides do not degrade. Each laboratory shall determine the frequency of remaking dilutions/mixed standards. Documentation supporting this decision shall be kept in the form of logbooks and standard chromatograms. A suggested guideline is six months for stock mixed standards and one month for working dilutions. Some pesticides may require more frequent dilution from the stock.

An archive file of all old mixed standards shall be kept and the dates the standards were used shall be indicated. The archive file shall be maintained a minimum of five years.

All working solutions shall be labeled with pesticide name, solvent, date of preparation of stock, date of preparation of dilution, expiration date, and initials of preparer. Each standard shall be identified by a unique and traceable code. Mixed working standards may be coded as long as complete documentation is maintained in a logbook.

5.3 Detector profiles

Standard retention time and response shall be characterized by analysis on the detectors used in each laboratory. These include but are not limited to: GC-ECD, GC-NPD, GC-FPD, GC-ELCD, GC-MSD, GC-AED, GC-ITD, LC-MS, and tandem MS. Libraries of all standards shall be developed for confirmatory instruments (GC-MS, GC-ITD, GC-AED, and LC-MS). Pesticides analyzed by HPLC shall be characterized using the appropriate detector.

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*Gail Parker*

*6-16-04*

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Revision 6

April 2004

PDP QA/Technical Meeting

- Added LC-MS and tandem MS to Subsection 5.3
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