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Cannabis Science Task Force Recommendations: Laboratory Quality Standards for Pesticides in Cannabis Plants and Products

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Cannabis Science Task Force Recommendations: Laboratory Quality Standards for Pesticides in Cannabis Plants and Products

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Acknowledgements

Members of the Cannabis Science Task Force steering committee and working groups have contributed countless hours of expertise to develop science-based laboratory quality standards. The collective efforts and recommendations of the Cannabis Science Task Force are detailed in this report.

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- Jeff Doughty, Capitol Analysis Group
- Julie Kowalski, Trace Analytics
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- Ben Hart, Testing Technologies
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Contributing members from Washington State agencies include:

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Executive Summary

Although it is required that cannabis products sold in Washington State be tested for harmful substances and potency, the science needed to develop adequate testing protocols has not caught up to the industry. To protect consumers and to deliver more accurate laboratory results, the Legislature created the Cannabis Science Task Force (Task Force) to develop acceptable laboratory quality standards under the provisions of House Bill 2052 (2019).

The Washington State Department of Ecology (Ecology) prepared this report to the Legislature on behalf on the Cannabis Science Task Force (Task Force) as required by RCW 43.21A.735(6), which states:

> "The cannabis science task force must submit a report to the relevant committees of the legislature by July 1, 2020, that includes the findings and recommendations for laboratory quality standards for pesticides in plants for marijuana product testing laboratories. The report must include, but is not limited to, recommendations relating to the following:
>
> (a) Appropriate approved testing methods.
>
> (b) Method validation protocols.
>
> (c) Method performance criteria.
>
> (d) Sampling and homogenization protocols.
>
> (e) Proficiency testing.
>
> (f) Regulatory updates related to (a) through (e) of this subsection, by which agencies, and the timing of these updates."

The Task Force members are professionals with expertise in chemistry, laboratory quality assurance and quality control, and state government policy. They represent the Washington State departments of Agriculture (WSDA), Health (DOH), and Ecology (Ecology), as well as the Liquor and Cannabis Board (WSLCB) and cannabis testing laboratories.

To strengthen testing protocols for pesticides in cannabis plants and products, the Task Force recommends:

- Using existing agricultural method validation protocols and method performance measures developed by the United States Department of Agriculture (USDA), adapted to cannabis plants and products.
- Establishing an interagency cooperative team staffed by WSLCB, WSDA, and DOH to maintain the adopted protocols and provide technical assistance to cannabis laboratories.
- Performing regulatory updates to facilitate these recommendations.

Consumer protections rely on assurances that laboratories can accurately test cannabis products to meet Washington state standards. Typically, the development of testing protocols relies on a depth of federal expertise and resources available to conduct research, coordinate standardization, and apply risk-management strategies. In this case, states have legalized cannabis without federal support, so this traditional framework does not exist.

Adequate and up-to-date testing protocols are needed to provide critical guidance to cannabis testing laboratories and to leverage Ecology's laboratory accreditation model. Ecology currently administers its established accreditation program for more than 400 drinking water and environmental laboratories in Washington and across the country.

Accreditation is an essential piece of a robust quality assurance program. Accreditation uses

established testing protocols to verify that a laboratory meets the criteria necessary to conduct specific testing practices.

For cannabis, it is key that Washington's regulatory agencies establish and maintain up-to-date testing protocols to fill the gap left by the lack of a federal cannabis testing framework. For Ecology to take over lab accreditation, regulatory changes must occur to allow its cannabis laboratory accreditation to function as it does for other state and federal regulatory programs (Figure 1). These regulatory changes are critical to ensure that laboratory quality standards are clear to laboratories, regulatory authorities, and lab auditors alike. These regulatory changes are also important to ensure the independence of the accreditation body from those making and using the testing data. Clear laboratory quality standards are an important tool in our state's effort to generate credible cannabis testing data to guide regulatory decisions and provide consumer protections.

Recommendations

To provide cannabis testing laboratories critical guidance and for Ecology to begin the accreditation of cannabis laboratories, several actions are needed. With the recognition that RCW 69.50.587 states that the liquor and cannabis board may adopt rules that address the findings and recommendations in the task force reports provided under RCW 43.21A.735, the Task Force recommends the following actions:

1. Implement the Task Force proposals:
   a. WSLCB adopts approved testing methods.
   b. WSLCB adopts method validation protocols and method performance criteria.
   c. WSLCB adopts sampling[1] and homogenization protocols.
   d. Ecology updates its existing proficiency testing guidance with cannabis testing laboratory criteria.
2. The WSLCB, WSDA, and DOH forms an interagency cooperative team that houses the authority and expertise to maintain the implemented Task Force proposals by July 1, 2022
   Note: As of June 2020, members from the WSLCB, WSDA, and DOH have held meetings, and will continue to meet, to determine the regulatory authority and appropriations needed to form this interagency team. It may be necessary for agency request legislation to be put forth to align agency authorities.
3. Agencies must perform regulatory updates in the following sequential order:
   a. Establish up-to-date protocols, using the Task Force proposals (a.- c., above), either in WSLCB rules (Chapter 314-55 WAC) or in applicable guidance documents by July 1, 2022.
   b. Amend DOH Chapter 246-70 WAC, as appropriate, to compliment or clarify rule updates performed by WSLCB by July 1, 2022.

[1] "Sampling" refers to in-laboratory practices only; this is commonly termed "sub-sampling". Lot and batch sampling, as specified in WAC 314-55-101, falls outside of the scope of laboratory quality standard updates provided by the Task Force.

c. Ecology begins amending rules and guidance (Chapter 173-50 WAC) with Task Force recommendations on proficiency testing for cannabis products by July 1, 2022.
d. Amend WSLCB Chapter 314-55 WAC to remove all existing accreditation rules by July 1, 2024.

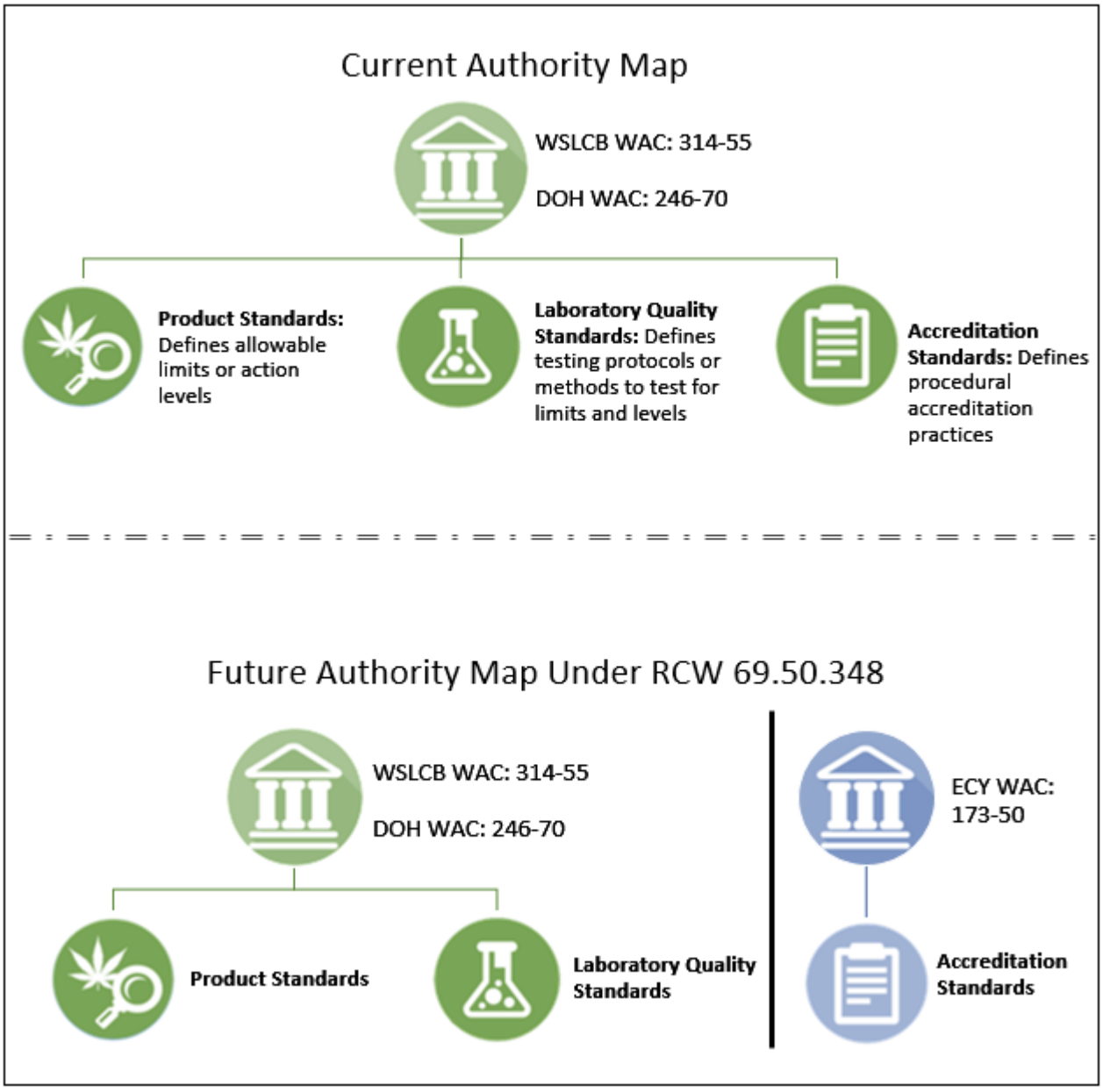

Figure 1. Comparison of current regulating authority design and future design under RCW 69.50.348.

Introduction

Background

On May 7, 2019, Gov. Jay Inslee signed House Bill 2052, which transfers the authority and responsibility for cannabis[2] testing laboratory accreditation requirements to the Department of Ecology (Ecology) from the Washington State Liquor and Cannabis Board (WSLCB) on July 1, 2024, into law. This legislation also established a Cannabis Science Task Force to develop recommendations for laboratory quality standards. Building on several recommendations set forth in Ecology's 2019 Cannabis Laboratory Accreditation Recommendations report[3], House Bill 2052 addressed some fundamental challenges facing the cannabis testing industry in Washington State.

The transfer of laboratory accreditation oversight will place cannabis testing laboratories under Ecology's well-established framework. By 2024, Ecology's Laboratory Accreditation Unit will provide the formal recognition that a laboratory is capable of providing accurate and defensible analytical data, much like Ecology currently does for the more than 400 environmental and drinking water laboratories in Washington state and across the country[4]. Laboratory accreditation ensures a laboratory possesses the technical competence to perform an identified scope of work through specified procedures and methods that make up laboratory quality standards. Accreditation is reliant on strong laboratory quality standards typically set by federal agencies who oversee protection of our agricultural and consumer products.

The Cannabis Science Task Force was established to make recommendations for appropriate cannabis laboratory quality standards. The Task Force functions as a multi-agency and industry scientist collaboration and is focused on defining and drafting meaningful science-based practices for cannabis laboratory testing. A phased approach was outlined for the Task Force deliverables (RCW 43.21A.735), with a first report due to the Legislature on July 1, 2020 that focuses on the required laboratory quality recommendations for pesticides in cannabis plants[5] and compliant intermediate cannabis products (Figure 1). A second report, due on December 1, 2021, will focus on recommendations for laboratory quality standards for potency and heavy metals testing. This report will also cover recommendations for establishing a robust cannabis-specific proficiency testing program.

[2] The term "cannabis" is used throughout this document. "Marijuana" will be used in discussions where the referenced context requires this matrix-specific term.
[3] https://fortress.wa.gov/ecy/publications/SummaryPages/1903004.html
[4] https://ecology.wa.gov/Regulations-Permits/Permits-certifications/Laboratory-Accreditation
[5] The term "flower" is used in place of "plants" throughout this document, as flower is the component of the cannabis plant that is tested.

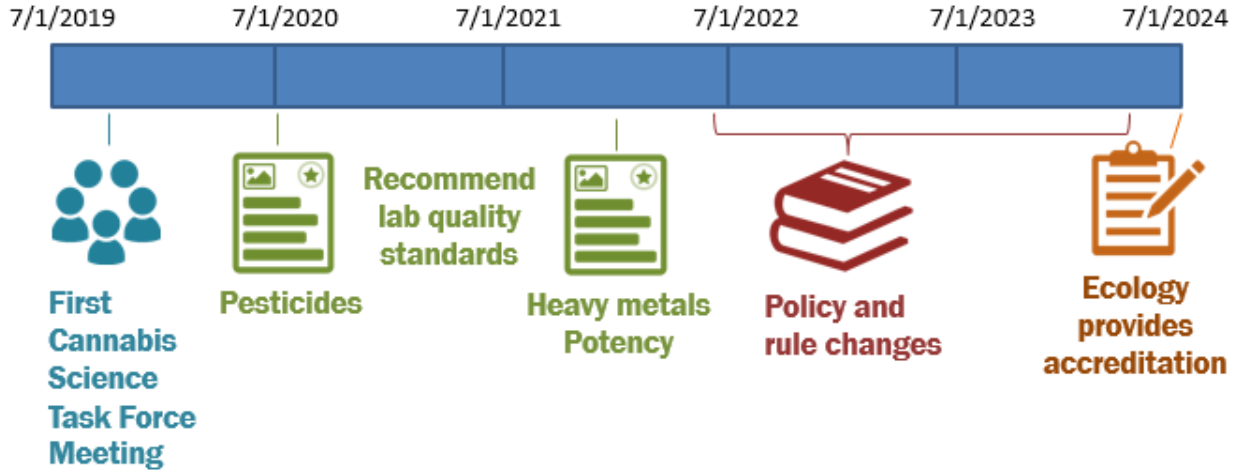

Figure 2. Timeline for Task Force deliverables, policy and rule updates, and transfer of cannabis laboratory accreditation to Ecology.

The goal of the Task Force recommendations is to provide a science-based framework for the testing laboratories to operate effectively and to provide the appropriate information for accreditation to adequately determine whether a laboratory has the capability to provide accurate and defensible data; together building stronger consumer protections. The report will detail recommendations for testing pesticides in cannabis plants as required in RCW 43.21A.735:

1. Appropriate approved testing methods
2. Method validation protocols
3. Method performance criteria
4. Sampling and homogenization protocols
5. Proficiency testing
6. Regulatory updates

This report will include additional recommendations for compliant intermediate cannabis products.

The Need for Appropriate Laboratory Quality Standards

Ecology's 2019 Cannabis Laboratory Accreditation Recommendations report (Sekerak, 2019) found that current laboratory quality standards were insufficient and lacking in some critical items necessary for a meaningful regulatory testing program. Many of the insufficiencies exist because of conflict between state and federal cannabis laws and the resulting lack of federal oversight for standardized cannabis testing practices. This problem is not unique to Washington. Many states that have legalized recreational or medicinal-use cannabis are struggling with this problem. As a result, the requirements for testing practices vary greatly by state, and each state has struggled to define its own quality standards.

In comparison, federal agencies, such as the United States Environmental Protection Agency (EPA) and Department of Agriculture (USDA), provide a framework for states to use in their regulatory environmental, health and agricultural testing programs. This framework includes an extensive anthology of peer-reviewed analytical methods, validation protocols, quality assurance and quality control practices, and project planning and sampling guides. This federal framework does not exist for state cannabis testing programs. For this reason, Ecology recommended

establishing a Cannabis Science Task Force (Task Force) to aid in the design of a robust and comprehensive cannabis testing program at the state level.

Laboratory quality standards are the elements used in the evaluation of a product's compliance with established product standards. They consist of approved methods, method validation protocols, and performance measures and criteria applied to the testing of the product. Establishing appropriate and well-defined laboratory quality standards is essential to communicate to the testing laboratories what standardized practices and procedures are appropriate.

Laboratory quality standards help ensure the data that laboratories generate are credible and can be used to provide consumer protections. They should represent sound scientific protocols, and detail practical and specific guidance for the testing subject matter. Well-defined laboratory quality standards provide accreditation with the critical elements to assess the competence and integrity of a laboratory. Together, well-established product standards, laboratory quality standards, and accreditation standards should function to garner confidence for consumers and the industry they support (Figure 3).

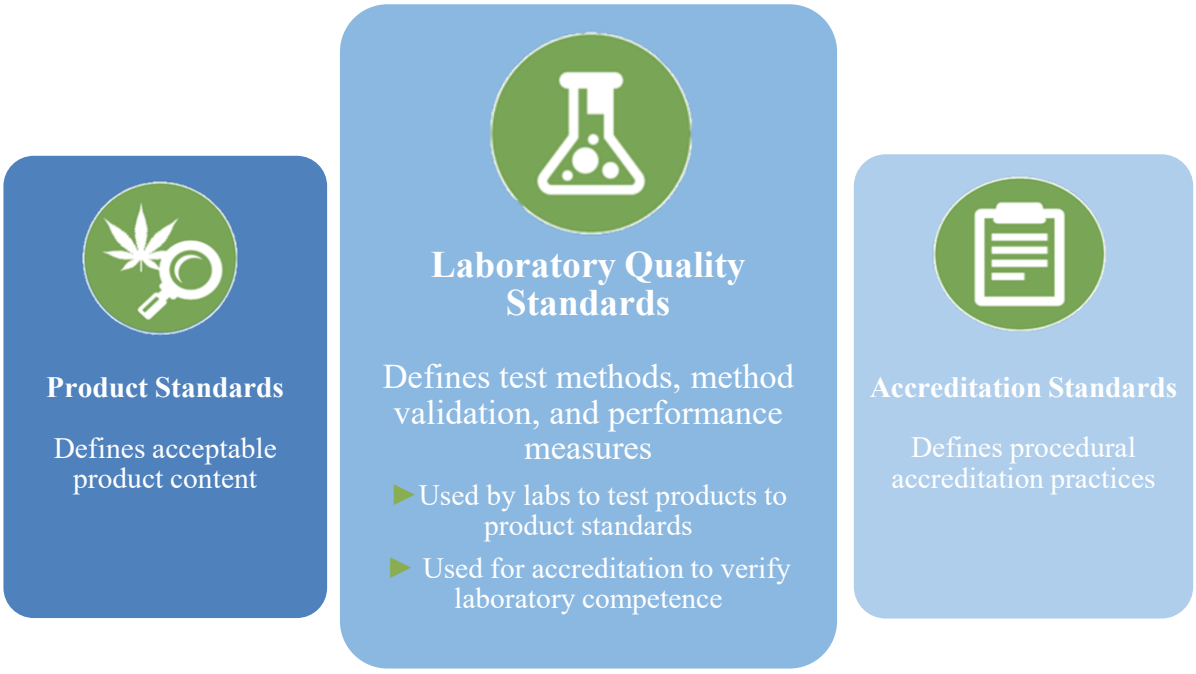

Figure 3. Product standards, laboratory quality standards, and accreditation standards.

Standards and the Current Authority

Current laboratory quality standards outlined in Chapter 314-55 WAC fall under the jurisdiction of the WSLCB. The laboratory quality standards should clearly detail approved methods, method validation protocols, and performance criteria, which are applied to the testing of the products. Current rule is insufficient in these areas. The standards must ensure appropriate information is generated and that the data are useful and of high enough quality to inform decision-making. Laboratory quality standards must be sufficient to provide the critical elements necessary for a robust accreditation. Under RCW 43.21A.736, the Task Force is charged with defining appropriate science-based laboratory quality standards. RCW 69.50.587 states "*the liquor and cannabis board may adopt rules that address the findings and recommendations in the task force reports".*

For cannabis testing laboratories, the WSLCB also holds the authority under Chapter 69.50 RCW to establish accreditation standards and execute laboratory accreditation activities themselves or through a third-party accreditation provider. Presently, the WSLCB uses a third-party contractor, the RJ Lee Group, to serve as its accreditation provider. Accreditation standards include elements such as defined regulatory authority (i.e., to grant, deny, suspend, and revoke accreditation), the accreditation certification cycle (e.g., 1-year period), on-site audit frequencies, application process, fee structure, and other procedural specifics. As provided in RCW 69.50.348, Ecology will assume this authority by July 1, 2024.

The WSLCB and Department of Health (DOH) share the authority under Chapter 69.50 RCW for establishing product standards. Product standards are the regulatory requirements designed to ensure compliant products have specified compositions and are free of specified contaminants. Current cannabis product standards include potency levels, pesticides limits, mycotoxin limits, and packaging requirements. The Task Force is not charged with making recommendations to product standards; however, some recommendations may be necessary to reinforce a more robust testing program.

Formation of the Cannabis Science Task Force

RCW 43.21A.735 established a Cannabis Science Task Force (Task Force) consisting of Agency appointees from the Departments of Ecology (Ecology), Agriculture (WSDA), Health (DOH), and the Liquor and Cannabis Board (WSLCB). The Task Force was designed to include a Steering Committee and two science-focused workgroups: the Laboratory Quality Standards (Analytical) workgroup led by the WSDA and the Proficiency Testing (PT) workgroup led by Ecology. Involvement and participation from cannabis industry scientists is an integral part of the design to succeed. Tribal and industry scientists were also invited to participate via emails distributed to the certified cannabis laboratories and through notification on Ecology's webpage.

Steering Committee

The Steering Committee is composed of Agency designated appointees and three members from certified cannabis laboratories selected by the Agency appointees. Additional members include non-voting chemists from the WSDA, WSLCB, and Ecology.

Steering Committee

- Annette Hoffmann Ph.D., Ecology, Environmental Assessment Program (EAP) Manager, and Committee Chair
- Jessica Archer, Ecology, EAP Section Manager
- Shelly Rowden, DOH
- Brad White, WSDA
- Kendra Hodgson, WSLCB
- Amber Wise, Medicine Creek Analytics, representing the Puyallup Tribe of Indians
- Nick Mosely, Confidence Analytics
- Jeff Doughty, Capitol Analysis Group
- Sara Sekerak, Ecology, Lead Task Force Chemist, and PT Workgroup Lead (non-voting member)
- Mike Firman, WSDA chemist and Analytical Workgroup Lead (non-voting member)
- Nicholas Poolman, WSLCB chemist (non-voting member)

The first Task Force Steering Committee meeting took place on August 21, 2019 to introduce selected members and present the Task Force objectives. Ongoing Steering Committee meetings continue to be held monthly and are open to the public. Dates, times, locations, and agendas are posted to Ecology's EzView webpage[6] prior to each public meeting. Following each meeting, all presentation materials and the WebEx recordings are also posted to Ecology's EzView webpage.

[6] https://www.ezview.wa.gov/site/alias__1962/37551/cannabis_science_task_force.aspx

Task Force Charter

The Task Force charter describes goals, members, and processes necessary to conduct the Task Force business. The Steering Committee adopted the final charter on October 18, 2019[7].

Workgroups

Analytical Workgroup

RCW 43.21A.735 directs that the first Task Force Legislative report contains recommendations for laboratory quality standards for pesticides in cannabis plants. The Task Force designees from Ecology, the WSLCB, DOH, and WSDA solicited for chemists with the appropriate expertise for the Analytical Workgroup. They targeted chemists with experience in pesticide analysis and analysis of agricultural commodities or food products. The agency designees selected members based on questionnaire responses sent to cannabis testing labs and other cannabis industry individuals that participated in Ecology's 2019 report. Responding individuals working as pesticide chemists in WSLCB certified cannabis testing labs were selected because of their relevant pesticide experience and knowledge of the challenges of testing cannabis in the current system.

Additional chemists from the DOH, WSDA, WSLCB, and Ecology serve as members in this workgroup. These chemists bring experience in both performing pesticide analyses and in working with analytical methods that generate data to support current regulation in Washington.

The workgroup meets bi-monthly. A summary of the workgroup meetings proceedings is presented in Appendix C.

Contributing Members

- Mike Firman, WSDA, Chemical and Hop Lab Manager/Chemist and workgroup lead
- Ben Hart, Testing Technologies, chemist
- Julie Kowalski, Trace Analytics, chemist
- Kyle Shelton, Medicine Creek Analytics, chemist
- Matthew Hall, Praxis, chemist
- Tania Sasaki, Confidence Analytics, chemist
- Nicholas Poolman, WSLCB, chemist
- Caroline West and Steve Officer, DOH, chemists
- John Weakland, Ecology, Organic Chemistry Supervisor/chemist
- Sara Sekerak, Ecology, chemist

Proficiency Testing Workgroup

Proficiency testing (PT) serves as a widely-accepted and necessary tool to test a laboratory's capability to produce accurate and defensible data in regulatory testing programs. Proficiency testing serves as a critical element of accreditation. While PT samples are readily available for

[7] https://www.ezview.wa.gov/site/alias__1962/37551/cannabis_science_task_force.aspx

most state and federal regulatory testing programs, the availability of appropriate cannabis PT samples is limited or non-existent due to the federal legal status and prohibition on interstate transport of cannabis products.

The Task Force designees from Ecology, WSLCB, DOH, and WSDA formed its second workgroup to develop recommendations around enhancing cannabis proficiency testing within Washington State. The agency designees selected workgroup members from the same pool responding to the questionnaire used to select Analytical Workgroup members and Task Force members. They selected members with a wider base of experience and expertise, as proficiency testing is a required practice for all testing practices.

The Task Force will provide recommendations from this workgroup in the second required report to the Legislature. The PT Workgroup will leverage off the highly specialized expertise of the Analytical Workgroup as that group works through all aspects of testing pesticides, and the future focus areas covering potency, metals, and other testing. A well-designed PT program would need to consider both specific analytical practices and overarching program attributes. The second report to the Legislature will include the full recommendations from PT Workgroup.

Contributing Members

- Sara Sekerak, Ecology, chemist
- Taber Salewsky, Praxis, chemist
- Steven Loague, Integrity Labs, chemist
- James Burns, Treeline, Lab Director
- Nicholas Poolman, WSLCB, chemist
- Steve LaCroix, DOH, Quality Assurance Officer/microbiologist
- Rebecca Wood, Ecology, Lab Accreditation Unit Supervisor/chemist

Recommendations

To strengthen Washington's laboratory quality standards for pesticides in cannabis plants (flower) and cannabis intermediate products, the Cannabis Science Task Force (Task Force) concluded that several actions are necessary. Recognizing that RCW 69.50.587 states the liquor and cannabis board may adopt rules that address the findings and recommendations in the task force reports provided under RCW 43.21A.735, the Task Force recommends the following actions:

1. **The appropriate regulatory authorities adopt the Task Force proposals as laboratory quality standards for pesticides in cannabis:**

   i. **The WSLCB adopts appropriate approved testing methods:** No single analytical method is required for testing pesticides. The laboratory may select any preparation method, instrument, and determinative method under a performance-based methods approach. The selected methods and instrument then must be validated according to the established method validation requirements and meet the required method performance criteria measures.

   ii. **The WSLCB adopts method validation protocols, and method performance criteria:** The (Task Force) Summary of Adaptations to the Pesticide Data Program (PDP) Model Standard Operating Procedure (SOP) and the five United States Department of Agriculture (USDA) PDP SOPs should be required for method validation protocols and method performance criteria for use in testing pesticides in cannabis (Appendices A, B, F).

   iii. **The WSLCB adopts sampling and homogenization protocols:** No single method is required. The laboratory may select any preparation method or analytical method that contains sampling and homogenization protocols. Protocols described in the PDP SOPs cover aspects regarding validation protocols and method performance criteria processes for in-laboratory sampling and homogenization.

   iv. **Ecology sets guidance for proficiency testing:** Ecology incorporates language into its accreditation rule and guidance, as necessary, pertaining to proficiency testing.

2. **WSLCB, WSDA, and DOH forms an interagency cooperative team, or *Client*, that holds the authority and expertise to facilitate and maintain the adopted Laboratory Quality Standards.**

   As of June 2020, a group of agency representatives has held meetings, and will continue to meet, to determine the regulatory authority and appropriations needed to form this interagency team. It may be necessary for agency request legislation to be put forth to align agency authorities. The agency representatives are evaluating the scope of work as well as staffing roles and responsibilities of the Client.

   The Client members coordinate rule modifications for their respective agencies. The Client should hold scientific expertise in chemistry and microbiology, food and agricultural testing, pesticide testing and other laboratory testing practices. The Client role provides program oversight by assuming and maintaining all responsibilities of the USDA (and EPA) described within the adopted PDP (Appendices A and B).

   Client tasks include:

i. Combining the (Task Force) *Summary of Adaptations to the PDP Model SOPs* document and the five USDA PDP SOPs (Appendices A and B) into a client-written SOP or manual to facilitate ease of use by the laboratories and accreditation provider by July 1, 2022.

ii. Provide timely and appropriate technical assistance to certified laboratories for the adopted laboratory quality standards facilitated by the use of the PDP documents (Appendices A and B).

iii. Ensure data generated under the PDP documents are appropriate and of high enough quality to support the intended WSLCB, or other established, regulatory use.

iv. Use sound and relevant science when making future modifications and updates to the adopted laboratory quality standards and supporting PDP documents (Appendices A and B).

3. Sequential regulatory updates performed by the WSLCB, DOH, and Ecology:

i. The WSLCB makes timely regulatory updates for the adoption and implementation of the recommended laboratory quality standards by testing laboratories. This may be done without delay, as earlier implementation of the laboratory quality standards will benefit both the labs and the current accreditation provider. This step could be achieved by revising Chapter 314-55 Washington Administrative Code (WAC) to incorporate the Task Force recommendations, or by establishing the laboratory quality standards outside of rule. However, the current language requiring laboratories to follow the *Cannabis Inflorescence and Leaf Monograph* published by the *American Herbal Pharmacopoeia (AHP)* should be removed from WAC 314-55-0995(3)(b), at a minimum. The AHP document does not constitute an adequate set of laboratory quality standards for cannabis testing laboratories and accreditation. Regulatory updates must be completed **by July 1, 2022**. This timing is essential in order to provide Ecology with the critical elements necessary to amend Chapter 173-50 WAC for cannabis testing laboratory accreditation. Well-defined promulgated rule or established guidance will enable Ecology to make the most appropriate updates to Chapter 173-50 WAC for accrediting cannabis laboratories, including, but not limited to fees and fee structure.

ii. Simultaneously to WSLCB rulemaking, the DOH amends Chapter 246-70 WAC, as appropriate, to compliment, harmonize, or clarify rule updates performed by the WSLCB. Revisions to this WAC must be completed **by July 1, 2022.**

iii. B**y July 1, 2024,** WSLCB amends Chapter 314-55 WAC to remove all quality assurance and quality control references to accreditation or "certification" practices to facilitate the transfer of cannabis testing laboratory accreditation to Ecology. Business, operational, or licensing requirements for cannabis testing laboratories will remain under WSLCB rule.

iv. Ecology amends Chapter 173-50 WAC to include, at a minimum, the accreditation fee structure for cannabis testing laboratories by **July 1, 2024**. Ecology will make updates to its Laboratory Accreditation Procedural Manual (2010) to include cannabis-specific accreditation and procedural practices as necessary.

Discussion of Recommendations

Recommended Laboratory Quality Standards

Appropriate Approved Testing Methods

Currently, there are a wide variety of pesticide methods and practices used for testing pesticides in cannabis. Most of these methods are adapted versions from widely accepted agricultural or food testing methodologies. The Task Force Analytical Workgroup discussed many of these methods but did not identify a single pesticide method that was superior. It was decided that requiring the use of one specific method might limit the flexibility of testing if regulatory requirements changed (e.g., adding new priority pesticides or lowering the thresholds).

As a means for the testing program to remain relevant into the future, it was concluded that no single method should be required for testing pesticides in cannabis. The recommendation was to instead implement a performance-based methods practice. Under this approach, no specific preparation method, instrument, or detection method would be required. Rather, each laboratory can select their own preparation and analytical methods (and instrument) as an analytical practice for testing pesticides. To prove the performance of the methods is acceptable, each method would require meeting the established regulatory method validation requirements, and implementing the required method performance measures.

Method Validation Protocols and Method Performance Criteria

The USDA Pesticide Data Program (PDP) model was selected as the basis for the guidance on method validation and method performance criteria for pesticides in cannabis. The USDA PDP model employs the performance-based method concept specifically for agricultural testing of pesticide residues on agricultural commodities. The USDA program relies on an established set of standard operating procedures (SOPs) for use in the collection of samples and performance of analytical determinations. The framework and attributes of the agricultural-based USDA PDP model seemed a fitting choice for developing guidance for pesticides in cannabis flower. The framework would also be easily adaptable for other cannabis product.

The scientists in the Task Force Analytical Workgroup carefully assessed the USDA PDP SOPs for appropriateness and applicability. The workgroup then made recommendations to the Task Force Steering Committee on adaptations of the SOPs based on their critical review and deliberation. Adaptions to the USDA PDP SOPs are necessary to address cannabis specific facets, current rule requirements, and to remove reference to USDA-specific roles and responsibilities. Most notably, the removal of the USDA roles and responsibilities heightened the need to define an appropriate "Client" for cannabis testing.

To replace the USDA as the Client, the new Client must possess expertise in pesticide testing practices and protocols, and authority to update, modify, and provide guidance on appropriate use of these practices. Due to the structure of the PDP model, the Client must be able provide prompt technical assistance and direction to the cannabis laboratories performing their daily work under the PDP documents. A summary of adaptations document details all the changes to the USDA PDP SOPs (Appendix B). Together the Summary of Adaptations to the PDP Model SOPS and PDP SOPs define the method validation protocols and method performance criteria.

The USDA PDP SOPs supporting the Task Force PDP model include:

- PDP-QC - Chemical Compounds, PDP Commodity Groupings, Method Validation and Quality Control (Rev. 9, 09/01/19)
- PDP-LABOP - Sample Processing and Analysis (Rev. 10, 07/01/18)
- PDP-DATA - Data and Instrumentation (Rev. 6, 04/01/18)
- PDP-ADMIN - Administrative Procedures for the Pesticide Data Program (Rev. 7, 07/01/2019)
- PDP Glossary - Abbreviations and Terms used in SOPs (Rev. 10, 01/01/1)

Sampling and Homogenization Protocols

No additional recommendation was needed to address sampling and homogenization protocols. Under the recommended performance-based method approach, labs will be able to select and validate any method or methods. In-lab sampling (sub-subsampling) and homogenization protocols may be incorporated in the selected determinative method or are contained in a required complementary preparation method. Protocols described in the PDP SOPs cover aspects of validation and performance measures, including those regarding sampling and homogenization processes defined by the method(s) selected. Adoption of the performance-based methods approach, the *Summary of Adaptations to the PDP Model SOPs*, and PDP SOPs will programmatically address this component[8].

Proficiency Testing

Proficiency test (PT) evaluations are a process where a known sample (PT sample) is provided for analysis, but the chemical constituents and their respective concentrations are unknown to the laboratory performing the analysis. Accreditation uses PT evaluation results to establish and assess a laboratory's capability to produce accurate data through implementation of their laboratories methods.

The Analytical Workgroup recommended that PT samples that are "in-matrix" (e.g., marijuana flower with greater than 0.3% delta 9 tetrahydrocannabinol [Δ-9-THC]) would be the most representative to test laboratory capabilities. For cannabis flower PT samples, flower that contains concentrations of pesticides that represent natural plant growth application, termed as "incurred" is preferred. PT samples for flower that are "spiked" (added to after harvest) with pesticides may be used when incurred PTs are unavailable.

Implementing the in-matrix recommendation would be a challenge, as the federal illegal status of marijuana currently prevents PT providers from producing and shipping the THC-containing cannabis PTs across state line. Additionally, no PT providers currently operate in Washington State. The Task Force's Proficiency Testing Workgroup is presently researching the current PT sample and PT evaluation program challenges. Recommendations to improve proficiency testing within Washington State will be summarized in the second report due to the Legislature by December 2021.

[8] Further discussion on requirements for samples submitted to the laboratory is in Appendices C and D. The Task Force adopted a two-sample requirement, as shown in Appendix E (11/15/2019, Motion #2).

Regulatory Updates

Regulatory updates for the adoption and implementation of laboratory quality standards necessitate revisions by the WSLCB to Chapter 314-55 WAC to incorporate the Task Force recommendations effectively. Established laboratory quality standards dictate the mandatory elements that the laboratories must use, follow, and meet to generate the necessary data to use for the intended regulatory purposes. Accreditation also relies on established standards to ensure the laboratories' analytical performance capabilities and that the laboratories are meeting the requirements of the Client. Weak standards, or no standards, could lead to meaningless accreditations, as well as unusable, questionable, or low quality data that is not fit for decisions regarding enforcement or consumer protections.

Specifically, it is also recommended that the WSLCB amend WAC 314-55-0995(3)(b) to remove the requirement that laboratories must follow analytical requirements in the most current version of the *Cannabis Inflorescence and Leaf Monograph* published by the *American Herbal Pharmacopoeia*. The *Cannabis Inflorescence and Leaf Monograph* (Upton et al., 2014) is not a peer-reviewed, validated analytical method or compendium of said methods. It does not explicitly detail analytical methods, require the use of any one validated method, or provide comprehensive analytical requirements to guide quality testing practices. Rather, the *Cannabis Inflorescence and Leaf Monograph* delivers conflicting information and practices that do not support current rule and appropriate laboratory-implemented testing practices.

Ecology's Environmental Laboratory Accreditation Program currently employs in-matrix proficiency testing as conventional requirement for accreditation. Rule updates to Ecology's Chapter 173-50 WAC are not anticipated as necessary to facilitate the Task Force cannabis in-matrix recommendation when Ecology becomes the accreditation authority on July 1, 2024. Ecology should update its *Procedural Manual for the Environmental Laboratory Accreditation Program* (Ecology, 2010) to include cannabis-specific accreditation and procedural practices as necessary.

By July 1, 2024, the WSLCB will amend Chapter 314-55 WAC to remove all references to accreditation to facilitate the transfer of cannabis testing laboratory accreditation to Ecology. Likewise, Ecology will adopt the necessary rule updates to incorporate cannabis testing laboratory accreditation under Chapter 173-50 WAC by July 1, 2024.

Defining the Client

In order to leverage the USDA's laboratory quality standards for pesticides in plants (USDA PDP), the Washington state Departments of Agriculture (WSDA), Health (DOH), and the Liquor Control Board (WSLCB) have agreed to work together to form an interagency cooperative team to serve as the *Client*. To match the USDA's Client role, chemists and other scientists with scientific backgrounds and expertise would need to serve as prominent Client entities. The Client is responsible to ensure that scientifically sound practices are required and adhered to, and that the quality and the level of uncertainty of the data produced from those practices is appropriate when used for enforcement or other Client-defined purposes, such as risk assessments. Because of the legal status of cannabis, a cooperative of Washington State agencies need to fill this role. Currently, members from the WSLCB, DOH, and WSDA are meeting to determine authority and funding needed to form the Client, by further defining the scope of work as well as staffing roles

and responsibilities of the Client. The agency members will determine if agency request legislation is needed to expand existing statutory authority.

For Washington State, the Client would ensure that respective agencies establish rules that are appropriate and meaningful when coupled with analytical practices, for example clearly defining the required pesticide isomers to be tested and updating action levels to contain appropriate significant figures. The Client should be designed with the capacity to serve in a technical oversight role to provide prompt assistance to testing laboratories implementing established laboratory quality standards. Specifically, for laboratory quality standards for the analysis of pesticides in cannabis flower and intermediate cannabis products, the client role is necessary to facilitate adoption and intent of the Client-established version of *Summary of Adaptations to the PDP Model SOPs* and the five USDA PDP SOPs. For the Client to function optimally, the Client ultimately should assume all roles and functions described in the USDA PDP currently performed by the USDA, including providing technical assistance and amending the PDP model documents with additional laboratory quality standard attributes. Further discussion on the client responsibilities necessary to support the PDP SOP model is detailed in Appendix D.

The Client role is critical to facilitate and maintain the use of the *Summary of Adaptations to the PDP Model SOPs* (Appendix A) and the accompanying laboratory quality standards adopted for pesticides, at a minimum. For Ecology's cannabis accreditation to be successful, it is necessary for that the Client role to be established and functioning by July 1, 2022 for Ecology to begin its accreditation rulemaking.

Conclusion

The recommendations of the Cannabis Science Task Force fulfill the intent of HB 2052 and present a pathway for establishing, implementing and maintaining critical laboratory quality standards for testing pesticides in cannabis plants and products in Washington State.

The Cannabis Science Task Force developed its recommendations by leveraging and adapting method validation protocols and method performance measures originally established by the United States Department of Agriculture. The Task Force recognized that adoption of adequate and up-to-date testing protocols is imperative to ensure these laboratories can operate effectively and are critical for accreditation to adequately determine whether a laboratory is capable of providing accurate and defensible data.

To maintain the adopted protocols and to provide technical assistance to the cannabis laboratories, the Task Force recommends establishing an interagency cooperative team. The team would be staffed by the Department of Health, the Department of Agriculture, and the Liquor and Cannabis Board. The current Task Force representatives from the respective agencies are meeting to determine necessary appropriations and regulatory authority for this interagency cooperative team. In December 2021, a second Task Force report will deliver additional recommendations for laboratory quality standards for potency and heavy metals, and provide a pathway for a more robust proficiency testing program.

Definitions

Accreditation (WAC 173-50 definition) – The formal recognition by the department [Ecology] that an environmental laboratory is capable of producing accurate and defensible analytical data. This recognition is signified by the issuance of a written certificate, accompanied by a scope of accreditation indicating the parameters for which the laboratory is accredited. The term "accredit" as used in this chapter is intended to have the same meaning as the term "certify" as used in RCW 43.21A.230.

Accreditation Standards (as used within this report) – Established criteria that describe the accreditation evaluation process to ensure accredited laboratories have the demonstrated capability to provide accurate, defensible data. Accreditation standards include descriptions of authority (i.e., granting, denying, suspending, and revoking accreditation), accreditation certification cycle length (e.g., 1-year period), on-site audit frequencies, application process, fee structure, and other procedural specifics of the accreditation process. More specifically, the accreditation standard may identify critical items (e.g., appropriate implementation and use of methods and standard operating procedures, use of quality control samples, and passing proficiency testing sample results) that will be assessed or evaluated as a part of the accreditation process.

Analytical method – A procedure consisting of several laboratory procedures, which when completed, produces a quantitative and/or qualitative result for the tested substance.

Blank matrix – A matrix that does not produce an analytical response by the analytical method under investigation for the analytes(s) of interest (USDA, 2015).

Client (as used within this report) – A regulatory agency identified entity housing personnel with authority and expertise to adopt and establish rule (or guidance) for laboratory quality standards based on sound science practices. The entity additionally serves to establish, maintain, and provide technical assistance for adopted laboratory quality standards.

Commodity grouping (as used in the USDA PDP SOPs): PDP commodity groups established to facilitate method evaluation. Grouping is based on EPA commodity grouping under 40 CFR 180, with modifications to further combine those commodities having similar matrix characteristics for analytical purposes (USDA, 2015).

In-lab sampling or sub-sampling is a procedure by which a small, representative sample is taken from a larger sample.

Laboratory Quality Standards (as used within this report) – Established criteria designed to produce accurate and reproducible data. Deliberate and intentionally designed laboratory quality standards ensure that established product standards can be met. In broad terms, laboratory quality standards are defined methods, method validation protocols, and performance criteria (e.g., use of quality control samples and their tolerance limits). These provide laboratories standardized requirements to follow, and also give accreditation providers critical elements to assess during the accreditation process.

Limit of quantitation (LOQ) – The smallest measured amount of analyte in a sample that can be reliably quantified with a specified degree of precision.

Matrix blank (as used in the USDA PDP SOPs) – Ideally, a previously characterized sample which shows no detectable or defined response for the analyte of interest within that analyte's chromatographic time segment (CTS). If a suitable sample is not available, a portion of one of the samples or purchased sample may be used (USDA, 2015).

Method validation – The process of demonstrating that an analytical method is suitable for its intended use. It involves conducting a variety of studies to evaluate method performance under defined conditions (EPA, 2006).

Performance-based methods approach – Conveys "what" needs to be accomplished, but not prescriptively "how" to do it. It is a measurement system based upon established performance criteria for accuracy and precision with use of analytical test methods. Under this measurement system, laboratories must demonstrate that a particular analytical test method is acceptable for demonstrating compliance. Performance-based method criteria may be published in regulations, technical guidance documents, permits, work plans, or enforcement orders.

Performance criteria – Defined, measurable performance characteristics of an analytical method or process-specific requirements for accuracy, precision, recovery, specificity (selectivity), sensitivity (limits of detection), inclusivity, exclusivity, linearity, range, and scope of application. Criteria may also be set by defining process (i.e., method validation protocols).

Pesticide (as used by the USDA and referenced in the USDA PDP***)*** **–** Means (1) any substance or mixture of substances intended for preventing, destroying, repelling, or mitigating any pest, (2) any substance or mixture of substances intended for use as a plant regulator, defoliant, or desiccant, and (3) any nitrogen stabilizer, except that the term "pesticide" shall not include any article that is a "new animal drug" within the meaning of section 321(w) [1] of title 21, that has been determined by the Secretary of Health and Human Services not to be a new animal drug by a regulation establishing conditions of use for the article, or that is an animal feed within the meaning of section 321(x) [1] of title 21 bearing or containing a new animal drug. The term "pesticide" does not include liquid chemical sterilant products (including any sterilant or subordinate disinfectant claims on such products) for use on a critical or semi-critical device, as defined in section 321 of title 21. For purposes of the preceding sentence, the term "critical device" includes any device which is introduced directly into the human body, either into or in contact with the bloodstream or normally sterile areas of the body and the term "semi-critical device" includes any device which contacts intact mucous membranes but which does not ordinarily penetrate the blood barrier or otherwise enter normally sterile areas of the body (7 U.S.C. §136 et seq., 2012).

Pesticide (as specified in WAC 246-70-030(22) and WAC 314-55-010) **–** Means, but is not limited to: (a) Any substance or mixture of substances intended to prevent, destroy, control, repel, or mitigate any insect, rodent, snail, slug, fungus, weed, and any other form of plant or animal life or virus, except virus on or in a living person or other animal which is normally considered to be a pest; (b) any substance or mixture of substances intended to be used as a plant regulator, defoliant, or desiccant; and (c) any spray adjuvant. Pesticides include substances commonly referred to as herbicides, fungicides, insecticides, and cloning agents.

Proficiency testing sample (PT sample) – A sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within acceptance limits specified in the regulations. The qualitative and/or quantitative composition of the reference material is unknown to the laboratory at the time of the analysis (EPA, 2005).

Product standards (as used within this report) – Established regulatory requirements that products or materials that are produced for consumers must meet. Compliant products under these standards

are asserted to be safe, free from contaminants, and produced to a specified composition or dosage requirement. Current cannabis standards include potency levels, pesticides action limits, mycotoxin limits, packaging requirements, and others.

Quality assurance (QA) – An integrated system of management activities involving planning, implementation, documentation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client (EPA, 2001).

QA manual – A document describing the policies, organization, objectives, and specific QA and QC practices within a laboratory.

Quality control (QC) – The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality (EPA, 2001).

Quality system – The means by which an organization manages its quality aspects in a systematic, organized manner. It provides a framework for planning, implementing, and assessing work performed by an organization and for carrying out required QA/QC activities. It encompasses a variety of technical and administrative elements, including:

- policies and objectives
- organizational authority
- responsibilities
- accountability
- procedures and practices (EPA, 2002)

Sample – Representative portion of material taken from a larger quantity of homogenate for the purpose of examination or analysis, which can be used for judging the quality of a larger quantity.

Standard operating procedure (SOP) – A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps, and that is officially approved as the method for performing certain routine or repetitive tasks (EPA, 2001).

Validated methods – The methods that have undergone validation.

Validation (method) – The process of demonstrating or confirming the performance characteristics through assessments of data quality indicators for a method of analysis.

Frequently Used Acronyms

Task Force	Cannabis Science Task Force
DOH	Washington State Department of Health
Ecology	Washington State Department of Ecology
PDP	Pesticide Data Program
PT	Proficiency test
QA	Quality assurance
QAO	Quality Assurance Officer
QC	Quality control
SOP	Standard operating procedure
USDA	U.S. Department of Agriculture
WSDA	Washington State Department of Agriculture
WSLCB	Washington State Liquor and Cannabis Board

References

[AOAC] Association of Official Analytical Chemists, 2016. Appendix F: Guidelines for Standard Method Performance Requirements. AOAC International. http://www.eoma.aoac.org/app_f.pdf.

Ecology, 2010. Procedural Manual for the Environmental Laboratory Accreditation Program. Washington State Department of Ecology. https://fortress.wa.gov/ecy/publications/documents/1003048.pdf.

[EPA] United States Environmental Protection Agency, 2006. Validation and Peer Review of U.S. EPA Radiochemical Methods of Analysis. https://19january2017snapshot.epa.gov/sites/production/files/2014-05/documents/radiochemmethod_validity_guide.pdf.

[EPA] United States Environmental Protection Agency, 2001. EPA Requirements for Quality Management Plans. EPA QA/R-2. https://www.epa.gov/sites/production/files/2016-06/documents/r2-final.pdf.

[EPA] United States Environmental Protection Agency, 2002. Guidance for Developing Quality Systems for Environmental Programs (EPA QA/G1). Document EPA/240/R-02/008.

Sekerak, Sara, 2019. Cannabis Laboratory Accreditations Recommendations. Washington State Department of Ecology. https://fortress.wa.gov/ecy/publications/SummaryPages/1903004.html.

7 U.S.C. United States Code, 2012. Title 7 – Agriculture: Chapter 6 - Insecticides and Environmental Pesticide Control, Subchapter II - Environmental Pesticide Control, Sec. 136 – Definitions. U.S. Government Publishing Office. www.gpo.gov

[USDA] United States Department of Agriculture, 2015. PDP Glossary. Revision 10, effective 01/01/2015. (Appendix B).

[USDA] United States Department of Agriculture, 2015. PDP-Data: Data and Instrumentation. Revision 10, effective 04/01/2018. (Appendix B).

[USDA] United States Department of Agriculture, 2015. PDP-LABOP: Sample Processing and Analysis. Revision 10, effective 07/01/2018. (Appendix B).

[USDA] United States Department of Agriculture, 2015. PDP-QC: Chemical Compounds, PDP Commodity Groupings, Method Validation and Quality Control. Revision 9, effective 09/01/2019. (Appendix B).

Appendices

Appendix A. Summary of Adaptations

The following document was produced by the Cannabis Science Task Force and adopted by them on January 17, 2020.

Summary of Adaptation to the USDA PDP SOP Model Documents

For each model document the document name is listed first with version number then the general changes to the document followed by the specific changes to the document.

USDA PDP – QC SOP, Chemical Compounds, PDP Commodity Groupings, Method Validation and Quality Control (Rev. 9, 09/01/19)

Sections without comment are recommended as/is after adjustment for the general changes.

General Changes

References to "United States Department of Agriculture Agricultural Marketing Services, Science & Technology Pesticide Data Program", "USDA/AMS", "USDA PDP", "PDP" or other personnel/roles of the USDA shall be replaced by the "client".

The document refers to USDA forms and attachments to this PDP SOP. The client shall designate appropriate forms that captures the information contained in the USDA forms.

Specific Changes

5.1.2.2.1 Remove "scheduled for EPA Registration Review, as documented on the current EPA Office of Pesticide Programs Registration Review Schedule" and replace with "are the required lists of pesticides designated by the WSLCB and DOH."

5.1.2.3 Remove all section language and replace with "All methods must test priority 1 compounds. The client may designate other compounds as priority 1,2,3,4 compounds."

5.2.1.1 Remove section.

5.2.1.2 Add language: "Certified standards compliant with ISO Guide 34 should be used when available. All standards require a certificate of analysis."

5.2.3.5 Remove section.

5.2.4 "Labeled" includes "labeled by reference", where a lab may put a code on a small vial and have a document that has the required information that can be linked to the code on the vial. It does not mean that all the information has to be written on the vial." Add language: "A separate standard preparation area is not required if there are appropriate cleaning procedures and controls to ensure against cross contamination."

5.2.5 "Labeled" includes "labeled by reference", where a lab may put a code on a small vial and have a document that has the required information that can be linked to the code on the vial. It does not mean that all the information has to be written on the vial." Add language: "A separate standard preparation area is not required if there are appropriate cleaning procedures and controls to ensure against cross contamination."

5.3.1 Remove section language and replace with "All pesticide compounds designated as required by WSLCB and DOH are the marker compounds. Priority 1 compounds and marker compounds shall be the same list of compounds."

5.3.2.2 Remove section language.

5.4.1 Remove “PDP laboratories” and replace with “certified laboratories”.

5.6 Remove “If local agreement cannot be reached the PDP Technical Director shall be contacted to determine which modules should be performed”.

5.6.2.3 Remove “*If local agreement cannot be reached, the MPD Director shall be contacted for further resolution”* and replace with “the client shall be notified of all instrument changes”.

5.6.2.4 Remove *“If local agreement cannot be reached, the MPD Director shall be contacted for further resolution”* and replace with “the client shall be notified of all modifications to existing methods”.

5.7 Add language: “Marker Pesticide compounds are the required pesticides in lists established and maintained by the WSLCB and DOH.”

5.7.1.1 Remove “Technical Advisory Group (TAG)” and replace with “the client”.

5.7.2.1 Remove section language and replace with “The laboratory must spike all compounds for each commodity group.”.

5.7.2.2. Add language to beginning of section “Upon client approval, or if directed by the client, certified laboratories may employ the following:”.

5.7.2.3 Add language to beginning of section “Upon client approval, or if directed by the client, certified laboratories may employ the following:”.

5.7.2.4 Add language to beginning of section “Upon client approval, or if directed by the client, certified laboratories may employ the following:”.

5.7.3 Add language to beginning of section “Upon client approval, or if directed by the client, certified laboratories may employ the following:”.

5.7.4 Add language to beginning of section “Upon client approval, or if directed by the client, certified laboratories may employ the following:”.

5.9 Add “Cannabis Flower”

5.9.1 Remove “PDP Technical Director” and replace with “client”.

5.9.2 Remove section.

5.13.6 Remove “USDA/AMS expects any coding changes for calendar year samples to be submitted by May 31st following the end of the calendar year. This does not remove the requirement to report all data sets for the calendar year by March 31st of the following calendar year”.

5.15.3.1 Remove “PDP Technical Director with copies to the Method Validation Coordinator and the assigned liaison chemist” and replace with “client”

5.15.4 Remove “PDP Technical Director with copies to the Method Validation Coordinator and the assigned liaison chemist” and replace with “client”

Remove “(USDA/AMS PDP, 1400 Independence Ave, S.W> Washington DC 20250 or fax [(202) 619-1724].”

5.16.2.1 Remove “PDP Technical Director” and replace with “client”.

Remove “Details of this review process are specified in the SOP PDP-ADMIN”.

5.16.2.5 Remove “Technical Director” and replace with “client”.

5.17.4.2 Remove “normal RDE procedures” and replace with “client required data reporting procedures”.

5.17.6.3 Remove “RDE (the preferred option)” and replace with “client required data reporting procedures”.

5.17.8 Remove section language and replace with "QA codes shall include those defined by the client".

5.18.1.1, 5.18.2.1, 5.18.4.1 Replace all instances of "50-150%" with "70-130%".

5.20 Remove section language and replace with:

> "Certified labs are responsible for evaluating measurement uncertainty using appropriate practices and protocols. Appropriate guides and resources for evaluating measurement uncertainty include: The Joint Committee Guides in Metrology "Evaluation of measurement data – Guide to the expression of uncertainty in measurement (GUM)", ISO/IEC Guide 98, or EURACHEM/CITAC Guide "Quantifying Uncertainty in Analytical Measurements. Additional methods for calculating measurement uncertainty may be utilized with the approval of the client."
>
> Certified labs shall submit reports to the client annually or on a schedule set by the client."

5.19 Remove section language and replace with "Proficiency Testing requirements set by client shall be followed.".

USDA PDP – LABOP, Sample Processing and Analysis (Rev. 10, 07/01/18)

Sections without comment are recommended as/is after adjustment for the general changes.

The USDA also has responsibility for sampling and oversees sampling. The client requirements for sampling process and protocol supersedes sampling the parts of the SOP that refer to the client taking sampling actions.

General Changes

References to "United States Department of Agriculture Agricultural Marketing Services, Science & Technology Pesticide Data Program", "USDA/AMS", "USDA PDP", "PDP" or other personnel/roles of the USDA shall be replaced by "client".

The document refers to USDA forms and attachments to this PDP SOP. The client shall designate appropriate forms that captures the information contained in the USDA forms.

Specific Changes

5.1 Remove table and replace with "Follow all sample traceability and sample transfer requirement established in Chapter 314-55 WAC".

5.1.1.1 Remove "RDE sample information" and replace with "sample receipt log or electronic records log."

5.1.1.2 Remove "refer to current Monitoring Programs Division (MPD) Commodity Fact Sheet). Ensure that lot numbers on all units are the same, unless a specific Commodity Fact Sheet allows multiple lots to achieve required weight. Check that required information (variety, lot numbers, etc.) that can be determined is recorded in the RDE sample information (if not already recorded by sampler), and that the information in RDE and sample identification match each other. This may be done either directly in RDE or noted on a printed Sample Information Form (SIF) and entered into RDE before or during reporting" and replace with "refer to Chapter 314-55 WAC sample requirements."

5.1.3 Remove "in the "Lab Comment" section of the RDE sample information. The laboratory shall contact the MPD Sampling Manager if there are questions as to the sample's viability' with "and reject the sample".

5.1.5 Remove "usually between one and seven pounds. The acceptable weight range is $\pm$ 20% of the target weight (e.g., for 5 lb. samples: 4-6 lbs). ***Note:*** *Determination of the weight of the sample being homogenized is optional; however, if the weight is determined, it shall be entered in the "Sample Size" field*

of the RDE sample information" and replace with "Samples shall be in adherence to requirements established in Chapter 314-55 WAC".

5.1.5.1 Remove section.

5.1.5.2 Remove section.

5.1.6 Remove "70%" and replace with "90% or 3.6g"

5.1.6.2 Remove "***Note:*** *Some commodities use lot numbers that include a time stamp. For example, if three jars are labeled 15502B1130, 15502B1132, and 15502B1133, the lot number is 15502B and the last four digits are the time stamp. Times should be within a three-hour window. Lot number formats differ widely among commodities and companies. Contact the client for guidance if there are questions regarding viability".*

5.1.7 Remove " in the "Reason NOT Analyzed" field of the RDE sample information."

5.1.8 Remove "or e SIFs"

5.1.8.1 Remove section.

5.1.8.2 Remove "a corresponding RDE SIF, the laboratory shall contact the appropriate State Sampling Manager within 24 hours and copy the MPD at amsmpo.date@ams.wsda.gov" and replace with "the Chapter 314-55 WAC required information the lab shall reject the sample."

5.1.8.3 Remove "If an eSIF contains an error that cannot be resolved with the Sampling State contact MPD at amsmpo.data@ams.usda.gov" and replace with "If the sample collection information contains and error that cannot be resolved the sample shall be rejected."

5.1.8.4 Remove section.

5.1.9 Remove "MPD" and replace with "client". Remove "monthly" and replace with "periodically upon client's defined schedule."

5.1.10 Remove section language and replace with "Forms and form information requirements shall be in accordance with those set by the client."

5.1.12 Remove section.

5.1.13 Remove "(unless documented on the SIF)"

5.2.1 Remove "PDP" and replace with "regulatory"

5.2.3 Remove "Fresh Fruits and vegetables" and replace with "cannabis samples".

5.3 Remove "Fresh Produce, Animal Tissue, Nuts, and Grains" and replace with "Cannabis Samples" Remove section language and table, then add:

"**For cannabis flower:**

The lab is to receive two samples. Each sample is representative of the whole batch. The lab determines at random what sample to test for pesticides and what sample to use for other tests. The selected sample is homogenized with a mechanical process until the sample is entirely homogeneous. The sample is homogenized prior to weighing and extraction. The hold time for cannabis flower is 72 hours prior to extraction."

5.4 Remove section language.

5.7.1 Remove "-40˚C" and replace with "-30˚C".

5.7.2 Add language: "For long-term storage, all portions of samples shall be stored at -30˚C"

5.7.3. Remove section.

5.7.4 Remove section.

USDA PDP – DATA, Data and Instrumentation (Rev. 6, 04/01/18)

Sections without comment are recommended as/is after adjustment for general changes

The USDA also has responsibility for sampling and oversees sampling. If the client does not have responsibility for sampling the parts of the SOP that refer to the client taking sampling actions will need to be removed.

General Changes

References to "United States Department of Agriculture Agricultural Marketing Services, Science & Technology Pesticide Data Program", "USDA/AMS", "USDA PDP", "PDP" or other personnel/roles of the USDA shall be replaced by the "client".

The document refers to USDA forms and attachments to this PDP SOP. The client shall designate appropriate forms that captures the information contained in the USDA forms.

This document has many references to "codes" used on reporting to specify additional information other than the amount found. The client should determine how this additional data should be reported and what "codes", if any, to use for reporting. Labs should still keep the information available and provide it on request.

Specific Changes

5.4.4 Remove "See SOP PDP-ADMIN for records storage and archival requirements" and replace with Records shall be stored for at least three years.

7.4.1.2 Remove "MPD" and replace with "client".

8.2.2 Remove "At a minimum, hardcopies of data sets shall include the following:" and replace with "At a minimum, hardcopies or locked, traceable and verifiable electronic copies of data sets shall include the following:"

Remove "PDP Sample Information Forms (SIFs) [if paper SIFs were submitted by the Sample Collector]" and replace with "Any documents submitted to the laboratory with the sample"

8.2.3 Remove "PDP Technical Director, Method Validation Coordinator, and liaison chemist (refer to SOP PDP-QC)" and replace with "the client".

9.4 Remove section.

9.5 Remove " PDP Tolerance Table" and replace with "Action Levels and Compounds Lists"

9.5.1 Remove all section language and replace with "WSLCB maintains the action levels for compounds (Chapter 314-55 WAC)".

9.5.2 Remove language and add "The DOH maintains a list of compounds to be tested for medical cannabis".

9.6 Remove section language and replace with "WSLCB and DOH action level limits shall be adhered to and applied."

9.7 Remove section language and replace with "WSLCB and DOH action level limits shall be adhered to and applied."

11 Remove "Remote Data Entry System" and replace with "Required Data Report System"

Remove all section language and add “the WSLCB reporting system shall be used for report data.”.

Add new section:

X.x. Routine Recovery Checks and Acceptance Limits

Acceptable limits for individual recovery results should normally be within the range of the mean recovery +/- 2x RSD. For each commodity group the mean recovery results and RSDs may be taken from initial method validation or from on-going recovery results (within laboratory reproducibility). A practical default range of 70-130 % may be used for individual recoveries in routine analysis. Recoveries outside the above mentioned range would normally require re-analysis of the batch, but the results may be acceptable in certain justified cases. For example, where the individual recovery is unacceptably high and no residues are detected, it is not necessary to re-analyze the samples to prove the absence of residues. However, consistently high recoveries or RSDs outside ± 20% must be investigated. Adapted in concept from SANTE/11813/2017, “Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed”, Section C44, November 2017 rev.0.

USDA/PDP Glossary, Abbreviations and Terms used in SOPs (Rev. 10, 01/01/15)

Document for reference- No changes/edits

Appendix B. USDA PDP Model Standard Operating Procedures

The referenced United Stated Department of Agriculture standard operating procedures are reproduced here, starting with the following page.

Appendix B

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

SOP No.: PDP-QC		Page 1 of 43
Title: Chemical Compounds, PDP Commodity Grouping, Method Validation and Quality Control		
Revision: 9	Replaces: 02/01//2018	Effective: 09/01/2019

1. Purpose:

To provide a reference of PDP required compounds, a listing of available marker pesticides and process controls, specification of PDP commodity groupings, requirements for method validation and continuing quality control (QC) for 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 that may impact the program.

3. Outline of Procedure:

Attachment 1 – Method Evaluation Flowchart
Attachment 2 – PDP Compound Groups, Pesticides Codes and Multi-residue Compound Groupings for Fruit and Vegetables
Attachment 3 – EPA, Codex, and Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) Commodity Groupings
Attachment 4 – FDA Information for percent fat, water, sugar and pH content for Commodity Groupings
Attachment 5 – Method Evaluation Reporting Forms [LOD Verification, Determination of Method Range, Precision and Accuracy Data Collection]
Attachment 6 – Process Control and Spike Recovery Acceptability Flowchart

4. References:

- de Kok et. al., *The Stability of Pesticide Standards and Solutions*, 5th European Pesticide Residue Workshop, Stockholm, Sweden, June 13-16, 2004
- Avramides, *The Stability of Pure Standards and Stock Standard Solutions for Pesticide Residue Determination Using Gas Chromatography*, 5th European Pesticide Residue Workshop, Stockholm, Sweden, June 13-16, 2004
- Vieth et. al, *Storage Stability of Stock Solutions and Solid Pesticide Standards*, 5th European Pesticide Residue Workshop, Stockholm, Sweden, June 13-16, 2004
- National Environmental Laboratory Accreditation Conference (NELAC), *Standards,* Appendix D, Section D.1.1.2.1, Laboratory Control Samples (LCS), June 5, 2003
- U.S. FDA, *Standard Operating Procedures for the Total Diet Study*, KCM TD G2, revision 0, Quality Assurance, January, 1993
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- U.S. FDA, *Pesticide Analytical Manual Volume I (**PAM**) 3rd Edition, C*hapter 2, https://www.fda.gov/media/74477/download
- U.S. FDA, *Approximate pH of Foods and Food products* http://www.webpal.org/SAFE/aaarecovery/2_food_storage/Processing/lacf-phs.htm
- Codex Alimentarius Commission, *Pesticide Residues in Food and Feed*, http://www.fao.org/fao-who-codexalimentarius/codex-texts/dbs/pestres/en/

5. 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.

5.1 Required Compounds

5.1.1 Refer to applicable commodity/compound-specific memoranda for commodity specific testing profiles.

5.1.2 Priority Levels

5.1.2.1 Each analyte of interest for each assigned commodity shall be designated with a priority level by the USDA/AMS. Priority levels for the individual compounds in the commodity-specific memoranda posted to the PDP Extranet are based on data needs identified by data users/stakeholders (e.g., U.S. Environmental Protection Agency, U.S. Food and Drug Administration, grower groups, industry, consumer/environmental groups), current tolerances and Action Levels (ALs), and national/international Maximum Residue Levels (MRLs). In addition, compounds that may not have tolerances in the U.S., but are known to be used in countries that export food to the U.S. are included; these compounds are comprised of compounds identified by EPA as having a high probability of consumption in selected imported products, and analytes identified by FDA or USDA Foreign Agricultural Service (FAS) as of interest in selected imported

products, where applicable to a given commodity. It is recognized that not all compounds/metabolites on a given list are amenable to multiresidue testing and final screening lists will be determined based on method validation and ongoing testing results.

5.1.2.2 In the various commodity-specific memoranda (separate documents posted to the PDP Extranet), compounds identified as Priority 1 compounds are the most critical and those identified as Priority 4 are the least critical. The priority level is a combination of data needs and expected feasibility of current methods to recover a given compound. General priority levels are assigned according to the following protocol:

5.1.2.2.1 Priority 1 compounds are selected multiresidue-amenable pyrethroids, organophosphates, and carbamates and their associated metabolites. Priority 1 compounds are required for all commodities. These compounds are critical because they are scheduled for EPA Registration Review, as documented on the current EPA Office of Pesticide Programs Registration Review Schedule.

5.1.2.2.2 Priority 2 compounds include other multiresidue-amenable compounds with a current tolerance for the given commodity that are highly important because they also have upcoming reviews scheduled or have been identified by a stakeholder as a highly important data need. Cyphenothrin, imiprothrin, and tetramethrin are also included as priority level 2 compounds for all commodities. Additionally, chemicals used in other countries may be included as Priority 2 compounds, dependent upon their anticipated method behavior.

5.1.2.2.3 Priority 3 compounds include other analytes with tolerances (including food handing establishment tolerances) or ALs (e.g., environmental contaminants/extraneous residues – aldrin, BHC, chlordane, DDD, DDE, DDT, dieldrin, endrin, heptachlor, and heptachlor epoxide) for the given commodity and are routinely analyzed by multiresidue methods. Priority 3 compounds may also include chemicals used in other countries, dependent upon their anticipated method behavior.

5.1.2.2.4 Priority 4 compounds include pesticides that have current tolerances, but likely require single analyte methods (e.g., glyphosate/AMPA, paraquat/diquat, EBDCs). Priority 4 compounds may also include chemicals used in other countries, dependent upon their anticipated method behavior.

5.1.2.3 Laboratories should include all Priority 1 compounds, as many Priority 2 compounds as possible, and as many Priority 3 compounds as feasible.

5.1.2.4 In some cases, PDP will authorize the development of new methods to detect certain compounds (e.g., triazole metabolites, phenoxies, formetanate hydrochloride).

5.2 Standards

5.2.1 Ordering Analytical Standards

5.2.1.1 Standards may be obtained from the EPA Repository, registrants, or commercial vendors. When requesting standards from the Repository, identify your laboratory as a PDP laboratory in the comment section of the order form so that the Repository staff will know that the order takes precedence. If the request is urgent, note that in the Comment section of the order form as well.

The EPA repository is located at:

EPA National Pesticide Standard Repository
Environmental Science Center
701 Mapes Road
Fort Meade, MD 20755-5350
Phone: (410) 305-2931
FAX: (410) 305-2999

https://www.epa.gov/pesticide-analytical-methods/national-pesticide-standard-repository

5.2.1.2 Procurement of standards from all sources must meet the following minimum requirements:

Availability of a current and valid “Certificate of Analysis” (CoA) (as a minimum requirement the certification shall identify the substance, its purity, and the production lot), traceability, and current expiration date.

An exemption for CoA and current expiration date is allowed for extraneous environmental contaminants that are covered by FDA Action Levels and compounds that have been revoked and no longer have existing U.S. registrations. Extraneous environmental contaminants include aldrin, BHC, chlordane, DDT (and metabolites), dieldrin, endrin, heptachlor (and metabolite), and lindane. Examples of revoked compounds that no longer have existing U.S. registrations include parathion ethyl, chlorfenvinphos, and fenchlorphos.

For all other analytical standards, in some cases, a current and valid CoA may not accompany the analytical standard. In this case, the laboratory shall contact the vendor to determine if one is available; if one is not available, the laboratory is exempt from the requirement to maintain a current and valid CoA for that standard.

5.2.2 Receipt of Analytical Standards

Custody of a standard begins when the standard is received in the laboratory. Each standard shall be given a code that uniquely identifies the standard from neat material to final dilutions. Receipt of standards shall be documented and each standard shall be traceable. Records shall include name, unique code, purity, lot number, date received, and expiration date (see 5.2.1.2 for exemption).

5.2.3 Storage of Analytical Standards

5.2.3.1 Neat standards shall be kept in a separate standards freezer, preferably at approximately -20°C or lower unless degradation occurs at such temperatures. In these cases, neat standards shall be stored at the recommended temperature.

5.2.3.2 Stock standards and dilutions including mixed standards shall be kept in refrigerators or freezers separate from those used for samples. Stock standards and dilutions shall be stored in teflon-lined, screw-capped, glass bottles or sealed glass ampules.

5.2.3.3 Access to the freezers and refrigerators shall be controlled and standards usage documented through the use of appropriate records (e.g., log books). These records shall contain at a minimum: standard name and/or unique code, date and time removed, initials of person removing standard, date and time returned, initials of person returning standard.

5.2.3.4 Refrigerator and freezer temperatures shall be checked either by taking readings each working day, or by automatic temperature recording devices.

5.2.3.5 When a neat standard is removed from freezer storage, the standard should be stored in a desiccator while it is brought to room temperature to minimize the potential for hydrolysis.

5.2.4 Preparation of Stock Standard Solutions

Stock standard solutions shall be prepared in a separate standard preparation area to avoid contamination of samples with pesticide standards. Each stock standard shall be given a unique identifying code and shall be labeled with a minimum of: pesticide name, concentration, solvent, date of preparation, initials of preparer, and expiration date of solution. Written SOPs for stock standard preparation shall include the method for preparing standards, calculations used in standard preparation, documentation that provides for standard traceability and safety guidelines.

5.2.5 Preparation of Intermediate Dilutions

Intermediate dilutions, including mixed standards, shall be prepared in a separate standard preparation area. Each standard shall be given a unique identifying code and shall be labeled with pesticide name, concentration, solvent, date of preparation, initials of preparer, and expiration date of solutions. Written SOPs shall include the method for standard preparation and documentation that provides for standard traceability.

5.2.6 Standard Checking

5.2.6.1 Stock solutions of neat pesticide standards not previously prepared or not currently in use in the laboratory shall be prepared in duplicate and the two standards compared to each other. Responses for standards of comparable concentrations must match within 15% relative percent difference (RPD):

$$RPD = \frac{|RF_1 - RF_2|}{\left[\frac{RF_1 + RF_2}{2}\right]} \times 100$$

where RF_1 is the response factor[1] of the first analytical standard and RF_2 is the response factor of the second standard. If standards do not match, potential sources of variation should be reviewed, and a third standard shall be made and compared. This process shall be continued until two matching standards are prepared.

5.2.6.2 New stock solutions that are prepared from neat pesticides currently used in the laboratory shall be compared to the old stock solution. The two standards must match within 15% RPD. 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.

5.2.6.3 Documentation of the standard checking process shall be kept through appropriate records (i.e. logs). Chromatograms of all standards shall be kept indicating the standard comparisons of old and new standards and the calculated difference.

5.2.7 Expired Standard Verification

If a laboratory has an expired neat analytical standard and cannot obtain a replacement with a valid expiration date from an approved PDP vendor or the EPA National Pesticide Standard Repository, with a deviation from USDA/AMS on file, the laboratory may proceed with validation and analysis of samples using the expired standard under the following conditions:

5.2.7.1 If the standard is recertified by the vendor and new documentation is obtained, it shall be recorded in the laboratory's standard records.

5.2.7.2 If the standard is not recertified, it shall be compared to an unexpired neat when one is available to verify its integrity.

[1] Area or height of each standard divided by the concentration of that standard.

5.2.7.2.1 If the two standards' response factors are within 15%, the expired standard being used shall be considered fit for purpose and this data shall be recorded in the laboratory's records.

5.2.7.2.2 If the two standards' response factors are not within 15%, USDA/AMS shall be contacted.

5.2.7.2.2.1 If there are residues, USDA/AMS and the laboratory's Technical Program Manager (TPM) and Quality Assurance Officer (QAO) shall develop an agreement on how to proceed with samples containing residues (e.g., re-extract and analyze with unexpired standard, code data as estimates, change to "unable to analyze," etc.). The agreement shall be documented and recorded in the laboratory's records. USDA/AMS will update any transmitted data in the USDA/AMS database.

5.2.7.2.2.2 If there are non-detects and the expired standard produces a response less than the response of the unexpired standard, the LOD shall be raised (consult with USDA/AMS to determine the level) and this information shall be recorded in the laboratory's records. USDA/AMS will update any transmitted data in the USDA/AMS database. The expired standard being used shall be considered fit for purpose for qualitative analysis only and this declaration shall be recorded in the laboratory's records.

5.2.8 Working Dilutions/Mixed Standards

5.2.8.1 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 maintained. 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.

5.2.8.2 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.

5.2.8.3 All working/mixed standards shall be identified by a unique and traceable code. Working/mixed standard records shall contain a minimum of pesticide name, solvent, date of preparation, expiration date, and preparer.

5.2.9 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-FPD, GC-ELCD, GC-XSD, GC-MSD, GC-ITD, LC-MS, and tandem MS. Libraries of all standards shall be developed for confirmatory instruments (GC-MS and LC-MS systems).

5.2.10 Disposal of Analytical Standards

Each laboratory shall establish the proper procedures for disposal (e.g., disposal by a licensed contractor) of expired analytical standards (both neat standards and dilutions). Disposal shall be in accordance with the laboratory's Chemical Hygiene Plan and shall be documented.

5.3 Method Validation Background

5.3.1 Marker compounds and commodity groups were created to facilitate the validation and ongoing QC of the enormous number of combinations of pesticides and commodities included in PDP. Each concept seeks to group pesticides or commodities by common properties and exploits these common properties to reduce the possible combinations to a manageable number.

5.3.2 This method evaluation framework makes the following assumptions:

5.3.2.1 Commodities are grouped in such a way that assessment of method performance in one commodity in the group can be extended to apply to all commodities in the group.

5.3.2.2 Marker pesticides are chosen to be representative of a broad range of similar pesticides. The assessment of method performance for these pesticides can be extended to apply to similar pesticides.

5.3.2.3 LOD is specific to a pesticide-commodity pair and must be evaluated for every pesticide-commodity pair.

5.3.2.4 Although a method may be extended to other commodities and pesticides, a minimum amount of LOD verification and recovery data must be obtained to confirm this assumption.

5.3.3 This SOP details various scenarios and their corresponding method validation requirements.

5.3.4 When problems occur, such as instrument reproducibility and/or linearity, an investigation of causes shall be conducted. A flow diagram is attached (*see Attachment 1 – Method Evaluation Flowchart*) which further clarifies these concepts.

5.4 General Method Validation Requirements

5.4.1 Methods selected for use by PDP laboratories, and significant changes to approved methods, are subject to prior approval by USDA/AMS.

5.4.2 The laboratory shall complete all required method validation modules, with the exception of precision and accuracy data collection (extracted, analyzed, and reviewed) prior to the extraction of any routine analytical sample sets.

5.4.3 An extraction/detection system includes the whole method: extraction, clean-up, chromatography, and analytical technique.

5.5 Method Validation Evaluation Guidelines

5.5.1 The following scenarios shall be followed for validation of new methods or changes/additions to existing methods. The following scenarios of changes/additions are possible:

5.5.1.1 Implementing a new method (5.6.1)

5.5.1.2 Changing an analytical method

5.5.1.2.1 Extraction (5.6.2.1)
5.5.1.2.2 Post-extraction/pre-instrumentation (5.6.2.2)
5.5.1.2.3 Instrumentation - new Limit of Detection (LOD) (5.6.2.3)
5.5.1.2.4 Minor Modifications (5.6.2.4)

5.5.1.3 Adding a new commodity grouping (5.6.3)

5.5.1.4 Adding a raw agricultural commodity or a processed commodity to an existing commodity group. (5.6.4)

5.5.1.5 Adding pesticides related to marker pesticide groups to an existing commodity group (5.6.5). (*see Attachment 2 – PDP Compound Groups, Pesticides Codes and Multi-residue Compound Groupings for Fruit and Vegetables).*

5.5.1.6 Adding a new pesticide that is not related to marker pesticide groups to an existing commodity group. (5.6.6)

5.5.2 Evaluation takes place through the performance of method evaluation modules. These modules are chosen to meet the requirements of each scenario. The modules are:

- Establishment of LODs and Limits of Quantitation (LOQs) (5.10)
- Verification of LODs/LOQs (5.11)
- Determination of Method Range (from 1xLOQ to 10xLOQ) (5.13)
- Precision and Accuracy Data Collection at 2xLOQ (5.14)
- Method Evaluation Reporting (5.15)

5.5.3 Section 5.6 of this SOP lists each scenario and the modules that must be performed in that scenario. Sections 5.10 through 5.15 outline the detailed procedures to be followed for each module.

5.6 Method Validation Scenarios

The TPM and QAO will determine which scenario described in the following subsections applies for the analytes/commodities/methods pairings *(see Attachment 2 – PDP Compound Groups, Pesticides Codes and Multi-residue Compound Groupings for Fruit and Vegetables).* If local agreement cannot be reached, the Monitoring Programs Division (MPD) Director shall be contacted to determine which modules should be performed.

5.6.1 New method implementation – Proceed with:

- Establishment of LODs and (LOQs) (5.10)
- Verification of LODs/LOQs for all compounds (5.11)
- Determination of Method Range for marker compounds (5.13)
- Precision and Accuracy Data Collection for all compounds (5.14)
- Method Evaluation Reporting (5.15)

5.6.2 Method changes

5.6.2.1 Major Extraction Change - Examples would be using a different solvent, solid phase extraction (SPE) sorbent bed, or a new technique. Proceed with:

- Establishment of LODs and (LOQs) (5.10)
- Verification of LODs/LOQs for all compounds (5.11)
- Determination of Method Range for marker compounds (5.13)
- Precision and Accuracy Data Collection for all compounds (5.14)
- Method Evaluation Reporting (5.15)

5.6.2.2 Major changes in post-extraction/pre-instrumentation procedures (cleanup) - Proceed with:

- Verification of LODs/LOQs for all compounds (5.11)
- Determination of Method Range for marker compounds (5.13)
- Precision and Accuracy Data Collection for all compounds (5.14)
- Method Evaluation Reporting (5.15)

5.6.2.3 Instrumentation Changes - The TPM and QAO will determine if the instrument change warrants completion of the following sections. *This is dependent upon the extent*

of modification. If local agreement cannot be reached, the MPD Director shall be contacted for further resolution.

For new LOD - Proceed with:

- Establishment of LODs/LOQs for all compounds (5.10)
- Verification of LODs/LOQs for all compounds (5.11)
- Method Evaluation Reporting (5.15)

The laboratory shall use best professional judgment to determine if Precision and Accuracy Data Collection (subsection 5.14) is necessary.

5.6.2.4 Minor modifications of existing method - The TPM and QAO will determine which portions of the following sections will be completed. *This is dependent upon the extent of modification. If local agreement cannot be reached, the MPD Director shall be contacted to determine which sections should be performed.*

- Establishment of LODs and LOQs of affected analytes (5.10)
- Verification of LODs/LOQs of affected analytes (5.11)
- Determination of Method Range of affected markers (5.13)
- Precision and Accuracy Data Collection of affected analytes (5.14)
- Method Evaluation Reporting (5.15)

5.6.3 Adding a new commodity group - Proceed with:

- Verification of established LODs/LOQs for all required pesticides in the new commodity (5.11)
- Determination of Method Range for the marker pesticides (5.13)
- Precision and Accuracy Data Collection for all required analytes (5.14)
- Method Evaluation Reporting (5.15)

5.6.4 Adding a raw agricultural commodity or processed commodity (i.e., canned/frozen/dried/ juice) to an existing commodity group. Proceed with:

- Verification of established LODs/LOQs for all required pesticides (5.11)
- Precision and Accuracy Data Collection (2 points) for all required pesticides (5.14)
- Method Evaluation Reporting (5.15)

The laboratory shall use best professional judgment to determine if additional validation is necessary based on matrix behavior.

5.6.5 Adding pesticides related to the marker pesticide groups to an existing commodity group – Proceed with:

- Establishment of LODs and LOQs for each pesticide added (5.10)
- Verification of LODs/LOQs for each pesticide added (5.11)
- Precision and Accuracy Data Collection for each pesticide added (5.14)
- Method Evaluation Reporting (5.15)

5.6.6 Adding pesticides that are not related to the marker pesticide groups to an existing commodity group: (For example, the addition of imidacloprid analyzed by the same multiresidue procedure. The new pesticide may then become a marker pesticide for similar pesticides that are later added.) - Proceed with:

- Establishment of LODs and LOQs for each pesticide added (5.10)
- Verification of LODs/LOQs for each pesticide added (5.11)
- Determination of Method Range for compound(s) that are to become marker(s) (5.13)
- Precision and Accuracy Data Collection for each pesticide added (5.14)
- Method Evaluation Reporting (5.15)

5.7 Marker Pesticides

5.7.1 Assigning Compounds to Marker Groups

5.7.1.1 Compounds are placed into marker groups based on a combination of analyte chemistry and method performance behavior. Initial compound designations are made by the Technical Advisory Group (TAG), with applicable analytical laboratory input based on known method behavior, if those data are available. For new compounds, behavior data may not be available.

5.7.1.2 Final marker group assignment, and any marker group assignment changes, are based on laboratory experience. USDA/AMS maintains an "Effective Date" field that tracks initial group assignment as well as any changes in that initial assignment.

5.7.2 Multi-residue Screening

5.7.2.1 A laboratory may choose to use marker groups, rotate spike mixtures between analytical sets, or spike all compounds analyzed, as long as each extraction/detection system is adequately represented within each set.

5.7.2.2 For laboratories using marker groups, each laboratory shall select at least one compound from each applicable group (*see Attachment 2 – PDP Compound Groups, Pesticides Codes and Multi-residue Compound Groupings for Fruit and Vegetables)* to serve as a marker pesticide. Applicable groups are those that contain at least one compound analyzed by that laboratory for that commodity. For each applicable group, a marker pesticide shall be included for each extraction/detection system used to analyze that group.

5.7.2.3 For laboratories rotating spike mixtures between analytical sets, each laboratory shall ensure that each extraction/detection system is adequately represented within each set.

5.7.2.4 For laboratories analyzing multiple commodities, a single list of marker compounds may be specified to represent all commodities. The lists of required compounds for commodities analyzed should be combined and at least one compound from each applicable group chosen to serve as a marker compound.[2]

5.7.3 Selected/single analyte residue studies utilize the selected analyte as the marker pesticide.

5.7.4 "Marginal Performing Analytes" are analytes that do not meet linearity, calibration integrity, ion ratio, recovery (individual or mean), or precision and accuracy criteria during method validation or continuing quality control (QC) as specified in Section 5.18. Marginal performing analytes are determined in conjunction with USDA/AMS.

5.8 Process Control Compounds

[2] For laboratories analyzing multiple commodities, compounds in single groupings only need apply to that required commodity.

Samples analyzed by each extraction/detection system shall include the analysis of a process control compound. More than one process control may be required. The laboratory shall make every effort to choose a compound that is not expected to be an incurred residue.

5.9 PDP Commodity Groupings

Fruits and Vegetables: Apples (AP), Apple Juice (AJ), Applesauce (AC), Asparagus (AS), Avocado (AV), Baby Foods (see *Attachment* 3 for corresponding codes), Bananas (BN), Basil (BS), Blueberries (BB), Broccoli (BR), Cabbage (CG), Canned Beans (BC), Canned Beets (BT), Canned Cranberries (RC), Canned Garbanzo Beans (ZB), Canned Olives (OL), Canned Peaches (CC), Canned Peas (SD), Canned Pineapples (NC), Canned Spinach (SC), Canned Sweet Corn (CD), Canned Tomatoes (TC), Cantaloupe (CN), Carrots (CR), Cauliflower (CF), Celery (CE), Cherries (CH), Cherry Tomatoes (CT), Cilantro (CL), Cranberries (CA), Cucumbers (CU), Dried Garbanzo Beans (ZD), Dried Plums/Prunes (PD), Eggplant (EP), Frozen Peas (PS), Frozen Raspberries (RZ), Frozen Spinach (SF), Frozen Strawberries (SZ), Frozen Sweet Corn (CS), Frozen Winter Squash (WZ), Grapefruit (GF), Grapes (GR), Grape Juice (GJ), Green Beans (GB), Green Onions (GO), Greens (GS), Honeydew Melons (HD), Hot Peppers (HP), Kale (GK), Kiwi (KW), Lettuce (LT), Mangoes (MA), Mushrooms (MU), Mustard Greens (MG), Nectarines (NE), Onions (ON), Oranges (OG), Orange Juice (OJ), Papaya (YA), Peaches (PC), Pears (PE), Pear Juice (PJ), , Pineapples (PN), Plums (PU), Potatoes (PO), Radish (RD), Raspberries (RS), Snap Peas (SN), Spinach (SP), Strawberries (ST), Summer Squash (SS), Sweet Bell Peppers (PP), Sweet Cherries (CH), Sweet Corn (CB), Sweet Potatoes (SW), Tangerines (TA), , Tomatoes (TO), Watermelon (WM), Winter Squash (WS)

Cereal Grains (Low Oil): Barley (BY), Corn Grain (CO), Oats (OA), Rice (RI), Wheat (WH), Wheat Flour (WF)

Cereal Grains (High Oil): Almonds (AL), Peanut Butter (PB), Soybeans (SY),

Animal Tissue/High Protein: Beef (adipose – BA, liver – BL, muscle – BM), Catfish (FC), Eggs (EG), Pork (adipose – KA, muscle – KM), Poultry (adipose – PA, liver – PL, muscle – PM, breast – PR, thigh – PT), Salmon (FS)

Dairy Products: Butter (BU), Heavy Cream (CM), Milk (MK)

Water: Untreated Drinking Water (WU), Treated Drinking Water (WR), Bottled Water (WB), Groundwater (WG)

Single Commodities: For example, Corn Syrup (CY), Dairy-based Infant Formula (DF), Raisins (RA), Soy-based Infant Formula (YF), Tomato Paste (TP), Honey (HY).

5.9.1 Based on their experience with a commodity, laboratories may request changes to the assigned commodity groupings from the MPD Director.

5.9.2 Environmental Protection Agency (EPA), Codex, and Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) commodity grouping information and the FDA Information for percent fat, water, sugar and pH content for Commodity Groupings can be found in attachments 3 and 4 of this SOP.

5.10 Establishment of LODs and LOQs

5.10.1 Method Noise

5.10.1.1 Method noise is the combination of instrument noise and the matrix noise contributions.

5.10.1.2 Method noise determination must be completed for all required PDP analytes.

5.10.1.3 Method noise will be determined utilizing instruments and operating conditions, which are routinely used for the analysis of samples. Noise for the LOD and LOQ calculations will be determined by examining chromatograms of the blank commodity in the chromatographic time segment of the pesticides of interest.

5.10.2 Establishment of LOD

5.10.2.1 LOD may be estimated by whatever means the laboratory chooses to employ, but the response shall be at least 3x signal to noise.

For MS systems, ions used for quantitation and for qualitative analysis/confirmation shall meet the 3x signal to noise requirement.

For example: 1) take two equal portions from the same matrix blank extract; 2) spike one aliquot with a known amount of the analyte of interest; 3) inject both aliquots under the same conditions; 4) magnify the baseline of the unfortified blank at the analyte retention time window of interest to obtain the instrument response for the tallest (height) or the broadest (area) noise; and 5) convert the response into concentration (ppm, ppb, or ppt) from the known concentration of the spiked extract. Compare the two concentrations (blank vs. spiked) to estimate the LOD.

5.10.2.2 LODs may be established at a level greater than 3x noise.

5.10.2.3 In addition to signal-to-noise considerations, LODs estimated for zero noise instruments (e.g. triple quadrupoles) may also include consideration of replication injection data (e.g. injecting an LOD standard 10x).

5.10.2.4 The reported LOD shall be the highest value obtained using the validated method. For instance, for dual column systems, the confirmatory column LOD must be AT LEAST that of the primary/quantitative column.

5.10.2.5 For multi-peak compounds, such as many of the pyrethroids, the laboratory may base the LOD on the largest peak if a mass spectrometry system is used for both quantitation and confirmation. If other systems are used for quantitation, the laboratory may base the LOD on the larger peak if the smaller peak is <20% of the total response.

5.10.2.6 LOD is method dependent and shall be experimentally verified in matrix as detailed in Section 5.10.1.

5.10.3 Establishment of LOQ

5.10.3.1 LOQ will be calculated/determined for each analyte in each commodity tested following the establishment of LOD.

5.10.3.2 For all detection systems other than mass spectrometry, LOQ will be established by multiplying the response of method noise level by at least ten and then converting the total response into concentration (i.e., ppm, ppb, or ppt), or by multiplying

the LOD by no less than ten/thirds (10/3) if the LOD is established above 3x method noise.

5.10.3.3 For mass spectrometric systems, ions to be used for qualitative analysis/confirmation shall be at least 3x signal to method noise. Ions to be used for quantitation shall be at least 10x signal to method noise.

5.10.3.3.1 In order to maximize the number of compounds screened by MS systems while maximizing the number of scans per second and dwell times, it may be desirable to perform the initial identification and quantitation using fewer than three ions for some or all of the compounds. Presumptive-positive samples shall be re-injected or data reprocessed to meet all MS confirmation criteria.

5.10.3.4 The reported LOQ shall be the highest value obtained using the validated method.

5.11 Verification of LODs/LOQs

5.11.1 During method validation, all calculated or established LODs must be verified by fortifying duplicate blank commodities at approximately the LOD level and subjecting them to the analytical method for each extraction/detection system used in the analysis of PDP samples. In the instance where the LOD=LOQ this verification suffices for the LOD and LOQ. If method range is performed (see subsection 5.13) for verification of LOQ then section 5.11 is not required.

5.11.2 Verification consists of the observation of detectable peaks in the chromatogram at 3x the current noise level (run within the last three months). Variability is expected to be high. Therefore, recoveries can be reported as present or not present. If detectable peaks are not observed, the LOD must be re-estimated and the verification repeated.

5.11.3 Prepare summary form(s) of the acquired data for all systems and all columns used for analysis and/or confirmation *(see Attachment 5 - Method Evaluation Reporting Forms).*

5.11.4 For water only, the LOD for each reported compound shall be verified, at least every two years, by extraction of a single LOD spike. Reporting these results to USDA/AMS is

optional. If the LOD Verification Form in Attachment 5 is used, then recording only one LOD spike is required.

5.12 Changing LODs

5.12.1 LODs may be raised for analytes in an individual sample set at the discretion of the TPM.

5.12.2 LODs may not be lowered without verification subject to the analytical method, TPM approval, and QAO review.

5.13 Determination of Method Range

5.13.1 During method validation, samples fortified with marker compounds (only marker compounds are required, however, other compounds may be used in addition to the markers, if desired) are to be run through the entire analytical method on the primary analytical system. If more than one type of chromatography system (e.g., GC versus LC) and/or detector system (e.g., FPD versus MSD) combinations are to be used for quantification, they must be likewise evaluated.

5.13.2 Fortify samples in triplicate at approximately 1xLOQ, 5xLOQ, and 10xLOQ for each marker or compound being validated. Process these fortified samples through the entire analytical method. A reagent and matrix blank shall be subjected to the analytical method along with the fortified samples.

5.13.3 For each data point, calculate the Percent Recovery compared to known standards to three significant figures if greater than 100% or to two significant figures if less than 100%.

5.13.4 Calculate the mean Percent Recovery (%R) and Coefficient of Variation (%CV) for each level. A definition of Horwitz expected intralaboratory and interlaboratory %CVs may be found in SOP PDP-Glossary. The appropriate values may be used as a guideline when evaluating data.

5.13.5 Prepare summary form(s) of the acquired data by analyte, level, and commodity group *(see Attachment 5 - Method Evaluation Reporting Forms).*

5.13.6 Method Range Extension

If more than 20 findings per life of the commodity for a particular analyte/commodity pair exceed the highest validated spiking level, then in order to verify the ability of the method to extract the analyte at the higher level, the laboratory shall fortify at least one spike at or above the level of the highest finding. Reagent and matrix blanks shall accompany these spikes. If the matrix spike recoveries do not meet QC criteria (per section 5.18) any affected findings shall be coded (or recoded) as estimates. Method range extension spikes may be reported via RDE as "other" spikes. Marker pesticide spikes may be used to represent other compounds in that group. Method range extension for a given commodity can represent another commodity in that group. Laboratories may perform the range extension at various times:

- preemptively during initial validation (based on intelligence or experience with the commodity),
- in subsequent batches following the high finding,
- periodically (e.g. annually) to conserve resources, or
- internal blind check samples may be used for this purpose.

Method range extension results should be reported to USDA/AMS following QA review. USDA/AMS expects any **coding changes** for calendar year samples to be submitted by May 31st following the end of the calendar year. This does not remove the requirement to report all data sets for the calendar year by March 31st of the following calendar year.

5.14 Precision and Accuracy Data Collection

5.14.1 The precision and accuracy data collection shall be compiled from the commodity groupings as specified by USDA/AMS. Each marker, single analysis, new or other required PDP analyte shall be spiked at 2xLOQ and evaluated using a minimum of seven data points, with at least two points from each commodity in the group analyzed in a particular laboratory.

5.14.2 The required data points shall be obtained from:

- 2xLOQ data points completed after Determination of Method Range

and/or

- data points from matrix spikes analyzed concurrently with samples.

These two options provide slightly different data. The second option is preferable since it provides information about the repeatability of the method over time. The first option is permitted when running concurrent spikes would extend the data collection over more than six months and/or concurrent spikes would make the size of sample sets unmanageable.

5.14.3 For each data point, calculate the Percent Recovery compared to known standards to three significant figures if greater than 100% or to two significant figures if less than 100%.

5.14.4 Calculate the mean Percent Recovery (%R) and Coefficient of Variation (%CV) for each pesticide using the seven data points. A definition of Horwitz expected intralaboratory and interlaboratory %CVs may be found in SOP PDP-Glossary. The appropriate values may be used as a guideline when evaluating data and/or determining whether analytes should be considered a Marginal Performing Analyte. In addition, Marginal Performing Analytes may be determined based on linearity, calibration integrity, or individual recovery values.

5.14.5 Prepare summary form(s) of the acquired data *(see Attachment 5 - Method Evaluation Reporting Forms*). Refer to Sections 5.17 for PDP acceptance criteria.

5.15 Method Evaluation Reporting

5.15.1 The methodology, method evaluation records, summary form(s), chromatograms, and any other supporting data generated during method evaluation shall be maintained by the laboratory.

5.15.2 Local Approval

5.15.2.1 Any request for and written modification of an approved analytical method shall be reviewed and approved by the QAO and TPM.

5.15.2.2 All validation documentation shall be reviewed and approved by the QAO and TPM.

5.15.3 Letter of Intent

5.15.3.1 Once the Verification of LODs and LOQs and Determination of Method Range has been completed, reviewed, and approved by the QAO and TPM, a Letter of Intent shall be submitted to the MPD Director with copies to the Method Validation Coordinator and the assigned laboratory liaison stating that these modules have been completed, reviewed, and approved and will be submitted at a later date with the Precision and Accuracy Data.

5.15.3.2 This letter shall also include a list of commodity(ies) and analyte(s) with their LOD(s) that the laboratory intends to analyze and shall be submitted within 90 days of the applicable commodity entering the program.

5.15.3.3 The Letter of Intent is not required if all required method validation data will be/is submitted within 90 days of the commodity entering the program.

5.15.3.4 USDA/AMS will perform a brief preliminary review and upon laboratory request, will issue a provisional letter of concurrence allowing the laboratory to transmit data to their laboratory liaison for review while the full method validation package undergoes a multi-level review by USDA/AMS. Data may be changed, in consultation with the lab, based on the results from the full method validation package review.

5.15.4 Upon conclusion of the Precision and Accuracy Data Collection module, summary form(s) of validation documentation, and a brief narrative shall be sent by email to the MPD Director with copies to the Method Validation Coordinator and the assigned laboratory liaison with a cover memo detailing the submission (state which scenario(s) and module(s) that the submission is intended to represent).

5.15.5 A narrative accompanying the validation documentation shall include the following.

5.15.5.1 Description of the method.

5.15.5.2 Identification of any data that is only intended to be used for confirmation. Otherwise, USDA/AMS will evaluate the data as if quantitation will be performed on the instrument/analyte combination.

5.15.5.3 Requests for designation of any analytes as Marginal Performing Analytes - if USDA/AMS agrees to consider any analytes as Marginal Performing Analytes, that designation will be documented in the Letter of Concurrence.

5.15.5.4 Identification of previous method validation data used. The laboratory shall be responsible for clearly identifying the data used and the rationale for their use. For example, if a previously validated commodity returns and the laboratory has not made any method changes and will be using the same instrumentation, the laboratory shall submit a letter to USDA/AMS explaining how the previous validation data will be used.

Example narrative for a data package:

Enclosed is the complete method validation summary of all compounds we are screening for in commodity "y" to support the addition of the commodity to the 2010 PDP program. The specific scenario used in validation was 5.6.1, New Method Implementation. Required modules included establishment and verification of LODs and LOQs, determination of method range, precision and accuracy data collection, method evaluation reporting for GC/MSD, GC/FPD, GC/XSD, and LC/MS/MS instrumentation. For compound "a", GC/FPD is the primary detection system and LC/MS/MS data is intended for confirmation purposes only. The following analytes were dropped during method development due to difficulty in analysis (e.g., solubility, poor chromatography, sensitivity, and/or loss in SPE cleanup): compound "b", compound "c", and compound "d". Due to problems with recovery, the following analytes should be considered Marginal Performing Analytes and if it is agreed, will be coded as such in reporting: compound "e", compound "f", and compound "g". If there are questions about this submission please contact: XXXXXX. All references to this submission should use QA# ###-####.

Example narrative for a previously validated returning commodity with no method, analyte, or instrumentation changes:

In 2011, commodity "y" returned to the 2011 PDP program. This commodity was previously validated in 2008 and there have been no changes to the method, target analytes, and instrumentation since then. Therefore, the 2008 validation data submitted on Month, Day, Year, is still applicable. If there are questions about this submission please contact: XXXXXX. All references to this submission should use QA# ###-####.

An example format for the submission follows:

Title
Summary to include purpose, results, data anomalies.
Methods
Sample Preparation (example):

- 50g homogenized sample extracted with 100 ml ACN by gently mixing
- 5ml extract purified by a C-18 SPE cartridge, eluted with MeOH, and concentrated to 5ml
- 1 ml eluate further purified by florisil SPE and eluted with 5 ml 50:50 hexane/acetone
- Eluate dried down to 0.5 ml, re-suspended in acetone, and filtered
- Derivatizaton accomplished by reaction with dansyl chloride.

Analysis (example):

- Instrument GC/HPLC/detector
- Column (DB-)
- Post-column derivatization (where applicable).

5.16 Method Validation Evaluation by USDA/AMS

5.16.1 Letter of Intent

5.16.1.1 Letters of Intent shall be tracked and maintained in centralized files by the Method Validation Coordinator.

5.16.1.2 The USDA/AMS laboratory liaison assigned to that facility submitting a Letter of Intent shall review the letter and verify the submitted LOD/LOQ values against electronically submitted data (upon availability) and upon laboratory request issue a provisional letter of concurrence (see Section 5.15.3).

5.16.2 Method Validation Data Packages

5.16.2.1 After receipt by the MPD Director, Method Validation

Coordinator and USDA/AMS laboratory liaison, method validation data packages undergo a multi-tiered review by USDA/AMS. Details of this review process are specified in SOP PDP-ADMIN.

5.16.2.2 The method validation package is reviewed to ascertain the physical presence and completeness of data submitted for method validation and to determine whether these data adhere to PDP criteria and are to be considered validated.

5.16.2.3 For data that do not meet PDP criteria for linearity, calibration integrity, ion ratios, individual or mean recovery (50-150%) or reproducibility (%CV values within the expected Horwitz intralaboratory values) USDA/AMS and the laboratory shall use scientific judgment to determine whether the compound shall be considered validated, designated as a Marginal Performing Analyte or designated as unvalidated for that pesticide/commodity pair.

Note: *The Horwitz values are used as guidelines only and do not preclude a compound from being considered validated.*

5.16.2.4 Once the USDA/AMS review of the method validation package has been completed, the laboratory TPM and QAO will receive a Letter of Concurrence that identifies the status of the instrument/detector results for the commodity/analyte pairing (e.g., validated, not validated, Marginal Performing Analyte, incomplete). If the data are deemed incomplete by USDA/AMS, the Letter of Concurrence will identify the deficiency and include a request for the remaining data e.g., monitoring of daily matrix fortifications or addition of a spike compound with the same functional group to the fortification profile).

5.16.2.5 Once a compound is designated as a Marginal Performing Analyte, that designation shall not be changed unless approved by the MPD Director.

5.17 Blanks and Spikes Required Per Set and Continuing QC

5.17.1 Sample set

A sample set is a group of samples, which are spiked individually with the designated process control(s), extracted with the required QC samples, and analyzed with the applicable required QC samples. Each set shall not exceed 35 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.

5.17.2 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, appropriate action must be taken and documented.

5.17.3 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, appropriate action must be taken, such as reviewing reagent blank information.

5.17.4 Matrix Spike

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. Analysis of a matrix spike provides valuable information on matrix interference

effects as a result of the co-eluted matrix components, affecting the accuracy or detection capability for the analytes of interest.

5.17.4.1 A second portion of the same material used for the matrix blank shall be used for the matrix spike(s). Laboratories may design their QC spiking schemes to meet their needs. A laboratory may choose to use marker groups as defined in Section 5.7 of this SOP, rotate spike mixtures between analytical sets, or spike all compounds analyzed, as long as each extraction/detection system is adequately represented within each set and the minimum requirement of all compounds reported by the laboratory to be spiked at least quarterly in each commodity, is met.

5.17.4.2 The spike shall be added prior to extraction at approximately 2x LOQ (or less). Additional spikes may be added to satisfy the quarterly spiking of each commodity with all reported compounds, as part of a validation study, 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 QA/QC Recovery Form shall indicate which commodity was used for the matrix spikes. Results for all spiked compounds shall be reported to USDA/AMS through normal RDE procedures.

5.17.4.3 The matrix spike(s) shall meet the criteria requirements specified in section 5.18.2. All reported compounds (markers, required, and any other compound reported by that laboratory) shall be spiked at least quarterly for each commodity. All components of sample sets shall be subject to the same analytical process as detailed in the method SOPs.

5.17.4.4 Recoveries for compounds designated as Marginal Performing Analytes shall be coded with a "P" (Marginal Performing Analyte) in the Exception field of the QA/QC Recovery form.

5.17.4.5 If reported, recoveries for unvalidated compounds shall be coded with a "U" (Unvalidated Residue) in the Exception field of the QA/QC Recovery form.

5.17.4.6 Incurred residue levels may be subtracted from spike recovered prior to calculating the percent recovery if the conditions specified in SOP PDP-DATA are met.

5.17.5 Reporting Fortification Recoveries

5.17.5.1 "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).

5.17.5.2 "Other" spikes are additional fortifications reported by the laboratory. The laboratory can request that USDA/AMS adds 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.

5.17.6 Quarterly 2xLOQ Spikes

5.17.6.1 All reported compounds (markers, required, and any other compounds reported by the laboratory) shall be spiked at least quarterly at 2x LOQ (or less) for each commodity.

5.17.6.2 The laboratory may choose to rotate spikes on a regular basis as long as the requirements in Subsection 5.17.4.1 are met.

5.17.6.3 The spike results shall be reported to USDA/AMS via RDE (the preferred option) or in Excel spreadsheets. Results shall also be addressed in the semi-annual QA Reports submitted to USDA/AMS.

5.17.7 Process Control Spikes

A process control spike is intended to assure the integrity of a particular sample within an analytical system.

5.17.7.1 Each sample set component, except the reagent and matrix blanks, shall be spiked with a process control at approximately 5x the Limit of Quantitation (LOQ) prior to the extraction step of the analytical procedure. However, if the intent of the process

control is to monitor the percent recovery of a clean-up step, or of a derivatization, then the process control shall be added to the extract before the clean-up or derivatization step.

5.17.7.2 The laboratory shall make an effort to choose a compound that is not expected to be an incurred residue. The value reported as "percent recovery" may be the original, re-injected, re-aliquoted, or re-extracted (from homogenate) determination value [either value from primary detection system or averaged value (e.g., dual column results averaged)].

5.17.8 QA/QA Recovery Form Codes

The following codes shall be entered in the Exception field of the QA/QC Recovery form. See Section 5.17.4 for additional details.

Code	QA Spike Exception
E	Estimated
I	Incurred Residue
M	Matrix Interference
N	Not Recovered
P	Marginal Performing Analyte
S	Incurred Residue Subtracted
U	Unvalidated Residue

5.18 Criteria for Method Validation and Continuing QC

5.18.1 Method Validation Criteria

5.18.1.1 PDP criteria for percent recovery for determination of method range and precision and accuracy data collection is 50-150%.

5.18.1.2 Horwitz intralaboratory values are used as a guideline for determining reproducibility acceptability. The laboratory shall indicate any compounds that they feel are not acceptable and/or those that should be classified as Marginal Performing Analytes. These laboratory recommendations are subject to approval by USDA/AMS.

5.18.1.3 Some analytes may not meet method validation criteria for linearity, calibration integrity, ion ratios, recovery (individual or mean), or precision (%CV). Rather than not including them in the laboratory's screening list, USDA/AMS and the laboratory may decide that marginal data are preferable to no data. These compounds shall be designated as Marginal Performing Analytes. Details on USDA/AMS review of method validation data can be found in Section 5.15.

5.18.2 Matrix Spike Criteria

5.18.2.1 All spiked compounds shall have recoveries between 50 and 150%, within the statistically calculated range, or within a range agreed upon with USDA/AMS.

5.18.2.2 If a large number of analytes are in the spike, it becomes statistically likely that a few will be outside control limits. This may not indicate that the system is out of control. The laboratory shall have written criteria for when corrective action(s) will be necessary.

5.18.2.2.1 Some analytes may not be optimally recovered during method validation trials. Recoveries may be low and/or erratic and rather than not including them in the laboratory's screening list, the laboratory may consult with USDA/AMS to determine if marginal data may be preferable to no data. If reported by the laboratory, the codes for Marginal Performing Analytes shall be utilized. USDA/AMS will note the use of Marginal Performing Analytes in the Letter of Concurrence and the use of marginal performer codes for particular analyte/commodity pairs. Once a compound is designated as a Marginal Performing Analyte, that designation shall not be changed unless approved by the MPD Director.

5.18.2.2.2 Some analytes that behave acceptably during method validation may behave unacceptably during the analysis of routine batches. This may be due to the fact there is more commodity variability among actual samples than there is in the limited matrix utilized for method validation batches. As above, rather than dropping these analytes from the screening list, the laboratory should consult with USDA/AMS to determine if they should be reclassified as Marginal Performing Analytes. If a compound is reclassified as a Marginal Performing Analyte, an e-mail notification to the MPD Director, with a copy to the USDA/AMS laboratory liaison, shall be sent and approved/acknowledged by USDA/AMS, and that designation shall not be

changed unless an e-mail communication is sent by the MPD Director reversing the previous approval.

5.18.3 Response To Failure To Meet Matrix Spike Criteria Range

If a spike analyte fails, even after re-injection/re-aliquoting, it can and should be reported, because the recovery may reflect normal random variation inherent to pesticide residue analysis. For high recoveries, this practice is defensible. For low recoveries, best professional judgment should be used, although if recovery is 0%, that analyte should be reported as unable to detect in samples.

When a spiked pesticide recovery falls outside the range criteria, any one of the following options, or combination thereof, may be chosen by the TPM or designee. (See *Attachment 6 – Matrix Spike and Process Control Recovery Acceptability Flowchart.*)

5.18.3.1 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.

5.18.3.2 The sample set may be re-extracted from the frozen homogenate. If the spiked pesticide recovery falls within the range criteria, the rerun results shall be reported.

5.18.3.3 The original results may be reported with an explanation (e.g., recovery exceed 150% but all samples in the set are non-detects for that analyte; wrong mix spiked; spike spilled but process controls in samples are acceptable; control charts indicate a recurrent analyte/matrix; etc.) The TPM and QAO shall ensure that reported data is not compromised and the explanation shall be conveyed to headquarters (e.g., note in RDE, email message to USDA/AMS laboratory liaison and MPD Director).

5.18.3.4 Other options may be acceptable depending on the outcome of investigations and/or consultations with USDA/AMS. An explanation shall be conveyed to headquarters.

5.18.4 Process Control Criteria

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 approved by the laboratory QAO.

5.18.4.1 Absolute Range

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

5.18.4.2 Statistically Calculated Range

The mean recovery for a sample set's process control shall be calculated. Each process control recovery shall fall within its acceptance recovery range, which is the mean recovery plus and minus three standard deviations.

5.18.5 Response To Failure To Meet Chosen Process Control Criteria Range

If a process control fails, even after re-injection/re-aliquoting/re-extraction, the results may be reported, based on best professional judgment.

When a process control falls outside the chosen range criteria, any one of the following options, or combination thereof, may be chosen by the TPM or designee. (*See Attachment 6 – Process Control and Spike Recovery Acceptability Flowchart.*)

5.18.5.1 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.

5.18.5.2 The sample may be re-extracted from the frozen homogenate. If the process control recovery falls within the chosen range criteria, the re-run results shall be reported.

5.18.5.3 The original results may be reported with an explanation (e.g., pipette error, the PC recovery exceeds 150% but all analytes in the sample are non-detects, etc.). The

TPM and QAO shall ensure that reported data is not compromised and the explanation shall be conveyed to headquarters (e.g., note in RDE, email message to USDA/AMS laboratory liaison and MPD Director).

5.18.5.4 Other options may be acceptable depending on the outcome of investigations and/or consultations with USDA/AMS. An explanation shall be conveyed to headquarters.

5.18.6 Evaluation of Recoveries

Laboratories shall use control charting or other appropriate statistical tools to evaluate recoveries on a set-to-set basis and monitor trends over time.

5.19 Proficiency Testing

5.19.1 PDP PT Program Overview

5.19.1.1 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.

5.19.1.2 General multi-residue method samples for fruit and vegetables will be supplied by the Food Analysis Performance Assessment Scheme (FAPAS) and the California Department of Food and Agriculture (CDFA).

5.19.1.4 Rounds for commodities other than fruit and vegetables (e.g., meat, milk and dairy products, fish, grains, nuts, etc.) shall be supplied by CDFA. Additionally, applicable FAPAS rounds may be scheduled.

5.19.1.5 PT samples received may be significantly larger than the analytical portion required by the laboratory for analysis. In cases where the PT sample is more than twice the analytical weight needed, the laboratory may subsample duplicate portions for extraction and analysis as described below, due to the uncertainty regarding homogeneity of samples. Sample results that meet the QC criteria shall be averaged for reporting.

5.19.1.5.1 Samples shall be mixed in the container they came in, taking care to not spill any sample prior to subsampling.

5.19.1.5.2 Two sub-samples shall be weighed out for extraction and extracted as separate samples.

5.19.1.5.3 Each extract shall be analyzed as an individual sample

5.19.2 Reporting PT Results

5.19.2.1 For FAPAS, lists of potentially spiked pesticides are available from the provider websites. Rounds issued by CDFA are designed to focus only on those compounds validated by the applicable laboratory(ies). For all rounds, participants shall only be evaluated for those residues validated by their laboratory and not declared as Marginal Performing Analytes. The report provided will clearly identify these pesticides. Reporting of the Marginal Performing Analytes shall be optional.

5.19.2.2 For FAPAS, it is recognized that a laboratory may not have validated the commodity scheduled for that specific round. Standards used in routine analyses of assigned commodities should be used. Efforts will be made to provide a matrix blank for each round.

5.19.2.3 Report results according to provider instructions and requirements. Reporting to USDA/AMS via RDE is optional.

5.19.2.4 For FAPAS, LOD/LOQ and recovery values reported may be values obtained from previous routine batches of the laboratory's usual commodity(ies).

5.19.2.5 Reports for each round shall be posted to the PDP Extranet within 10 working days of receipt by USDA/AMS.

5.19.3 Laboratory Response

5.19.3.1 Upon receipt of PDP PT results, laboratories shall review results and initiate corrective actions when they are considered unacceptable by the PT scheme provider.

5.19.3.2 Where FAPAS is the provider, z-scores whose absolute values are greater than 3 are unsatisfactory.

Note: *FAPAS' assigned/target value is the consensus value of the submitted results (with appropriate exclusions as noted in the FAPAS reports) and the target standard deviation is determined based on Horwitz.*

5.19.3.3 For rounds provided by CDFA, unacceptable results shall be defined as those outside 50-150% recovery, or outside the statistically calculated range defined as ± 3xSD of the mean of last 20 data points of the laboratory's spike recovery for the compound, or outside a range agreed upon with USDA/AMS. Unvalidated or Marginal Performing Analytes need not meet these criteria, but should be addressed in the PT section of the semi-annual QA report.

5.19.3.4 If any corrective actions are initiated due to the results, USDA/AMS shall be informed within 30 days. Refer to SOP PDP-ADMIN for notification details.

5.20 Measurement Uncertainty

Measurement uncertainty shall be determined on an annual (calendar year) basis by USDA/AMS. USDA/AMS will calculate each year's value using 2x the standard deviation of program recovery data reported with each analytical data set. For example, during calendar year 2003, the mean program matrix spike recovery was 92% and the standard deviation was 26%. Results for 2003 would be expressed as "value ± 52%." USDA/AMS will be responsible for communicating program measurement uncertainty values to data users.

USDA/AMS does not require individual PDP laboratories to report their measurement uncertainty along with sample results.

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Title: Chemical Compounds, PDP Commodity Grouping, Method Validation and Quality Control		
Revision: 9	Replaces: 02/01//2018	Effective: 09/01/2019

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Revision 9 September 2019 Monitoring Programs Division

- Updated Attachment 2 by adding the following compounds: Benzoyindiflupyr, cyclaniliprole, ethaboxam, fenpicoxamid, isopyrazam, kinoprene, mancozeb, oxathiapiprolin, pydiflumetofen, thiram and tridemorph
- Updated CAS# for azinphos methyl oxygen analog in Attachment 2
- Updated Attachment 3 with new commodities: basil, mustard greens and radishes. Added commodity codes for dried garbanzo beans, canned cranberry, canned peas, canned sweet corn, frozen spinach and frozen sweet corn. Changed vegetable peas to frozen peas and fresh sweet corn code (CS) to code (CB)
- Updated Attachment 4 with new commodities: basil, mustard greens and radishes
- Updated Attachment 4 heading in section 3
- Added reference to U.S.FDA, Approximate pH of Foods and Food products in section 4
- Updated U.S. EPA, Pesticide Use Index, U.S. FDA, Pesticide Analytical Manual Volume 1 (PAM) 3rd Edition, Chapter 2 and Codex, Pesticide Residues in Food and Feed web links in section 4
- Updated the webpage for EPA Repository in section 5.2.1.1
- Added clarification to standard checking in section 5.2.6.1
- Rearranged commodities in alphabetical order, changed peas (PS) to frozen peas (PS), changed fresh sweet corn code (CS) to code (CB), added basil, canned cranberries, canned peas, canned spinach, canned sweet corn, cherries, dried garbanzo beans, frozen raspberries, frozen spinach, frozen strawberries, frozen sweet corn, frozen winter squash, kiwi, mustard greens and radishes to section 5.9.
- Updated reference to Attachment 4 in section 5.9.2
- Changed MP to USDA/AMS in section 5.11.4
- In section 5.15.4 removed the requirement to send a hard copy of the method evaluation to USDA/AMS by mail
- Changed PDP Technical Director to MPD Director throughout the document
- Changed USDA/AMS liaison and USDA/AMS liaison chemist to USDA/AMS laboratory liaison throughout the document
- Added USDA/AMS laboratory liaison to section 5.16.2.1
- Clarified section 5.17.4.3 by adding reference to section 5.18.2, matrix spike criteria

Revision 8 February 2018 Monitoring Programs Division

- Updated the heading for section 5.11 throughout the document
- Updated guidance to section 5.11 for LOQ verification
- In section 5.13.2 replaced “single PDP analyte” with “compound being validated”

- Updated Attachment 2 by adding the following compounds: aspon, aclonifen, anilofos, atraton, beflubutamid, bioallethrin, bromophos ethyl, chlordimeform, chlorthiophos, desmetryn, dichlofenthion, dimepiperate, dipropetryn, ditalimfos, diphacinone, dithiopyr, dioxacarb, etrimfos, famphur, fluensulfone, flazasulfuron, flucythrinate, fluorodifen, iprobenfos, isoprothiolane, mephosfolan, methacrifos, mefenacet, simetryn, prodiamine, pretilachlor, pyraclofos, pyridaphenthion, terbutryn, trichloronate
- Added commodity codes for canned tomatoes, canned garbanzo beans, canned peaches, dried plum/prunes and honey to section 5.9
- Updated attachment 3 with new commodities: canned peaches and dried plum/prunes

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- Updated Attachment 2 by adding the following compounds: 1,3 dichloropropene, 2,6 DIPN, 5-(4-chlorophenyl) oxazole-2-propionic acid (CPOPA), acetamide, allidochlor, amicarbazone, aminopyralid, asulam, barban, chlorpyrifos methyl O-analog, cloquintocet methyl, cloquintocet mexyl, cumyluron. cyclanilide, dinocap, ethephon, flumetralin, flupyradifurone, glufosinate, hexaflumuron, isofetamid, kasugamycin, melamine, merphos, metrafenone, niclosamide, oxythioquinox, perthane, piperalin, prohexadione calcium, propoxycarbazone, proquinazid, prothioconazole, quinchlorac, thidiazuron, thiencarbazone methyl
- Removed oxytetracycline from Attachment 2
- Added commodity codes for canned olives, kale, canned pineapple to section 5.9
- Updated section 4: link to Codex Alimentarius Commission, Pesticide Residues in Food and Feed
- Updated attachment 3 with new commodities: canned pineapples and canned olives
- Updated attachment 4 with new commodity: canned olives

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- Updated Attachment 2 by adding the following compounds: acequinocyl, ametoctradin, benalaxyl, benazolin, BHC-delta, BHC-epsilon, bifenox, carbophenothion methyl, chlorobenzilate, cloransulam methyl, cyantraniliprole, cyflufenamid, cyflumetofen, cyprosulfamide, dichlormid, diclosulam, diethofencarb, diniconazole, EPN, ethiofencarb sulfone, ethiofencarb sulfoxide, ethiprole, ethylan, etofenprox, fenoxycarb, fenpropidin, fenpyrazamine, fenthion sulfone, fenthion sulfoxide, fluazifop, flufenpyr ethyl, flumiclorac pentyl, fluopyram, fluthiacet methyl, fomesafen, furalaxyl, heptenophos, imazosulfuron, ipconazole, isocarbophos, isofenphos methyl, isoprocarb, isoproturon, isoxadifen ethyl, mecarbam, mefenpyr diethyl, mesotrione, metolcarb, monolinuron, nitrofen, penflufen, phorate OA sulfone, phorate OA sulfoxide, picoxystrobin, profluralin, profoxydim, pyraflufen, pyroxasulfone, quizalofop, rotenone, sedaxane, sulfallate, sulfoxaflor, terbufos OA sulfone, terbufos sulfoxide, thionazin, tolfenpyrad, topramezone, and tricyclazole

- Included additional matrix spike language to sections 5.17.4 and 5.18.2
- Removed references to AOAC PTs in section 5.19

Revision 5 August 2014 Monitoring Programs Division

- Updated references to USDA/AMS throughout document
- Updated prioritization protocols in sections 5.1.2.1, 5.1.2.2.2, 5.1.2.2.3, and 5.1.2.2.4
- Changed standard checking requirement to 15% RPD in sections 5.2.6, 5.2.6.2, 5.2.7.2.1, and 5.2.7.2.2
- Combined laboratory Letter of Intent requirements into section 5.15.3
- Updated USDA/AMS address in section 5.15.4
- Updated USDA/AMS Letter of Intent procedures in section 5.16.1

Revision 4 July 2013 Monitoring Programs Division

- Updated references to USDA/AMS throughout document
- Added commodity codes for avocado, catfish, dairy-based infant formula, raspberries, salmon, and soy-based infant formula to section 5.9
- Added procedures for subsampling PT samples to section 5.19.1.5
- Updated Attachment 2 by adding the following compounds: acrinathrin, AMPA, aviglycine HCl, bromopropylate, bupirimate, butocarboxim, butocarboxim sulfone, butocarboxim sulfoxide, chlorsulfuron, chlozolinate, clethodim 5 hydroxy sulfone, clethodim sulfone, clethodim sulfoxide, clofencet, crotoxyphos, crufomate, demeton-S, demeton-S sulfone, dichlofluanid, DMST,fenbutatin oxide, fenchlorphos, fenpropimorph, fensulfothion, fenthion o-analog, fipronil sulfone, fluquinconazole, flusilazole, flutriafol, fluxapyroxad, glyphosate, haloxyfop, iodosulfuron methyl, lenacil, lufenuron, mesosulfuron methyl, metaflumizole, methiocarb sulfone, methiocarb sulfoxide, oxytetracyline, paclobutrazol, penconazole, pencyuron, penthiopyrad, phoxim, pirimicarb desmethyl, primisulfuron, propaquizafop, prosulfuron, prothiofos, pyrazaophos, quizalofop ethyl, sethoxydim sulfoxide, spiroxamine, sulfosulfuron, tebufenpyrad, teflubenzuron, terbuthylazine, thifensulfuron methyl, thymol, toxaphene, and tribenuron methyl
- Updated Attachments 3 and 4 for the following commodities: avocado, catfish, infant formula (dairy-based and soy based), raspberries, and salmon

Revision 3 March 2012 Monitoring Programs Division

- Updated prioritization rationale in section 5.1.2
- Added exemption for CoA and current expiration date for revoked compounds to section 5.2.1.2
- Defined extraction/detection system in section 5.4.3

- Clarified process control compound requirements in section 5.8
- Combined PDP Commodity Groupings in section 5.9
- Added guidance for LOD establishment for zero noise instruments in section 5.10.2.2
- Changed multi-peak compound LOD requirements in section 5.10.2.5
- Changed LOD verification requirements in section 5.11.4
- Specified PT samples larger than routine analytical samples may be run in duplicate in section 5.19.1.5
- Clarified what constitutes unacceptable PT scores in section 5.19.3.2
- Updated Attachment 2 by adding the following compounds: 2,4-DMPF, DEET, Dialofos, Dioxathion, Endothall, Indaziflam, Leptophos o-analog, Metconazole, Quinalphos, Saflufencil, Triazophos

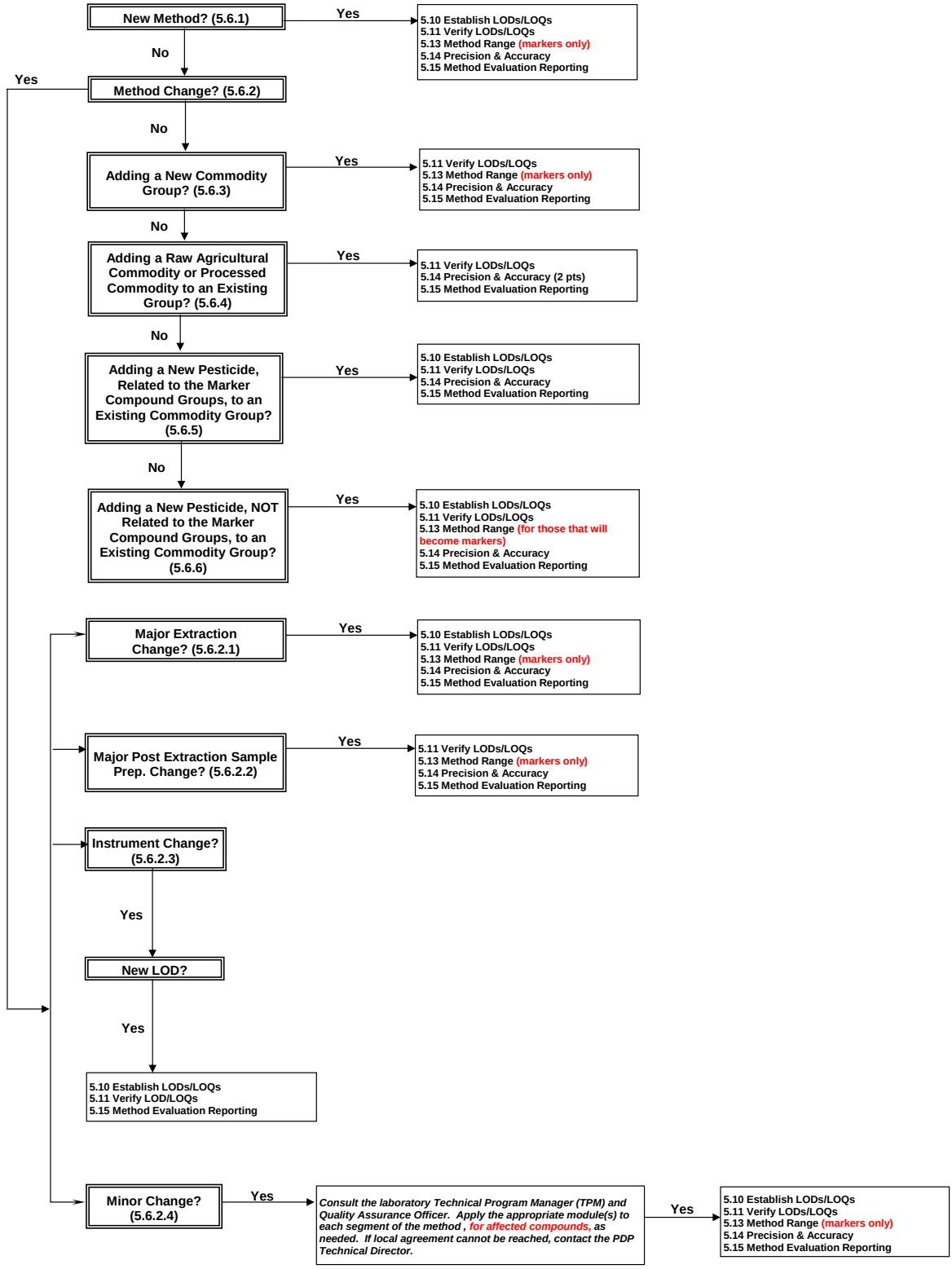
Revision 2 July 2011 Monitoring Programs Division

- Updated the entire SOP with the new program's name Monitoring Programs Division or MP instead of MPO
- Updated the Reference list.
- Removed the word "**Note"** in sections 5.2.1.2; 5.6.2.3; 5.6.4; 5.10.2; 5.20 leaving the paragraphs as instructions
- Updated section 5.2.7 by renumbering the subsections
- Added new commodities (Baby Foods, Papaya, Tangerines, Cherry Tomatoes, Snap Peas, Canned Beets) to 5.9
- Updated sections 5.15.5.3, 5.16.2.5 about approving MPAs
- Added the E code in section 5.17.8
- Updated requirements for section 5.18.2 by eliminating subsection 5.18.2.1
- Updated sections 5.18.2.2.1, 5.18.2.2.2 about MPAs (re)designation, replacing the letter of deviation with e-mail communication
- Updated section 5.18.2.2.2 by replacing the letter of deviation requirement with an e-mail communication
- Updated sections 5.19.1 and 5.19.2, by replacing "Ultra/GLEC" with "Ultra"
- Updated section 5.19.3.1 with new FAPAS requirements regarding z-scores
- In section 5.19.1.3 replaced "collected by GLEC" with "provided by MP"
- Updated Attachment 2 by adding a Group 4, Benzothiazoles/triazolones, to 'PDP Compound Groups for Fruit and Vegetables' list
- Updated Attachment 2 by adding the following compounds: Fosthiazate, Iprovalicarb, Rimsulfuron, Trifloxysulfuron, Uniconazole to PDP Pesticides Codes list
- Updated Attachment 3, 4 with new commodities: Baby Foods, Papaya, Tangerines, Cherry Tomatoes, Snap Peas, Canned Beets

Revision 1 July 2010 Monitoring Programs Office

- Renumbered the entire SOP replacing the sections' letters with numbers.
- Redefined Priority 1-4 compounds.
- Updared section 5.2.8 for Working Dilutions/Mixed Standards.
- Changed and updated section 5.5 Method Validation Evaluation Guidelines.
- Changed and updated section 5.6 Method Validation Scenarios.
- Updated section 5.7 Marker Pesticides to remove mandatory markers.
- Updated section 5.9 PDP Commodity Groupings.
- Removed section 5.12 LOD Check.
- Added section 5.13.6 Method Range Extension.
- Updated section 5.15.3 as part of Method Evaluation Reporting.
- Removed section 5.16.e.4
- Removed section 5.18.d.1.b
- Updated section 5.19.c (now 5.18.2) Matrix Spike Criteria.
- Updated section 5.19.e (now 5.18.3) Response to Failure To Meet Matrix Spike Criteria Range.
- Updated section 5.19.d (now 5.18.5) Response to Failure To Meet Chosen Process Control Criteria Range.
- Updated Attachment 1.
- Updated Attachment 2, by adding the following new compounds: Avermectin B_1, Bensulide oxygen analog, Cyhalofop butyl, Dimethipin, Disulfoton oxygen analog, Disulfoton oxygen analog sulfone, Disulfoton oxygen analog sulfoxide, Eprinomectin, Fenobucarb (BPMC), Flubendiamine, Flufenoxuron, Fluopicolide, Imidacloprid urea, Mandipropamid, Metaldehyde, Milbemectin, Pinoxaden, Promecarb, Prothioconazole, Pyrasulfotole, Pyridalyl, Tepraloxydim.
- Updated Attachments 3, 4 and 5.

Method Evaluation Flowchart



Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

PDP Compound Groups for Fruit and Vegetables

Group	Description
1	Phthalimides, conazoles and metabolites, carbamaldehydes, phenyl pyrroles, methoxy-acetamides, and neonicotinyls
2	Cyano/nitrile group(s) attached to double bond
3	Halogenated aromatics and chlorinated cyclics/cyclodienes
4	Benzothiazoles/triazolones
7	Dinitroanilines
8	Pyrethroids and metabolites and synergists
9	Triazines
11	Organophosphates and metabolites
14	Carbamates, thiocarbamates and metabolites
16	Uracils/ureas, imidazolinones, diacylhydrazines, and sulfonyl ureas
17	Nitrogenous heterocyclics
20	Phenoxy acids, ethanesulfonic acids (ESA), and oxanilic acids(OA)
21	Oxyhydrocarbons
22	Strobilurins
27	Tetronic acids
28	Cyclohexenone oxime
29	Macrocyclic lactones
99	Single

Note: Missing group numbers are attributed to the consolidation of groups. For example, Group 15, Thiocarbamates, was consolidated into Group 14, Carbamates.

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
1-naphthol	90-15-3	$C_{10}H_8O$	carbamate metabolite	14	382
1,2,4-triazole	288-88-0	$C_2H_3N_3$	triazole metabolite	1	A68
1,3-dichloropropene	542-75-6	$C_3H_4Cl_2$	fumigant nematicide		251
2,4-DB	94-82-6	$C_{10}H_{10}Cl_2O_3$	phenoxy acid	20	317
2,4-D	94-75-7	$C_8H_6Cl_2O_3$	phenoxy acid	20	026
2,4-dimethylphenyl formamide (DMPF)	60397-77-5	$C_9H_{11}NO$	amidine	2	AGR
2,4,5-T	93-76-5	$C_8H_5Cl_3O_3$	phenoxy acid	20	312
2,6-DIPN	24157-81-1	$C_{16}H_{20}$	substituted naphthalene	99	AFZ
3-hydroxycarbofuran	16655-82-6	$C_{12}H_{15}NO_4$	carbamate metabolite	14	512
4-dimethylaminosulphotoluidide (DMST); tolylfluanid metabolite	66840-71-9	$C_9H_{14}N_2O_2S$	phenylsulfamidemetabolite	1	AJU
5-hydroxythiabendazole	948-71-0	$C_{10}H_8N_3OS$	carbamate	1	B28
Abamectin	71751-41-2	$C_{48}H_{72}O_{14} + C_{47}H_{70}O_{14}$	avermectin (macrocyclic lactone)	29	948
Acephate	30560-19-1	$C_4H_{10}NO_3PS$	phosphoramidothioic acid	11	204
Acequinocyl	57960-19-7	$C_{24}H_{32}O_4$	unclassified acaricides	99	AKS
Acetamide	60-35-5	C_2H_5NO	amide	1	AAT
Acetamiprid	160430-64-8	$C_{10}H_{11}ClN_4$	neonicotinyls	1	B80
Acetochlor	34256-82-1	$C_{14}H_{20}ClNO_2$	chloroacetanilide	1	807
Acetochlor ethanesulfonic acid	187022-11-3	$C_8H_{21}NO_5S$	chloroacetanilide metabolite	20	ABN
Acetochlor oxanilic acid	194992-44-4	$C_{14}H_{19}NO_4$	chloroacetanilide metabolite	20	ABO
Acibenzolar-S-methyl	135158-54-2	$C_8H_6N_2OS_2$	thiadiazole	1	B51
Acifluorfen	50594-66-6	$C_{14}H_{21}NO_5S$	diphenyl ether	3	727
Aclonifen	74070-46-5	$C_{12}H_9ClN_2O_3$	nitrophenyl ether	3	D58
Acrinathrin	103833-18-7	$C_{26}H_{21}F_6NO_5$	pyrethroid	8	A03
Alachlor	15972-60-80	$C_{14}H_{20}ClNO_2$	acetamide	1	227
Alachlor ethanesulfonic acid	142363-53-9	$C_8H_{21}NO_5S$	chloroacetanilide metabolite	20	ABP
Alachlor oxanilic acid	171262-17-2	$C_{14}H_{19}NO_4$	chloroacetanilide metabolite	20	ABQ

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Aldicarb	116-06-3	$C_7H_{14}N_2O_2S$	carbamate	14	167
Aldicarb sulfone	1646-88-4	$C_7H_{14}N_2O_4S$	carbamate	14	168
Aldicarb sulfoxide	1646-87-3	$C_7H_{14}N_2O_3S$	carbamate	14	169
Aldrin	309-00-2	$C_{12}H_8Cl_6$	cyclodiene	3	001
Allethrin	584-79-2	$C_{19}H_{26}O_3$	pyrethroid	8	002
Allidochlor	93-71-0	$C_8H_{12}ClNO$	amide	1	768
Ametoctradin	865318-97-4	$C_{15}H_{25}N_5$	triazolopyrimidine fungicides	1	AKC
Ametryn	834-12-8	$C_9H_{17}N_5S$	triazine	9	156
Amicarbazone	129909-90-6	$C_{10}H_{19}N_5O_2$	triazolone	4	AGK
Aminomethylphosphonic acid (AMPA)	1066-51-9	CH_6NO_3P	organophosphate metabolite	99	957
Aminopyralid	150114-71-9	$C_6H_4Cl_2N_2O_2$	pyradine	20	AGO
Amitraz	33089-61-1	$C_{19}H_{23}N_3$	amidine	2	233
Anilazine	101-05-3	$C_9H_5Cl_3N_4$	triazine	9	033
Anilofos	64249-01-0	$C_{13}H_{19}ClNO_3PS_2$	organophosphate	11	D62
Aspon	3244-90-4	$C_{12}H_{28}O_5P_2S_2$	organophosphate	11	816
Asulam	3337-71-1	$C_8H_{10}N_2O_4S$	sulfonamide	14	ANG
Atraton	1610-17-9	$C_9H_{17}N_5O$	methoxytriazine	9	D64
Atrazine	1912-24-9	$C_8H_{14}ClN_5$	triazine	9	305
Avermectin B_1	71751-41-2	$C_{48}H_{72}O_{14}$ (avermectin B_{1a}) + $C_{47}H_{70}O_{14}$ (avermectin B_{1b})	macrocyclic lactone	29	AHQ
Aviglycine HCl	55720-26-8	$C_6H_{12}N_2O_3$	ethylene inhibitors	99	AKT
Azinphos ethyl	2642-71-9	$C_{12}H_{16}N_3O_3PS_2$	organophosphate	11	547
Azinphos methyl	86-50-0	$C_{10}H_{12}N_3O_3PS_2$	benzotriazine	11	042
Azinphos methyl oxygen analog	961-22-8	$C_{10}H_{12}N_3O_4PS$	oxon	11	769
Azoxystrobin	997888-88-8	$C_{22}H_{17}N_3O_5$	strobilurin	22	B48

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Barban	101-27-9	$C_{11}H_9Cl_2NO_2$	carbanilate	14	716
Benalaxyl	71626-11-4	$C_{20}H_{23}NO_3$	anilide fungicides	1	B45
Benazolin	3813-05-6	$C_9H_6ClNO_3S$	benzothiazole herbicides	4	832
Bendiocarb	22781-23-3	$C_{11}H_{13}NO_4$	carbamate	14	658
Beflubutamid	113614-08-7	$C_{18}H_{17}F_4NO_2$	amide	1	D69
Benfluralin	1861-40-1	$C_{13}H_{16}F_3N_3O_4$	dinitroaniline	7	191
Benomyl	17804-35-2	$C_{14}H_{18}N_4O_3$	benzimidazole	14	192
Benoxacor	98730-04-2	$C_{11}H_{11}Cl_2NO_2$	benzoxazine	1	A05
Bensulfuron methyl	83055-99-6	$C_{16}H_{18}N_4O_7S$	sulfonyl urea	16	ABR
Bensulide	741-58-2	$C_{14}H_{24}NO_4PS_3$	organophosphate	11	239
Bensulide oxygen analog	20243-81-6	$C_{14}H_{24}NO_4PS_3$	organophosphate	11	740
Bentazon	25057-89-0	$C_{10}H_{12}N_2O_3S$	thiadiazinone dioxide	17	758
Benthiavalicarb-isopropyl	177406-68-7	$C_{15}H_{18}FN_3O_3S$	benzothiazole	4	AGP
Benzovindiflupyr	1072957-71-1	$C_{18}H_{15}Cl_2F_2N_3O$	pyrazole	1	F53
BHC alpha	319-84-6	$C_6H_6Cl_6$	hexane ring	3	903
BHC beta	319-85-7	$C_6H_6Cl_6$	hexane ring	3	904
BHC, delta	319-86-8	$C_6H_6Cl_6$	hexane ring	3	905
BHC, epsilon	6108-10-7	$C_6H_6Cl_6$	hexane ring	3	ALH
Bifenazate	149877-41-8	$C_{17}H_{20}N_2O_3$	hydrazine carboxylate	14	B82
Bifenox	42576-02-3	$C_{14}H_9Cl_2NO_5$	nirophenyl ether herbicides	3	728
Bifenthrin	82657-04-3	$C_{23}H_{22}ClF_3O_2$	pyrethroid	8	930
Bioallethrin	260359-57-7	$C_{19}H_{26}O_3$	pyrethroid	8	ANP
Bitertanol	55179-31-2	$C_{20}H_{23}N_3O_2$	triazole	1	850
Boscalid	188425-85-6	$C_{18}H_{12}Cl_2N_2O$	anilide/pyridine	1	B75
Bromacil	314-40-9	$C_9H_{13}BrN_2O_2$	uracil	16	153
Bromophos ethyl	4824-78-6	$C_8H_8BrCl_2O_3PS$	organophosphate	11	602

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Bromopropylate	18181-80-1	$C_{17}H_{16}Br_2O_3$	bridged diphenyl	3	523
Bromoxynil	1689-84-5	$C_7H_3Br_2NO$	phenol	20	729
Bromuconazole-46	NA[2]	$C_{13}H_{12}BrCl_2N_3O$	conazole	1	ADU
Bromuconazole-47	NA[2]	$C_{13}H_{12}BrCl_2N_3O$	conazole	1	ADV
Bupirimate	41483-43-6	$C_{13}H_{24}N_4O_3S$	pyrimidine	17 or 3	872
Buprofezin	69327-76-0	$C_{16}H_{23}N_3OS$	thiadiazinone	17	B52
Butachlor	23184-66-9	$C_{17}H_{26}ClNO_2$	chloroacetanilide	1	806
Butocarboxim	34681-10-2	$C_7H_{14}N_2O_2S$	oxime carbamate	14	857
Butocarboxim sulfone	NA[2]	$C_7H_{14}N_2O_4S$	oxime carbamate metabolite	14	AKN
Butocarboxim sulfoxide	34681-24-8	$C_7H_{14}N_2O_3S$	oxime carbamate metabolite	14	AKO
Butylate	2008-41-5	$C_{11}H_{23}NOS$	thiocarbamate	14	783
Cadusafos	95465-99-9	$C_{10}HOPS_2$	phosphorodithionate	11	953
Captafol	2939-80-2	$C_{10}H_9Cl_4NO_2S$	phthalimide	1	174
Captan	133-06-2	$C_9H_8Cl_3NO_2S$	phthalimide	1	011
Carbaryl	63-25-2	$C_{12}H_{11}NO_2$	carbamate	14	102
Carbendazim	10605-21-7	$C_9H_9N_3O_2$	benzimidazole	14	666
Carbofuran	1563-66-2	$C_{12}H_{15}NO_3$	carbamate	14	180
Carbophenothion	786-19-6	$C_{11}H_{16}ClO_2PS_3$	organophosphate	11	202
Carbophenothion methyl	953-17-3	$C_9H_{12}ClO_2PS_3$	organophosphate	11	AGZ
Carboxin	5234-68-4	$C_{12}H_{13}NO_2S$	carboxamide	1	210
Carfentrazone ethyl	128639-02-1	$C_{15}H_{14}Cl_2F_3N_3O_3$	fluorophenyl triazole	4	B21
Chloramben	133-90-4	$C_7H_5Cl_2NO_2$	benzoic acid	20	952
Chlorantraniliprole	500008-45-7	$C_{18}H_{14}BrCl_2N_5O_2$	diamide; pyrazole	1	AGW
Chlordane cis	5103-71-9	$C_{10}H_6Cl_8$	cyclodiene	3	173
Chlordane trans	5103-74-2	$C_{10}H_6Cl_8$	cyclodiene	3	172
Chlordimeform	6164-98-3	$C_{10}H_{13}ClN_2$	formamidine	3	278
Chlorethoxyfos	54593-83-8	$C_6H_{11}Cl_4O_3PS$	phosphorothioate	11	A15

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Chlorfenapyr	122453-73-0	$C_{15}H_{11}BrClF_3N_2O$	pyrrole	1	B13
Chlorfenvinphos total	470-90-6	$C_{12}H_{14}Cl_3O_4P$	organophosphate	11	AAK
Chlorimuron ethyl	90982-32-4	$C_{15}H_{15}ClN_4O_6S$	sulfonyl urea	16	717
Chlorobenzilate	510-15-6	$C_{16}H_{14}Cl_2O_3$	bridged dipehnyl	3	015
Chloroneb	2675-77-6	$C_8H_8Cl_2O_2$	chlorobenzene	3	196
Chlorothalonil	1897-45-6	$C_8Cl_4N_2$	phthalimide	2	164
Chlorpropham	101-21-3	$C_{10}H_{12}ClNO_2$	carbamate	14	114
Chlorpyrifos	2921-88-2	$C_9H_{11}Cl_3NO_3PS$	phosphorothionic acid	11	160
Chlorpyrifos methyl	5598-13-0	$C_7H_7Cl_3NO_3PS$	phosphorothionic	11	235
Chlorpyrifos methyl O-analog	5598-52-7	$C_7H_7Cl_3NO_4P$	oxon	11	AAZ
Chlorpyrifos oxygen analog	5598-15-2	$C_9H_{11}Cl_3NO_4P$	oxon	11	772
Chlorsulfuron	64902-72-3	$C_{12}H_{12}ClN_5O_4S$	triazinylsulfonyl urea	16	718
Chlorthiophos	60238-56-4	$C_{11}H_{15}Cl_2O_3PS_2$	organophosphate	3	545
Chlozolinate	84332-86-5	$C_{13}H_{11}Cl_2NO_5$	dichlorophenyl dicarboxamide; oxazole	1	AJS
Clethodim	99129-21-2	$C_{17}H_{26}ClNO_3S$	cyclohexene oxime	28	AER
Clethodim 5-hydroxy sulfone	111031-11-9	$C_{17}H_{26}ClNO_6S$	cyclohexene oxime metabolite	28	AJM
Clethodim sulfone	111031-17-5	$C_{17}H_{26}ClNO_5S$	cyclohexene oxime metabolite	28	AJN
Clethodim sulfoxide	111031-14-2	$C_{17}H_{26}ClNO_4S$	cyclohexene oxime metabolite	28	AJO
Clodinafop propargyl	105512-06-9	$C_{17}H_{13}ClFNO_4$	aryloxyphenoxypropionic acid	20	B38
Clofencet	129025-54-3	$C_{13}H_{11}ClN_2O_3$	unclassified	99	AET
Clofentezine	74115-24-5	$C_{14}H_8Cl_2N_4$	tetrazine	99	699
Clomazone	81777-89-1	$C_{12}H_{14}ClNO_2$	pyridazone	17	719
Clopyralid	1702-17-6	$C_6H_3Cl_2NO_2$	pyridinecarboxylic acid	20	B46
Cloquintocet methyl	99607-70-2	$C_{18}H_{22}ClNO_3$	herbicide safener	1	AKE
Cloquintocet mexyl	99607-70-2	$C_{18}H_{22}ClNO_3$	herbicide safener	1	B39
Cloransulam methyl	147150-35-4	$C_{15}H_{13}ClFN_5O_5S$	triazolopyrimidine herbicides	1	ALP

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Clothianidin	210880-92-5	$C_6H_8ClN_5O_2S$	neonicotinyl	1	AEP
Coumaphos	56-72-4	$C_{14}H_{16}ClO_5PS$	phosphorothioate	11	124
Coumaphos oxygen analog	321-54-0	$C_{14}H_{16}ClO_6P$	oxon	11	614
CPOPA (5-(4-Chlorophenyl)oxazole-2-propionic Acid)	23464-95-1	$C_{12}H_{10}ClNO_3$	propionic acid	20	ALQ
Crotoxyphos	7700-17-6	$C_{14}H_{19}O_6P$	organophosphate	11	267
Crufomate	299-86-5	$C_{12}H_{19}ClNO_3P$	phosphoramidate	11	667
Cumyluron	99485-76-4	$C_{17}H_{19}ClN_2O$	Urea	16	ANJ
Cyanazine	21725-46-2	$C_9H_{13}ClN_6$	triazine	9	228
Cyantraniliprole	736994-63-1	$C_{19}H_{14}BrClN_6O_2$	pyrazole	1	AMB
Cyazofamid	120116-88-3	$C_{13}H_{13}ClN_4O_2S$	imidazole	1	AGA
Cyclanilide	113136-77-9	$C_{11}H_9Cl_2NO_3$	unclassified plant growth regulator	1	A81
Cyclaniliprole	1031756-98-5	$C_{21}H_{17}Br_2Cl_2N_5O_2$	pyrazole	1	F40
Cycloate	1134-23-2	$C_{11}H_{21}NOS$	thiocarbamate	14	232
Cyflufenamid	180409-60-3	$C_{20}H_{17}F_5N_2O_2$	amide	1	AKU
Cyflumetofen	400882-07-7	$C_{24}H_{24}F_3NO_4$	bridged dipehnyl	3	AMC
Cyfluthrin	68359-37-5	$C_{22}H_{18}Cl_2FNO_3$	pyrethroid	8	781
Cyhalofop butyl	122008-85-9	$C_{20}H_{20}FNO_4$	aryloxyphenoxypropionic herbicide	17	B59
Cyhalothrin (lambda)	91465-08-6	$C_{23}H_{19}ClF_3NO_3$	pyrethroid	8	AEM
Cyhalothrin (lambda epimer R157836)	68085-85-8	$C_{23}H_{19}ClF_3NO_3$	pyrethroid	8	AEN
Cyhalothrin total (L-cyhalothrin + R157836 epim	68085-85-8	$C_{23}H_{19}ClF_3NO_3$	pyrethroid	8	AEL
Cymoxanil	57966-95-7	$C_7H_{10}N_4O_3$	cyanoacetamide	2	877
Cypermethrin	52315-07-8	$C_{22}H_{19}Cl_2NO_3$	pyrethroid	8	597
Cyphenothrin	39515-40-7	$C_{24}H_{25}NO_3$	pyrethroid	8	ADH
Cyproconazole	94361-06-5	$C_{15}H_{18}ClN_3O$	conazole	1	A22
Cyprodinil	121552-61-2	$C_{14}H_{15}N_3$	anilinopyrimidine	17	B22
Cyprosulfamide	221667-31-8	$C_{18}H_{18}N_2O_5S$	herbicide safeners	1	AMD

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Cyromazine	66215-27-8	$C_6H_{10}N_6$	triazine	9	255
DCPA	1861-32-1	$C_{10}H_6Cl_4O_4$	phthalic acid	3	134
DCPA mono acid	887-54-7	$C_9H_4Cl_4O_4$	dicarboxylic acid	20	ABV
DDD o,p'	53-19-0	$C_{14}H_{10}Cl_4$	bridged biphenyl	3	909
DDD p,p'	72-54-8	$C_{14}H_{10}Cl_4$	bridged biphenyl	3	908
DDE o,p'	3424-82-6	$C_{14}H_8Cl_4$	bridged biphenyl	3	911
DDE p,p'	72-55-9	$C_{14}H_8Cl_4$	bridged biphenyl	3	910
DDT o,p'	789-02-6	$C_{14}H_9Cl_5$	bridged biphenyl	3	907
DDT p,p'	50-29-3	$C_{14}H_9Cl_5$	bridged biphenyl	3	906
DEET (N,N-diethyl-m-toluamide)	134-62-3	$C_{12}H_{17}NO$	amide	2	PBS
DEF (Tribufos)	78-48-8	$C_{12}H_{27}OPS_3$	organophosphate	11	217
Deltamethrin (includes parent Tralomethrin)	52918-63-5	$C_{22}H_{19}Br_2NO_3$	pyrethroid	8	612
Demeton	8065-48-3	$C_8H_{19}O_3PS_2$	phosphorothioate	11	023
Demeton-S	126-75-0	$C_8H_{19}O_3PS_2$	organothiophosphate	11	558
Demeton-S sulfone	2496-91-5	$C_8H_{19}O_5PS_2$	organothiophosphate metabolite	11	226
Desethyl atrazine	6190-65-4	$C_6H_{10}ClN_5$	triazine metabolite	9	964
Desethyl-desisopropyl atrazine	3397-62-4	$C_3H_4ClN_5$	triazine metabolite	9	784
Desisopropyl atrazine	1007-28-9	$C_5H_8ClN_5$	triazine metabolite	9	785
Desmedipham	13684-56-5	$C_{16}H_{16}N_2O_4$	carbamate	14	786
Desmetryn	1014-69-3	$C_8H_{15}N_5S$	methylthiotriazine	9	A88
Dialifos	10311-84-9	$C_{14}H_{17}ClNO_4PS_2$	organothiophosphate	11	244
Diazinon	333-41-5	$C_{12}H_{21}N_2O_3PS$	phosphorothioate	11	024
Diazinon oxygen analog	962-58-3	$C_{12}H_{21}N_2O_4P$	oxon	11	395
Dicamba	1918-00-9	$C_8H_6Cl_2O_3$	benzoic acid	20	155
Dichlobenil	1194-65-6	$C_7H_3Cl_2N$	nitrile	2	324
Dichlofenthion	97-17-6	$C_{10}H_{13}Cl_2O_3PS$	phenylorganothiophosphate	11	664
Dichlofluanid	1085-98-9	$C_9H_{11}Cl_2FN_2O_2S_2$	phenylsulfamide	1	588

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Dichlormid	37764-25-3	$C_8H_{11}Cl_2NO$	herbicide safeners	1	A43
Dichlorprop	120-36-5	$C_9H_8Cl_2O_3$	phenoxy acid	20	A25
Dichlorvos (DDVP)	62-73-7	$C_4H_7Cl_2O_4P$	phosphoric acid	11	338
Diclofop methyl	51338-27-3	$C_{16}H_{14}Cl_2O_4$	aryloxyphenoxypropionic acid	20	299
Dicloran	99-30-9	$C_6H_4Cl_2N_2O_2$	nitroaniline	7	144
Diclosulam	145701-21-9	$C_{13}H_{10}Cl_2FN_5O_3S$	triazolopyrimidine herbicides	1	ALU
Dicofol o,p'	10606-46-9	$C_{14}H_9Cl_5O$	bridged biphenyl	3	253
Dicofol p,p'	115-32-2	$C_{14}H_9Cl_5O$	bridged biphenyl	3	254
Dicrotophos	141-66-2	$C_8H_{16}NO_5P$	organophosphate	11	209
Dieldrin	60-57-1	$C_{12}H_8Cl_6O$	cyclodiene	3	028
Diethofencarb	87130-20-9	$C_{14}H_{21}NO_4$	carbanilite fungicides	22	B62
Difenoconazole	119446-68-3	$C_{19}H_{17}Cl_2N_3O_3$	triazole	1	B58
Diflubenzuron	35367-38-5	$C_{14}H_9ClF_2N_2O_2$	urea	16	651
Diflufenzopyr	109293-97-2	$C_{15}H_{12}F_2N_4O_3$	urea	16	AFY
Dimepiperate	61432-55-1	$C_{15}H_{21}NOS$	unclassified	99	E13
Dimethenamid	87674-68-8	$C_{12}H_{18}ClNO_2S$	acetamide	1	ADD
Dimethenamid ethanesulfonic acid	205939-58-8	$C_{12}H_{19}NO_5S_2$	acetamide metabolite	20	AEX
Dimethenamid oxanilic acid	NA[2]	$C_{12}H_{17}NO_4S$	acetamide metabolite	20	AEY
Dimethenamid P	87674-68-8	$C_{12}H_{18}ClNO_2S$	amide	1	AEB
Dimethipin	55290-64-7	$C_6H_{10}O_4S_2$	urea	16	787
Dimethoate	60-51-5	$C_5H_{12}NO_3PS_2$	phosphorodithionic acid	11	171
Dimethomorph	110488-70-5	$C_{21}H_{22}ClNO_4$	chlorophenyl morpholine	3	B77
Diniconazole	83657-24-3	$C_{15}H_{17}Cl_2N_3O$	conazole	1	AFN
Dinocap	131-72-6	$C_{18}H_{24}N_2O_6$	dinitrophenol	20	315
Dinoseb	88-85-7	$C_{10}H_{12}N_2O_5$	phenol	20	031
Dinotefuran	165252-70-0	$C_7H_{14}N_4O_3$	neonicotinyl	1	AFO
Dioxacarb	6988-21-2	$C_{11}H_{13}NO_4$	phenylmethyl carbamate	14	656

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Dioxathion	78-34-2	$C_{12}H_{26}O_6P_2S_4$	organothiophosphate	11	103
Diphacinone	82-66-6	$C_{23}H_{16}O_3$	indandione	99	B99
Diphenamid	957-51-7	$C_{16}H_{17}NO$	acetamide	1	330
Diphenylamine (DPA)	122-39-4	$C_{12}H_{11}N$	amine	3	125
Dipropetryn	4147-51-7	$C_{11}H_{21}N_5S$	triazine	9	735
Disulfoton	298-04-4	$C_8H_{19}O_2PS_3$	phosphorodithioate	11	117
Disulfoton oxygen analog	NA[2]	$C_8H_{19}O_3PS_2$	organophosphate	11	AHN
Disulfoton oxygen analog sulfone	NA[2]	$C_6H_{15}O_5PS_2$	organophosphate	11	AHV
Disulfoton oxygen analog sulfoxide	NA[2]	$C_6H_{15}O_4PS_2$	organophosphate	11	AHW
Disulfoton sulfone	2497-06-5	$C_8H_{19}O_4PS$	sulfone	11	216
Disulfoton sulfoxide	2497-07-6	$C_8H_{19}O_3PS_3$	sulfoxide	11	706
Ditalimfos	5131-24-8	$C_{12}H_{14}NO_4PS$	organophosphorus	11	E22
Dithianon	3347-22-6	$C_{14}H_4N_2O_2S_2$	quinine	17	AHO
Dithiopyr	97886-45-8	$C_{15}H_{16}F_5NO_2S_2$	pyridine	20	E24
Diuron	330-54-1	$C_9H_{10}Cl_2N_2O$	urea	16	032
Dodine	2439-10-3	$C_{15}H_{33}N_3O_2$	aliphatic nitrogenous fungicide	2	104
Emamectin benzoate	155569-91-8	$C_{49}H_{75}NO_{13}$ + $C_{48}H_{73}NO_{13}$	avermectin (macrocyclic lactone)	29	AGH
Endosulfan I	959-98-8	$C_9H_6Cl_6O_3S$	cyclodiene	3	900
Endosulfan II	33213-65-9	$C_9H_6Cl_6O_3S$	cyclodiene	3	901
Endosulfan sulfate	1031-07-8	$C_9H_6Cl_6O_4S$	cyclodiene	3	902
Endrin	72-20-8	$C_{12}H_8Cl_6O$	cyclodiene	3	034
Endothall	145-73-3	$C_8H_{10}O_5$	dicarboxylic acid	21	AKV
Epoxiconazole	135319-73-2	$C_{17}H_{13}ClFN_3O$	conazole	1	B53
Eprinomectin	123997-26-2	$C_{50}H_{75}NO_{14}$ (eprinomectin B_{1a}) + $C_{49}H_{73}NO_{14}$ (eprinomectin B_{1b})	macrocyclic lactone	29	AHR

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
EPN	2104-64-5	$C_{14}H_{14}NO_4PS$	phenyl phenylphosphonothioate insecticides	11	035
EPTC	759-94-4	$C_9H_{19}NOS$	thiocarbamate	14	200
Esfenvalerate	66230-04-4	$C_{25}H_{22}ClNO_3$	pyrethroid	8	714
Ethaboxam	162650-77-3	$C_{14}H_{16}N_4OS_2$	amide	1	C90
Ethalfluralin	55283-68-6	$C_{13}H_{14}F_3N_3O_4$	dinitroaniline	7	721
Ethephon	16672-87-0	$C_2H_6ClO_3P$	plant growth regulator	11	730
Ethiofencarb	29973-13-5	$C_{11}H_{15}NO_2S$	carbamate	14	858
Ethiofencarb sulfone	53380-23-7	$C_{11}H_{15}NO_4S$	carbamate	14	AMX
Ethiofencarb sulfoxide	53380-22-6	$C_{11}H_{15}NO_3S$	carbamate	14	AMY
Ethion	563-12-2	$C_9H_{22}O_4P_2S_4$	phosphorodithioic acid	11	107
Ethion di oxon	22756-17-8	$C_9H_{22}O_6P_2S_2$	oxon	11	538
Ethion mono oxon	17356-42-2	$C_9H_{22}O_5P_2S_3$	oxon	11	AAX
Ethiprole	181587-01-9	$C_{13}H_9Cl_2F_3N_4OS$	phenylpyrazole	1	AME
Ethofumesate	26225-79-6	$C_{13}H_{18}O_5S$	benzofuranyl alkylsulfonate	11	945
Ethoprop	13194-48-4	$C_8H_{19}O_2PS_2$	dipropyl phosphorodithioate	11	175
Ethoxyquin	91-53-2	$C_{14}H_{19}NO$	quinoline	99	111
Ethylan	72-56-0	$C_{18}H_{20}Cl_2$	organochlorine	3	066
Etofenprox	80844-07-1	$C_{25}H_{28}O_3$	pyrethroid ether	8	ADI
Etoxazole	1532333-91-1	$C_{21}H_{23}F_2NO_2$	oxazole	1	B84
Etridiazole	2593-15-9	$C_5H_5Cl_3N_2OS$	thiadiazole	1	722
Etrimfos	38260-54-7	$C_{10}H_{17}N_2O_4PS$	pyrimidine organothiophosphate	11	293
Famoxadone	131807-57-3	$C_{22}H_{18}N_2O_4$	dicarboximide/oxazole	1	AEW
Famphur	52-85-7	$C_{10}H_{16}NO_5PS_2$	phenylorganothiophosphate	11	603
Fenamidone	161326-34-7	$C_{17}H_{17}N_3OS$	imidazole	1	B64
Fenamiphos	22224-92-6	$C_{13}H_{22}NO_3PS$	phosphoramidate	11	236
Fenamiphos sulfone	31972-44-8	$C_{13}H_{22}NO_5PS$	sulfone	11	745

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Fenamiphos sulfoxide	31972-43-7	$C_{13}H_{22}NO_4PS$	sulfoxide	11	746
Fenarimol	60168-88-9	$C_{17}H_{12}Cl_2N_2O$	pyrimidine	3	271
Fenazaquin	120928-09-8	$C_{20}H_{22}N_2O$	unclassified acaricide	27	B73
Fenbuconazole	114369-43-6	$C_{19}H_{17}ClN_4$	conazole	1	A30
Fenbutatin oxide	13356-08-6	$C_{60}H_{28}OSn_2$	organotin acaride	99	639
Fenchlorphos (ronnel)	299-84-3	$C_8H_8Cl_3O_3PS$	phenyl organothiophosphate	11	105
Fenhexamid	126833-17-8	$C_{14}H_{17}Cl_2NO_2$	chlorocarboximide	1	B41
Fenitrothion	122-14-5	$C_9H_{12}NO_5PS$	phosphorothioate	11	391
Fenitrothion oxygen analog	2255-17-6	$C_9H_{12}NO_6P$	oxon	11	648
Fenobucarb (BPMC)	3766-81-2	$C_{12}H_{17}NO_2$	phenyl methylcarbamate	14	856
Fenoxaprop ethyl	66441-23-4	$C_{18}H_{16}ClNO_5$	aryloxyphenoxypropionic acid	20	777
Fenoxycarb	72490-01-8	$C_{17}H_{19}NO_4$	carbamate	14	811
Fenpicoxamid	517875-34-2	$C_{31}H_{38}N_2O_{11}$	amide	1	F92
Fenpropathrin	39515-41-8	$C_{22}H_{23}NO_3$	pyrethroid	8	808
Fenpropidin	67306-00-7	$C_{19}H_{31}N$	nitrogenous hetercyclic	17	AMF
Fenpropimorph	67564-91-4	$C_{20}H_{33}NO$	morpholine	3	886
Fenpyrazamine	473798-59-3	$C_{17}H_{21}N_3O_2S$	pyrazole	1	AMG
Fenpyroximate	111812-58-9	$C_{24}H_{27}N_3O_4$	phenoxypyrazol	1	AFS
Fensulfothion	115-90-2	$C_{11}H_{17}O_4PS_2$	phenyl organothiophosphate	11	243
Fenthion	55-38-9	$C_{10}H_{15}O_3PS_2$	phosphorothioate	11	177
Fenthion oxygen analog	6552-12-1	$C_{10}H_{15}O_4PS$	oxon	11	691
Fenthion sulfone	3761-42-0	$C_{10}H_{15}O_5PS_2$	organophosphate	11	660
Fenthion sulfoxide	3761-41-9	$C_{10}H_{15}O_4PS_2$	organophosphate	11	AKP
Fenuron	101-42-8	$C_9H_{12}N_2O$	urea	16	840
Fenvalerate	51630-58-1	$C_{25}H_{22}ClNO_3$	pyrethroid	8	546
Fipronil	120068-37-3	$C_{12}H_4Cl_2F_6N_4OS$	phenyl pyrazole	1	A82
Fipronil sulfone	120068-36-2	$C_{12}H_4Cl_2F_6N_4O_2S$	phenylpyrazole	1	A84

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Flazasulfuron	104040-78-0	$C_{13}H_{12}F_3N_5O_5S$	pyridiminyl sulfonylurea	16	AMH
Flonicamid	158062-67-0	$C_9H_6F_3N_3O$	nicotinoid	1	AGG
Fluazifop	69335-91-7	$C_{15}H_{12}F_3NO_4$	pyridine	17	ALW
Fluazifop butyl	69806-50-4	$C_{15}H_{12}F_3NO_4$	pyridine	17	292
Fluazinam	79622-59-6	$C_{13}H_4Cl_2F_6N_4O_4$	pyridine	17	B54
Flubendiamide	272451-65-7	$C_{23}H_{22}F_7IN_2O_4S$	diamide	17 or 1	AHS
Flucythrinate	70124-77-5	$C_{26}H_{23}F_2NO_4$	pyrethroid	8	229
Fludioxonil	131341-86-1	$C_{12}H_6F_2N_2O_2$	phenyl pyrrole	1	B23
Fluensulfone	318290-98-1	$C_7H_5ClF_3NO_2S_2$	unclassified nematicide	99	ANK
Flufenacet	142459-58-3	$C_{14}H_{13}F_4N_3O_2S$	anilide	1	B30
Flufenacet ethanesulfonic acid	NA[2]	$C_{11}H_{14}FNO_4S$	anilide metabolite	20	AFH
Flufenacet oxanilic acid	201668-31-7	$C_{11}H_{12}FNO_3$	anilide metabolite	20	AEZ
Flufenoxuron	101463-69-8	$C_{21}H_{11}ClF_6N_2O_3$	urea	16	AHG
Flufenpyr ethyl	188489-07-8	$C_{16}H_{13}ClF_4N_2O_4$	pyridazinone	17	ALR
Flumetralin	62924-70-3	$C_{16}H_{12}ClF_4N_3O_4$	growth inhibitor	7	834
Flumetsulam	98967-40-9	$C_{12}H_9F_2N_5O_2S$	pyrimidine	1	AAU
Flumiclorac pentyl	87546-18-7	$C_{21}H_{23}ClFNO_5$	dicarboximide	1	AAV
Flumioxazin	103361-09-7	$C_{19}H_{15}FN_2O_2$	N-phenylphthalimide	1	AFF
Fluometuron	2164-17-2	$C_{10}H_{11}F_3N_2O$	urea	16	701
Fluopicolide	239110-15-7	$C_{14}H_8Cl_3F_3N_2O$	pyridine	17 or 1	AHT
Fluopyram	658066-35-4	$C_{16}H_{11}ClF_6N_2O$	benzamide	1	AKG
Fluorodifen	15457-05-3	$C_{13}H_7F_3N_2O_5$	nitrophenyl ether	3	836
Fluoxastrobin	361377-29-9	$C_{21}H_{16}ClFN_4O_5$	strobilurin	22	AGJ
Flupyradifurone	951659-40-8	$C_{12}H_{11}ClF_2N_2O_2$	unclassified	1	ANE
Fluquinconazole	136426-54-5	$C_{16}H_8Cl_2FN_5O$	aryloxyphenoxypropionic acid	1	B78
Fluridone	59756-60-4	$C_{19}H_{14}F_3NO$	pyridine	17	736
Fluroxapyr-1-methylheptyl ester	81406-37-3	$C_{15}H_{22}Cl_2FN_2O_3$	pyridine	17	ADJ

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Flusilazole	85509-19-9	$C_{16}H_{15}F_2N_3Si$	conazole	1	950
Fluthiacet methyl	117337-19-6	$C_{15}H_{15}ClFN_3O_3S_2$	phenylpyrazole	1	AGM
Flutolanil	66332-96-5	$C_{17}H_{16}F_3NO_2$	caboxamide	1	B63
Flutriafol	76674-21-0	$C_{16}H_{13}F_2N_3O$	conazole	1	AFM
Fluvalinate	69409-94-5	$C_{26}H_{22}ClF_3N_2O_3$	pyrethroid	8	297
Fluxapyroxad	907204-31-3	$C_{18}H_{12}F_5N_3O$	anilide; pyrazole	22	AKW
Folpet	133-07-3	$C_9H_4Cl_3NO_2S$	phthalimide	1	126
Fomesafen	72178-02-0	$C_{15}H_{10}ClF_3N_2O_6S$	amide or nitrophenyl ether herbicide	1 or 3	ALX
Fonofos	944-22-9	$C_{10}H_{15}OPS_2$	phosphorodithioic acid	11	163
Fonofos oxygen analog	944-21-8	$C_{10}H_{15}O_2PS$	oxon	11	692
Forchlorfenuron	68157-60-8	$C_{12}H_{10}ClN_3O$	phenyl urea	16	B32
Formetanate hydrochloride	23422-53-9	$C_{11}H_{15}N_3O_2$	formamidine	1	723
Fosthiazate	98886-44-3	$C_9H_{18}NO_3PS_2$	organothiophosphate	11	B09
Furalaxyl	57646-30-7	$C_{17}H_{19}NO_4$	furanilide	1	AMZ
Furathiocarb	65907-30-4	$C_{18}H_{26}N_2O_5S$	benzofurayl methyl carbamate	14	AMR
Glufosinate	77182-82-2	$C_5H_{12}NO_4P$	quaternary ammonium	99	AJL
Glyphosate	1071-83-6	$C_3H_8NO_5P$	organophosphate	99	653
Halosulfuron	135397-30-7	$C_{12}H_{13}ClN_6O_7S$	sulfonyl urea	16	AFK
Halosulfuron methyl	100784-20-1	$C_{12}H_{13}ClN_6O_7S$	sulfonyl urea	16	AEH
Haloxyfop	69806-34-4	$C_{15}H_{11}ClF_3NO_4$	aryloxyphenoxypropionic acid	20	798
Heptachlor	76-44-8	$C_{10}H_5Cl_7$	cyclodiene	3	044
Heptachlor epoxide	1024-57-3	$C_{10}H_5Cl_7O$	cyclodiene	3	143
Heptenophos	23560-59-0	$C_9H_{12}ClO_4P$	organophosphate	11	841
Hexachlorobenzene (HCB)	118-74-1	C_6Cl_6	benzene ring	3	321
Hexaconazole	79983-71-4	$C_{14}H_{17}Cl_2N_3O$	conazole	1	954
Hexaflumuron	86479-06-3	$C_{16}H_8Cl_2F_6N_2O_3$	benzoylphenylurea chitin synthesis inhib	16	AMA
Hexazinone	51235-04-2	$C_{12}H_{20}N_4O_2$	triazine	9	633

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Hexythiazox	78587-05-0	$C_{17}H_{21}ClN_2O_2S$	thiazolidine carboxamide	1	B10
Hydroprene	41096-46-2	$C_{17}H_{30}O_2$	oxyhydrocarbon	21	AEC
Hydroxy atrazine	2163-68-0	$C_8H_{15}N_5O$	triazine metabolite	9	AED
Imazalil	35554-44-0	$C_{14}H_{14}Cl_2N_2O$	conazole	1	604
Imazamethabenz acid	NA[2]	$C_{15}H_{18}N_2O_3$	imidazolinone	16	AEE
Imazamethabenz methyl	81405-85-8	$C_{16}H_{20}N_2O_3$	imidazolinone	16	753
Imazamox	114311-32-9	$C_{15}H_{19}N_3O_4$	imidazolinone	16	ACA
Imazapic	104098-48-8	$C_{14}H_{17}N_3O_3$	imidazolinone	16	ACZ
Imazapyr	81334-34-1	$C_{13}H_{15}N_3O_3$	imidazolinone	16	ACB
Imazaquin	81335-37-7	$C_{17}H_{17}N_3O_3$	imidazolinone	16	ACC
Imazethapyr	81335-77-5	$C_{15}H_{19}N_3O_3$	imidazolinone	16	ACD
Imazosulfuron	122548-33-8	$C_{14}H_{13}ClN_6O_5S$	sulfonyl urea	16	AMK
Imidacloprid	138261-41-3	$C_9H_{10}ClN_5O_2$	neonicotinyl	1	967
Imidacloprid urea	120868-66-8	$C_9H_{10}ClN_3O$	neonicotinyl metabolite	1	AHF
Imiprothrin	72963-72-5	$C_{17}H_{22}N_2O_4$	pyrethroid	8	ADK
Indaziflam	950782-86-2	$C_{16}H_{20}FN_5$	triazine	9	AJP
Indoxacarb	173584-44-6	$C_{22}H_{17}ClF_3N_3O_7$	carbamate	14	ADG
Iodosulfuron methyl	144550-36-7	$C_{14}H_{14}IN_5O_6S$	triazinylsulfonyl urea	16	AKB
Ipconazole	125225-28-7	$C_{18}H_{24}ClN_3O$	conazole	1	AHY
Iprobenfos	26087-47-8	$C_{13}H_{21}O_3PS$	organophosphate	11	867
Iprodione	36734-19-7	$C_{13}H_{13}Cl_2N_3O_3$	dicarboximide	1	626
Iprodione metabolite isomer	63637-89-8	$C_{13}H_{13}Cl_2N_3O_3$	dicarboximide	1	231
Iprovalicarb	140923-17-7	$C_{18}H_{28}N_2O_3$	carbamates	14	AGE
Isocarbophos	24353-61-5	$C_{11}H_{16}NO_4PS$	phosphoramidothioate	11	ALE
Isofenphos	25311-71-1	$C_{15}H_{24}NO_4PS$	organophosphate	11	258
Isofenphos methyl	99675-03-3	$C_{14}H_{22}NO_4PS$	phosphoramidothioate	11	ANA
Isofenphos oxygen analog	31120-85-1	$C_{15}H_{24}NO_5P$	oxon	11	655

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Isofetamid	875915-78-9	$C_{20}H_{25}NO_3S$	amide/thiophene	1	ANH
Isoprocarb	2631-40-5	$C_{11}H_{15}NO_2$	carbamate	14	637
Isoprothiolane	50512-35-1	$C_{12}H_{18}O_4S_2$	unclassified	99	855
Isoproturon	34123-59-6	$C_{12}H_{18}N_2O$	phenyl urea	16	843
Isopyrazam	881685-58-1	$C_{20}H_{23}F_2N_3O$	pyrazole	1	AKX
Isoxadifen ethyl	163520-33-0	$C_{18}H_{17}NO_3$	herbicide safeners	1	AGL
Isoxaflutole	141112-29-0	$C_{15}H_{12}F_3NO_4S$	cyclopropylisoxazole	17	B15
Kasugamycin	6980-18-3	$C_{14}H_{25}N_3O_9$	bactericide	99	AKY
Kinoprene	42588-37-4	$C_{18}H_{28}O_2$	oxyhydrocarbon	21	E66
Kresoxim methyl	143390-89-0	$C_{18}H_{19}NO_4$	strobilurin	22	B42
Lactofen	77501-63-4	$C_{19}H_{15}ClF_3NO_7$	flurodiphenyl ether	3	A38
Leptophos oxygen analog	25006-32-0	$C_{13}H_{10}BrCl_2O_3P$	oxon	11	A40
Lenacil	2164-08-1	$C_{13}H_{18}N_2O_2$	uracil	16	859
Lindane (BHC gamma)	58-89-9	$C_6H_6Cl_6$	hexane ring	3	050
Linuron	330-55-2	$C_9H_{10}Cl_2N_2O_2$	urea	16	129
Lufenuron	103055-07-8	$C_{17}H_8Cl_2F_8N_2O_3$	benzoylphenylurea	16	AJV
Malathion	121-75-5	$C_{10}H_{19}O_6PS_2$	phosphorodithioate	11	052
Malathion oxygen analog	1634-78-2	$C_{10}H_{19}O_7PS$	oxon	11	208
Mancozeb	8018-01-07	$C_8H_{12}MnN_4S_8Zn$	dithiocarbamate	99	128
Mandipropamid	374726-62-2	$C_{23}H_{22}ClNO_4$	amide	1	AGX
MCPA	94-74-6	$C_9H_9ClO_3$	phenoxy	20	318
MCPB	94-81-5	$C_{11}H_{13}ClO_3$	phenoxy acid	20	620
Mecarbam	2595-54-2	$C_{10}H_{20}NO_5PS_2$	organophosphate	11	662
Mecoprop (MCPP)	7085-19-0	$C_{10}H_{11}ClO_3$	phenoxy acid	20	A42
Mefenacet	73250-68-7	$C_{16}H_{14}N_2O_2S$	anilide	1	D21
Mefenpyr diethyl	135590-91-9	$C_{16}H_{18}Cl_2N_2O_4$	herbicide safeners	1	AKH
Melamine	108-78-1	$C_3H_6N_6$	trimer of cyanamide	9	260

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Mepanipyrim	1102335-47-7	$C_{14}H_{13}N_3$	pyrimidine	17	AGF
Mephosfolan	950-10-7	$C_8H_{16}NO_3PS_2$	phosphoramidate	11	242
Merphos	150-50-5	$C_{12}H_{27}PS_3$	plant growth regulator	11	121
Mesosulfuron methyl	208465-21-8	$C_{17}H_{21}N_5O_9S_2$	pyrimidinylsulfonyl urea	16	AKJ
Mesotrione	104206-82-8	$C_{14}H_{13}NO_7S$	benzoylcyclohexanedione	1	AJA
Metaflumizone	139968-49-3	$C_{24}H_{16}F_6N_4O_2$	unclassified	1	AJW
Metalaxyl	57837-19-1	$C_{15}H_{21}NO_4$	acylalanine	1	607
Metaldehyde	108-62-3	$C_8H_{16}O_4$	polyaldehyde	99	B07
Metconazole	125116-23-6	$C_{17}H_{22}ClN_3O$	conazole	1	AHX
Metolcarb	1129-41-5	$C_9H_{11}NO_2$	carbamate	14	860
Metrafenone	220899-03-6	$C_{19}H_{21}BrO_5$	arylphenylketone	3	ANF
Methamidophos	10265-92-6	$C_2H_8NO_2PS$	phosphoramidothioic acid	11	170
Methacrifos	62610-77-9	$C_7H_{13}O_5PS$	organothiophosphate	11	E74
Methidathion	950-37-8	$C_6H_{11}N_2O_4PS_3$	phosphorodithioate	11	197
Methidathion oxygen analog	39856-16-1	$C_6H_{11}N_2O_5PS_2$	oxon	11	ACE
Methiocarb	2032-65-7	$C_{11}H_{15}NO_2S$	carbamate	14	195
Methiocarb sulfone	2179-25-1	$C_{11}H_{15}NO_4S$	carbamate metabolilte	14	634
Methiocarb sulfoxide	2635-10-1	$C_{11}H_{15}NO_3S$	carbamate metabolilte	14	256
Methomyl	16752-77-5	$C_5H_{10}N_2O_2S$	carbamate	14	159
Methoprene	40596-69-8	$C_{19}H_{34}O_3$	oxyhydrocarbon	21	ACV
Methoxychlor olefin	2132-70-9	$C_{16}H_{14}Cl_2O_2$	bridged biphenyl	3	276
Methoxychlor p,p'	72-43-5	$C_{16}H_{15}Cl_3O_2$	bridged biphenyl	3	275
Methoxychlor Total	72-43-5	$C_{16}H_{15}Cl_3O_2$	bridged biphenyl	3	055
Methoxyfenozide	161050-58-4	$C_{22}H_{28}N_2O_3$	diacylhydrazine	16	AES
Methyl pentachlorophenyl sulfide (MPCPS)	1825-19-0	$C_7H_3Cl_5S$	benzene ring	3	388
Metolachlor	51218-45-2	$C_{15}H_{22}ClNO_2$	acetamide	1	283
Metolachlor ethanesulfonic acid	NA[2]	$C_{15}H_{23}NO_5S$	chloroacetanilide metabolite	20	ACG

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Metolachlor oxanilic acid	152019-73-3	$C_{15}H_{21}NO_4$	chloroacetanilide metabolite	20	ACH
Metribuzin	21087-64-9	$C_8H_{14}N_4OS$	triazines	9	181
Metsulfuron methyl	74223-64-6	$C_{14}H_{15}N_5O_6S$	sulfonyl urea	16	ACI
Mevinphos E/Z	298-01-1	$C_7H_{13}O_6P$	butenoic acid	11	579
MGK-264	113-48-4	$C_{17}H_{25}NO_2$	synergist	8	058
MGK-326 (Dipropyl isocinchomeronate)	136-45-8	$C_{13}H_{17}NO_4$	synergist	8	ADL
Milbemectin	51596-10-2	$C_{31}H_{44}O_7$ (milbemycin A_3) + $C_{32}H_{46}O_7$ (milbemycin A_4)	macrocyclic lactone	29	AHP
Mirex	2385-85-5	$C_{10}Cl_{12}$	cyclodiene	3	352
Molinate	2212-67-1	$C_9H_{17}NO_5$	thiocarbamate	14	778
Monocrotophos	6923-22-4	$C_7H_{14}NO_5P$	phosphoric acid	11	343
Monolinuron	1746-81-2	$C_9H_{11}ClN_2O_2$	phenyl urea	16	682
Monuron	150-68-5	$C_9H_{11}ClN_2O$	urea	16	046
Myclobutanil	88671-89-0	$C_{15}H_{17}ClN_4$	triazole	1	679
Naled	300-76-5	$C_4H_7Br_2Cl_2O_4P$	organophosphate	11	303
Napropamide	15299-99-7	$C_{17}H_{21}NO_2$	amide	1	594
Naptalam (Alanap)	132-66-1	$C_{18}H_{13}NO_3$	amide	1	B18
Neburon	555-37-3	$C_{12}H_{16}C_{l2}N_2O$	urea	16	061
Niclosamide	50-65-7	$C_{13}H_8Cl_2N_2O_4$	molluscicide	3	ACL
Nicosulfuron	111991-09-4	$C_{15}H_{18}N_6O_6S$	sulfonyl urea	16	ACM
Nitrapyrin	1929-82-4	$C_6H_3Cl_4N$	pyridine	17	725
Nitrofen	1836-75-5	$C_{12}H_7Cl_2NO_3$	nitrophenyl ether herbicides	3	158
Norflurazon	27314-13-2	$C_{12}H_9ClF_3N_3O$	pyridazinone	17	596
Norflurazon desmethyl	23576-24-1	$C_{11}H_7ClF_3N_3O$	pyridazinone	17	720
Novaluron	116714-46-6	$C_{17}H_9ClF_8N_2O_4$	benzoyl urea	16	AFX
Omethoate	1113-02-6	$C_5H_{12}NO_4PS$	phosphorothioate	11	178
o-Phenylphenol	90-43-7	$C_{12}H_{10}O$	biphenyl	3	083

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Oryzalin	19044-88-3	$C_{12}H_{18}N_4O_6S$	dinitroaniline	7	737
Oxadiazon	19666-30-9	$C_{15}H_{18}Cl_2N_2O_3$	oxadiazon	1	625
Oxadixyl	77732-09-3	$C_{14}H_{18}N_2O_4$	oxazolidine	1	A46
Oxamyl	23135-22-0	$C_7H_{13}N_3O_3S$	carbamate	14	537
Oxamyl oxime	30558-43-1	$C_5H_{10}N_2O_2S$	carbamate	14	A47
Oxathiapiprolin	1003318-67-9	$C_{24}H_{22}F_5N_5O_2S$	pyrazole	1	F56
Oxychlordane	27304-13-8	$C_{10}H_4Cl_8O$	cyclodiene	3	349
Oxydemeton methyl	301-12-2	$C_6H_{15}O_4PS_2$	organophosphate	11	219
Oxydemeton methyl sulfone	17040-19-6	$C_6H_{15}O_5PS_2$	phosphorothioate	11	245
Oxyfluorfen	42874-03-3	$C_{15}H_{11}ClF_3NO_4$	diphenyl ether	3	713
Oxythioquinox	2439-01-2	$C_{10}H_6N_2OS_2$	quionoxaline	17	246
Paclobutrazol	76738-62-0	$C_{15}H_{20}ClN_3O$	growth inhibitor	1	A48
Parathion ethyl	56-38-2	$C_{10}H_{14}NO_5PS$	phosphorothionic acid	11	065
Parathion ethyl oxygen analog	NA[2]	$C_{10}H_{14}NO_6P$	oxon	11	370
Parathion methyl	298-00-0	$C_8H_{10}NO_5PS$	phosphorothionic acid	11	057
Parathion methyl oxygen analog	950-35-6	$C_8H_{10}NO_6P$	oxon	11	779
Pebulate	1114-71-2	$C_{10}H_{21}NOS$	thiocarbamate	14	161
Penconazole	66246-88-6	$C_{13}H_{15}Cl_2N_3$	conazole	1	956
Pencycuron	66063-05-6	$C_{19}H_{21}ClN_2O$	urea	16	AJX
Pendimethalin	40487-42-1	$C_{13}H_{19}N_3O_4$	dinitroaniline	7	230
Penflufen	494793-67-8	$C_{18}H_{24}FN_3O$	pyrazole	1	AKZ
Penoxsulam	219714-96-2	$C_{16}H_{14}F_5N_5O_5S$	triazolpyrimidine	1	AMS
Pentachloroaniline (PCA)	527-20-8	$C_6H_2Cl_5N$	aniline	3	351
Pentachlorobenzene (PCB)	608-93-5	C_6HCl_5	benzene ring	3	387
Penthiopyrad	183675-82-3	$C_{16}H_{20}F_3N_3OS$	pyridazinone	22	AKD
Permethrin cis	61949-76-6	$C_{21}H_{20}Cl_2O_3$	pyrethroid	8	222
Permethrin total	52645-53-1	$C_{21}H_{20}Cl_2O_3$	pyrethroid	8	539

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Permethrin trans	61949-77-7	$C_{21}H_{20}Cl_2O_3$	pyrethroid	8	223
Perthane	72-56-0	$C_{18}H_{20}Cl_2$	organochlorine	3	AGN
Phenmedipham	13684-63-4	$C_{16}H_{16}N_2O_4$	carbamate	14	791
Phenothrin	26002-80-2	$C_{23}H_{26}O_3$	pyrethroid	8	848
Phenthoate	2597-03-7	$C_{12}H_{17}O_4PS_2$	organophosphate	11	377
Phorate	298-02-2	$C_7H_{17}O_2PS_3$	phosphorodithionic acid	11	148
Phorate oxygen analog	2600-69-3	$C_7H_{17}O_3PS_2$	oxon	11	928
Phorate oxygen analog sulfone	2588-06-9	$C_7H_{17}O_5PS_2$	organophosphate	11	966
Phorate oxygen analog sulfoxide	2588-05-8	$C_7H_{17}O_4PS_2$	organophosphate	11	951
Phorate sulfone	2588-04-7	$C_7H_{17}O_4PS_3$	sulfone	11	189
Phorate sulfoxide	2588-03-6	$C_7H_{17}O_3PS_2$	sulfoxide	11	190
Phosalone	2310-17-0	$C_{12}H_{15}ClNO_4PS_2$	phosphorodithionic acid	11	166
Phosalone oxygen analog	2275-06-1	$C_{12}H_{15}ClNO_5PS$	oxon	11	929
Phosmet	732-11-6	$C_{11}H_{12}NO_4PS_2$	phosphorodithionic acid	11	165
Phosmet oxygen analog	3735-33-9	$C_{11}H_{12}NO_5PS$	oxon	11	237
Phosphamidon	13171-21-6	$C_{10}H_{19}ClNO_5P$	dimethyl phosphate	11	203
Phoxim	14816-18-3	$C_{12}H_{15}N_2O_3PS$	organothiophosphate	11	247
Picloram	1918-02-1	$C_6H_3Cl_3N_2O_2$	carboxylic acid	20	329
Picoxystrobin	117428-22-5	$C_{18}H_{16}F_3NO_4$	strobilurin	22	ALA
Pinoxaden	243973-20-8	$C_{23}H_{32}N_2O_4$	phenylpyrazole	22	AHH
Piperalin	3478-94-2	$C_{16}H_{21}Cl_2NO_2$	unclassified	99	AGV
Piperonyl butoxide	51-03-6	$C_{19}H_{30}O_5$	benzodioxole	8	070
Pirimicarb	23103-98-2	$C_{11}H_{18}N_4O_2$	carbamate	14	580
Pirimicarb desmethyl	30614-22-3	$C_{10}H_{16}N_4O_2$	dimethylcarbamate metabolite	14	873
Pirimiphos methyl	29232-93-7	$C_{11}H_{20}N_3O_3PS$	phosphorothioate	11	562
Prallethrin	23031-36-9	$C_{19}H_{24}O_3$	pyrethroid	8	ADC
Pretilachlor	51218-49-6	$C_{17}H_{26}ClNO_2$	chloroacetanilide	1	892

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Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Primisulfuron	113036-87-6	$C_{14}H_{10}F_4N_4O_7S$	pyrimidinylsulfonyl urea	16	AHA
Prochloraz	67747-09-5	$C_{15}H_{16}Cl_3N_3O_2$	imidazole	9	833
Procymidone	32809-16-8	$C_{13}H_{11}Cl_2NO_2$	dicarboximide	1	593
Prodiamine	29091-21-2	$C_{13}H_{17}F_3N_4O_4$	dinitroaniline	7	814
Profenofos	41198-08-7	$C_{11}H_{15}BrClO_3PS$	phosphorothioate	11	224
Profluralin	26399-36-0	$C_{14}H_{16}F_3N_3O_4$	dinitroaniline	7	A53
Profoxydim	139001-49-3	$C_{24}H_{32}ClNO_4S$	cyclohexene oxime	28	ANB
Prohexadione calcium	127277-53-6	$C_{20}H_{22}CaO_{10}$	unclassified plant growth regulator	99	ALO
Promecarb	2631-37-0	$C_{12}H_{17}NO_2$	phenyl methylcarbamate	14	385
Prothioconazole	178928-70-6	$C_{14}H_{15}Cl_2N_3OS$	conazole	1	AHJ
Prometon	1610-18-0	$C_{10}H_{19}N_5O$	triazine	9	942
Prometryn	7287-19-6	$C_{10}H_{19}N_5S$	triazine	9	249
Pronamide (propyzamide)	23950-58-5	$C_{12}H_{11}Cl_2NO$	amide	1	540
Propachlor	1918-16-7	$C_{11}H_{14}ClNO$	chloroacetanilide	1	675
Propachlor oxanilic acid	70628-36-3	$C_{11}H_{13}NO_3$	chloroacetanilide metabolite	20	AFA
Propamocarb hydrochloride	25606-41-1	$C_9H_{20}N_2O_2$	carbamate	14	AFU
Propanil	709-98-8	$C_9H_9Cl_2NO$	anilide	1	341
Propaquizafop	111479-05-1	$C_{22}H_{22}ClN_3O_5$	aryloxyphenoxypropionic acid	17	ALK
Propargite	2312-35-8	$C_{19}H_{26}O_4S$	sulfite	1	623
Propazine	139-40-2	$C_9H_{16}ClN_5$	triazine	9	333
Propetamphos	31218-83-4	$C_{10}H_{20}NO_4PS$	phosphorothioate	11	636
Propham	122-42-9	$C_{10}H_{13}NO_2$	carbamate	14	310
Propiconazole	60207-90-1	$C_{15}H_{17}Cl_2N_3O_2$	conazole	1	264
Propoxur	114-26-1	$C_{11}H_{15}NO_3$	carbamate	14	162
Propoxycarbazone	145026-81-9	$C_{15}H_{18}N_4O_7S$	triazolone	1	AKK
Proquinazid	189278-12-4	$C_{14}H_{17}IN_2O_2$	unclassified	17	AMM
Prosulfuron	94125-34-5	$C_{15}H_{16}F_3N_5O_4S$	triazinylsulfonyl urea	16	AEG

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Prothioconazole	178928-70-6	$C_{14}H_{15}Cl_2N_3OS$	conazole	1	AHJ
Prothiofos	34643-46-4	$C_{11}H_{15}Cl_2O_2PS_2$	phenylorganothiophosphate	11	613
Pydiflumetofen	1228284-64-7	$C_{16}H_{16}Cl_3F_2N_3O_2$	pyrazole	1	G03
Pymetrozine	123312-89-0	$C_{10}H_{11}N_5O$	azomethine	9	ABF
Pyraclofos	77458-01-6	$C_{14}H_{18}ClN_2O_3PS$	heterocyclic organothiophosphate	11	F01
Pyraclostrobin	175013-18-0	$C_{19}H_{18}ClN_3O_4$	strobilurin	22	B61
Pyraflufen	129630-17-7	$C_{13}H_9Cl_2F_3N_2O_4$	phenylpyrazole	1	ALY
Pyraflufen ethyl	129630-19-9	$C_{15}H_{13}Cl_2F_3N_2O_4$	phenoxypyrazole	1	AGB
Pyrasulfotole	365400-11-9	$C_{14}H_{13}F_3N_2O_4S$	benzoylpyrazole	1	AHK
Pyrazon (Chloridazon)	1698-60-8	$C_{10}H_8ClN_3O$	pyridazinone	17	595
Pyrazophos	13457-18-6	$C_{14}H_{20}N_3O_5PS$	organophosphate	11	553
Pyrethrins	8003-34-7	$C_{21}H_{27}O_4$	pyrethrum, botanical	8	075
Pyridaben	96489-71-3	$C_{19}H_{25}ClN_2OS$	pyridazinone	17	B56
Pyridalyl	179101-81-6	$C_{18}H_{14}Cl_4F_3NO_3$	pyridine	17	AHU
Pyridaphenthion	119-12-0	$C_{14}H_{17}N_2O_4PS$	organophosphate	11	961
Pyrimethanil	53112-28-0	$C_{12}H_{13}N_3$	pyrimidine	17	B16
Pyriproxyfen	95737-68-1	$C_{20}H_{19}NO_3$	pyridine	17	B24
Pyroxasulfone	447399-55-5	$C_{12}H_{14}F_5N_3O_4S$	pyrazole	1	AMO
Quinalphos	13593-03-8	$C_{12}H_{15}N_2O_3PS$	organothiophosphate	11	661
Quinchlorac	84087-01-4	$C_{10}H_5Cl_2NO_2$	quinolinecarboxylic acid	20	B29
Quinoxyfen	124495-18-7	$C_{15}H_8Cl_2FNO$	pyridine	17	B57
Quintozene (PCNB)	82-68-8	$C_6Cl_5NO_2$	benzene ring	3	304
Quizalofop	76578-12-6	$C_{17}H_{13}ClN_2O_4$	aryloxyphenoxypropionic hebicide	20	ALZ
Quizalofop ethyl	76578-14-8	$C_{19}H_{17}ClN_2O_4$	aryloxyphenoxypropionic acid	20	750
Resmethrin	10453-86-8	$C_{22}H_{26}O_3$	pyrethroid	8	556
RH 9129	146887-38-9	$C_{19}H_{16}N_3ClO_2$	fenbuconazole metabolite	1	A54
RH 9130	146887-37-8	$C_{19}H_{16}N_3ClO_2$	fenbuconazole metabolite	1	A55

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Rimsulfuron	122931-48-0	$C_{14}H_{17}N_5O_7S_2$	sulfonyl urea	16	AJF
Rotenone	83-79-4	$C_{23}H_{22}O_6$	botanical insecticide	8	020
S-(2-hydroxy)propyl EPTC	759-94-4	$C_9H_{19}NOS$	thiocarbamate	14	ACO
Saflufenacil	372137-35-4	$C_{17}H_{17}ClF_4N_4O_5S$	urea	16	AHZ
Sedaxane	874967-67-6	$C_{18}H_{19}F_2N_3O$	pyrazole	1	ALB
Sethoxydim	74051-80-2	$C_{17}H_{29}NO_3S$	cyclohexene oxime	28	AEV
Sethoxydim sulfoxide	114480-24-9	$C_{17}H_{29}NO_4S$	cyclohexene oxime	28	AJR
Siduron	1982-49-6	$C_{14}H_{20}N_2O$	urea	16	ACT
Simazine	122-34-9	$C_7H_{12}ClN_5$	triazine	9	149
Simetryn	1014-70-6	$C_8H_{15}N_5S$	triazine	9	837
Spinetoram	187166-40-1	$C_{42}H_{69}NO_{10} + C_{43}H_{69}NO_{10}$	spinosyn (macrocyclic lactone)	29	AGY
Spinosad	168316-95-8	$C_{41}H_{65}NO_{10} + C_{42}H_{67}NO_{10}$	spinosyn (macrocyclic lactone)	29	ABB
Spirodiclofen	148477-71-8	$C_{21}H_{24}Cl_2O_4$	tetronic acid	27	B85
Spiromesifen	283594-90-1	$C_{23}H_{30}O_4$	tetronic acid	27	AGT
Spiromesifen enol metabolite	148476-30-6	$C_{17}H_{20}O_3$	tetronic acid metabolite	27	AGU
Spiromesifen, total (including enol metabolite)	283594-90-1	$C_{23}H_{30}O_4 + C_{17}H_{20}O_3$	tetronic acid	27	AFW
Spirotetramat	203313-25-1	$C_{21}H_{27}NO_5$	tetramic acid insecticide	27	AHM
Spiroxamine	118134-30-8	$C_{18}H_{35}NO_2$	unclassified	99	AJY
Sulfallate	95-06-7	$C_8H_{14}ClNS_2$	thiocarbamate	14	323
Sulfentrazone	122836-35-5	$C_{11}H_{10}Cl_2F_2N_4O_3S$	triazole sulfonamide	1	AAY
Sulfometuron methyl	74222-97-2	$C_{15}H_{16}N_4O_5S$	sulfonyl urea	16	ACP
Sulfosulfuron	141776-32-1	$C_{16}H_{18}N_6O_7S_2$	pyrimidinylsulfonyl urea	16	ADS
Sulfotep	3689-24-5	$C_8H_{20}O_5P_2S_2$	organophosphate	11	311
Sulfoxaflor	946578-00-3	$C_{10}H_{10}F_3N_3OS$	sulfoximine	99	ALS
Sulprofos	35400-43-2	$C_{12}H_{19}O_2PS_3$	organophosphate	11	609
Sulprofos oxygen analog	38527-90-1	$C_{12}H_{19}O_3PS_2$	oxon	11	ACQ
TCMTB	21564-17-0	$C_9H_6N_2S_3$	benzothiazole	17	793

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Tebuconazole	107534-96-3	$C_{16}H_{23}ClN_3O$	conazole	1	A58
Tebufenozide	112410-23-8	$C_{22}H_{28}N_2O_2$	diacylhydrazine	16	ABG
Tebufenpyrad	119168-77-3	$C_{18}H_{24}ClN_3O$	pyrazole	1	AJZ
Tebupirimfos	96182-53-5	$C_{13}H_{23}N_2O_3PS$	organophosphate	11	A59
Tebupirimfos oxygen analog	NA[2]	$C_{13}H_{23}N_2O_4P$	oxon	11	ACR
Tebuthiuron	34014-18-1	$C_9H_{16}N_4OS$	urea	16	780
Tecnazene	117-18-0	$C_6HCl_4NO_2$	nitrobenzene	3	147
Teflubenzuron	83121-18-0	$C_{14}H_6Cl_2F_4N_2O_2$	benzoylphenylurea	16	AKA
Tefluthrin	79538-32-2	$C_{17}H_{14}ClF_7O_2$	pyrethroid	8	B26
TEPP	107-49-3	$C_8H_{20}O_7P_2$	organophosphate	11	088
Tepraloxydim	149979-41-9	$C_{17}H_{24}ClNO_4$	cyclohexene oxime	28	AHL
Terbacil	5902-51-2	$C_9H_{13}ClN_2O_2$	uracil	16	152
Terbufos	13071-79-9	$C_9H_{21}O_2PS_3$	phosphorothioate	11	205
Terbufos oxygen analog	56070-14-5	$C_9H_{21}O_3PS_2$	oxon	11	A60
Terbufos oxygen analog sulfone	56070-15-6	$C_9H_{21}O_5PS_2$	organophosphate	11	752
Terbufos sulfone	56070-16-7	$C_9H_{21}O_4PS_3$	sulfone	11	963
Terbufos Sulfoxide	10548-10-4	$C_9H_{21}O_4PS_2$	organophosphate	11	AMP
Terbutryn	886-50-0	$C_{10}H_{19}N_5S$	methylthiotriazine	9	738
Terbuthylazine	5915-41-3	$C_9H_{16}ClN_5$	chlorotriazine	9	678
Tetrachlorvinphos	22248-79-9	$C_{10}H_9Cl_4O_4P$	chlorethylene phosphate	11	176
Tetraconazole	112281-77-3	$C_{13}H_{11}Cl_2F_4N_3O$	conazole	1	B72
Tetradifon	116-29-0	$C_{12}H_6Cl_4O_2S$	bridged biphenyl	3	108
Tetrahydrophthalimide (THPI)[1]	1469-48-3	$C_8H_9NO_2$	phthalimide	1	624
Tetramethrin	7696-12-0	$C_{19}H_{25}NO_4$	pyrethroid	8	947
Thiabendazole	148-79-8	$C_{10}H_7N_3S$	benzimidazole	1	157
Thiacloprid	111988-49-9	$C_{10}H_9ClN_4S$	neonicotinyl	1	B68
Thiamethoxam	153719-23-4	$C_8H_{10}ClN_5O_3S$	neonicotinyl	1	B43

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Thiazopyr	117718-60-2	$C_{16}H_{17}F_5N_2O_2S$	pyridine	17	B12
Thidiazuron	51707-55-2	$C_9H_8N_4OS$	plant growth regulator	16	794
Thiencarbazone methyl	317815-83-1	$C_{12}H_{14}N_4O_7S_2$	triazolone	4	AKL
Thifensulfuron	79277-67-1	$C_{11}H_{11}N_5O_6S_2$	sulfonyl urea	16	AEF
Thifensulfuron methyl	79227-27-3	$C_{12}H_{13}N_5O_6S_2$	triazinylsulfonyl urea	16	AEQ
Thiobencarb	28249-77-6	$C_{12}H_{16}ClNOS$	thiocarbamate	14	726
Thiodicarb	59669-26-0	$C_{10}H_{18}N_4O_4S_3$	carbamate	14	943
Thionazin	297-97-2	$C_8H_{13}N_2O_3PS$	organophosphate	11	250
Thiophanate methyl	23564-05-8	$C_{12}H_{14}N_4O_4S_2$	carbamate	14	611
Thiram	137-26-8	$C_6H_{12}N_2S_4$	dithiocarbamate	99	089
Thymol	89-83-8	$C_{10}H_{14}O$	phenol	3	ALG
Tolclofos methyl	57018-04-9	$C_9H_{11}Cl_2O_3PS$	organophosphate	11	B70
Tolfenpyrad	129558-76-5	$C_{21}H_{22}ClN_3O_2$	pyrazole	1	ANC
Tolyfluanid	731-27-1	$C_{10}H_{13}Cl_2FN_2O_2S_2$	phenylsulfamide	1	649
Topramezone	210631-68-8	$C_{16}H_{17}N_3O_5S$	benzoylpyrazole	1	AMO
Toxaphene	8001-35-2	$C_{10}H_{10}Cl_8$	organochlorine	3	090
Tralomethrin	66841-25-6	$C_{22}H_{19}Br_4NO_3$	pyrethroid	8	755
Triadimefon	43121-43-3	$C_{14}H_{16}ClN_3O_2$	conazole	1	608
Triadimenol	55219-65-3	$C_{14}H_{18}ClN_3O_2$	conazole	1	638
Triallate	2303-17-5	$C_{10}H_{16}Cl_3NOS$	thiocarbamate	14	621
Triasulfuron	82097-50-5	$C_{14}H_{16}ClN_5O_5S$	sulfonyl urea	16	ADP
Triazole acetic acid	28711-29-7	$C_4H_6N_3O_2$	triazole metabolite	1	ADX
Triazole alanine	86362-20-1	$C_5H_8N_4O_2$	triazole metabolite	1	ADW
Triazophos	24017-47-8	$C_{12}H_{16}N_3O_3PS$	organothiophosphate	11	536
Tribenuron methyl	101200-48-0	$C_{15}H_{17}N_5O_6S$	triazinylsulfonyl urea	16	ACS
Trichlorfon (as dichlorvos)	52-68-6	$C_4H_8Cl_3O_4P$	phosphate	11	130
Trichloronate	327-98-0	$C_{10}H_{12}Cl_3O_2PS$	organothiophosphate	11	569

PDP Pesticide Codes and Multi-residue Compound Groupings for Fruit and Vegetables

Compound Name	CAS#	Molecular Formula	Chemical Family	Group	Pesticide Code
Triclopyr	55335-06-3	$C_7H_4Cl_3NO_3$	acetic acid	20	731
Tricyclazole	41814-78-2	$C_9H_7N_3S$	benzothiazole	4	804
Tridemorph	24602-86-6	$C_{19}H_{39}NO$	morpholine	3	795
Trifloxystrobin	221007-60-9	$C_{20}H_{19}F_3N_2O_4$	strobilurin	22	B79
Trifloxysulfuron	145099-21-4	$C_{14}H_{14}F_3N_5O_6S$	sulfonyl urea	16	AJG
Triflumizole	68694-11-1	$C_{15}H_{15}ClF_3N_3O$	conazole	1	A61
Trifluralin	1582-09-8	$C_{13}H_{16}F_3N_3O_4$	dinitroaniline	7	151
Triforine	26644-46-2	$C_{10}H_{14}Cl_6N_4O_2$	formamide	1	915
Triticonazole	131983-72-7	$C_{17}H_{20}ClN_3O$	conazole	1	ADR
Uniconazole	83657-22-1	$C_{15}H_{18}ClN_3O$	conazole	1	AJJ
Vernolate	1929-77-7	$C_{10}H_{21}NOS$	thiocarbamate	14	201
Vinclozolin	50471-44-8	$C_{12}H_9Cl_2NO_3$	dichloroanilide	1	529
Zoxamide	156052-68-5	$C_{14}H_{16}Cl_3NO_2$	benzamide	1	B44

[1] Metabolite of captan and captafol.
[2] Not available.

EPA, Codex, and Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) Commodity Groupings

Commodity		PDP Code	PDP Commodity Group	EPA	Codex	PAM	
Almonds		AL	Cereal Grains (High Oil)	Tree nuts	Tree nuts	N/A	
Apples	Fruit	AP	Fruits and Vegetables	Pome fruits	Pome fruits	Non-fatty	Med. Sugar
	Juice	AJ			Fruit juice		Med. Sugar
	Sauce	AC			Manufactured food single ingredient		High Sugar
	Baby Food	IA			Manufactured food single ingredient		
	Single serving	AX			Pome fruits		Med. Sugar
Asparagus		AS	Fruits and Vegetables	Miscellaneous	Stalk & stem vegs.	Non-fatty	Low Sugar
Avocado		AV	Fruits and Vegetables	Miscellaneous	Assorted tropical & sub-tropical fruits - inedible peel	Fatty	Low Sugar
Bananas		BN	Fruits and Vegetables	Miscellaneous	Assorted tropical & sub-tropical fruits - inedible peel	Non-fatty	High Sugar
Barley		BY	Cereal Grains (Low Oil)	Cereal grains	Cereal grains	Non-fatty	Low Water
Basil		BS	Fruits and Vegetables	Herbs & Spices	Herbs & Spices	N/A	
Beans, canned	Black	BC	Fruits and Vegetables	Legume vegs.	Legume vegs.	Non-fatty	Low Water
	Garbanzo (Chick pea)	ZB				Fatty	Low Sugar
	Garbanzo (Chick pea) Dried	ZD				Fatty	Low Sugar
	Kidney	BC				Non-fatty	Low Water
	Pinto	BC				Non-fatty	Low Sugar
Beets		BT	Fruits and Vegetables	Root & Tuber vegs.	Root & tuber vegs.	Non-fatty	N/A
Beef	Adipose	BA	Animal Tissue/High Protein	Meat	Meat	N/A	
	Liver	BL					
	Muscle	BM					
Blueberry		BB	Fruits and Vegetables	Berry & Small Fruit	Berries & other small fruits	Non-fatty	Med. Sugar
Broccoli		BR	Fruits and Vegetables	Brassica leafy vegs.	Brassica leafy vegs.	Non-fatty	Low Sugar
Butter		BU	Dairy Products	Dairy	Derived milk products	Fatty	Low Water
Cabbage		CG	Fruits and Vegetables	Brassica leafy vegs.	Brassica leafy vegs.	Non-fatty	Low Sugar
Cantaloupe		CN	Fruits and Vegetables	Cucurbits	Cucurbits	Non-fatty	Med. Sugar
Carrots	Fresh	CR	Fruits and Vegetables	Root & tuber vegs.	Root & tuber vegs.	Non-fatty	Med. Sugar
	Baby Food	IC			Manufactured food single ingredient		
Cauliflower		CF	Fruits and Vegetables	Brassica leafy vegs.	Brassica leafy vegs.	Non-fatty	Low Sugar
Celery		CE	Fruits and Vegetables	Leafy vegs.	Stalk & stem vegs.	Non-fatty	Low Sugar
Cherries, Sweet		CH	Fruits and Vegetables	Stone fruits	Stone fruits	Non-fatty	Med. Sugar
Cilantro		CL	Fruits and Vegetables	Herbs & Spices	Herbs & Spices	N/A	

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

EPA, Codex, and Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) Commodity Groupings

Commodity		PDP Code	PDP Commodity Group	EPA	Codex	PAM	
Corn	Grain	CO	Cereal Grains (Low Oil)	Cereal grains	Cereal grains	Fatty	Low Water
	Sweet, Fresh	CB	Fruits and Vegetables		Cereal grains	Non-fatty	Med. Sugar
	Sweet, Frozen	CS					
	Sweet, Canned	CD					
	Syrup	CY	Single Commodities		Derived edible plant products	N/A	
Cranberry	Fresh/Frozen	CA	Fruits and Vegetables	Berry & Small Fruit	Berries & other small fruits	Non-fatty	N/A
	Canned	RC					
Cream, heavy		CM	Dairy Products	Dairy	Derived milk products	Fatty	Low Sugar
Cucumbers		CU	Fruits and Vegetables	Cucurbits	Cucurbits	Non-fatty	Low Sugar
Egg		EG	Animal Tissue/High Protein	Miscellaneous	Poultry products	Non-fatty	N/A
Eggplant		EP	Fruits and Vegetables	Fruiting vegs.	Fruiting vegs.	Non-fatty	Low Sugar
Fish, Catfish		FC	Single Commodities or	Miscellaneous	Aquatic animal products	Fatty	No sugar
Fish, Salmon		FS	Animal Tissue/High Protein	Miscellaneous	Aquatic animal products	Fatty	No sugar
Grapefruit		GF	Fruits and Vegetables	Citrus fruits	Citrus fruits	Non-fatty	Med. Sugar
Grapes	Fruit	GR	Fruits and Vegetables	Berry & Small Fruit	Berries & other small fruits	Non-fatty	High Sugar
	Juice	GJ			Fruit juice		Med. Sugar
Green Beans	Raw, fresh	GB	Fruits and Vegetables	Legume vegs.	Legume vegs.	Non-fatty	Low Sugar
	Baby Food	IG			Manufactured food single ingredient		
Greens	Collard	GS	Fruits and Vegetables	Brassica leafy vegs.	Leafy vegs. (including Brassica leafy vegs.)	Non-fatty	Low Sugar
	Kale	GK					
	Mustard	MG					
Honey		HY	Single Commodities	Miscellaneous		Non-fatty	High Sugar
Honey Dew Melon		HD	Fruits and Vegetables	Cucurbits	Cucurbits	Non-fatty	N/A
Infant formula, dairy-based		DF	Single Commodities		Manufactured food multiple ingredient	N/A	N/A
Infant formula, soy-based		YF	Single Commodities		Manufactured food multiple ingredient	N/A	N/A
Kiwi Fruit, Fresh		KW	Fruits and Vegetables	Berry & Small Fruit	Assorted tropical & sub-tropical fruits - inedible peel	Non-fatty	Med. Sugar
Lemons		LM	Fruits and Vegetables	Citrus fruits	Citrus fruits	Non-fatty	Low Sugar
Lettuce	Bunch	LT	Fruits and Vegetables	Leafy vegs.	Leafy vegs.	Non-fatty	Low Sugar
	Bagged	LB					
Mangoes		MA	Fruits and Vegetables	Miscellaneous	Assorted tropical & sub-tropical fruits - inedible peel	Non-fatty	Med. Sugar
Milk, whole		MK	Dairy Products	Dairy	Milks	Fatty	Low Sugar
Mushrooms		MU	Fruits and Vegetables	Edible fungi	Fruiting vegs.	Non-fatty	Low Sugar
Nectarines		NE	Fruits and Vegetables	Stone fruits	Stone fruits	Non-fatty	Med. Sugar
Oats		OA	Cereal Grains (Low Oil)	Cereal grains	Cereal grains	Fatty	Low Water
Onions	Bulb	ON	Fruits and Vegetables	Bulb vegs.	Bulb vegs.	Non-fatty	Low Sugar
	Green	GO					
Orange	Fruit	OG	Fruits and Vegetables	Citrus fruits	Citrus fruits	Non-fatty	Med. Sugar
	Juice	OJ			Fruit juice		

EPA, Codex, and Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) Commodity Groupings

Commodity		PDP Code	PDP Commodity Group	EPA	Codex	PAM	
Olives	Fruit	OL	Fruits and Vegetables	Tropical & sub-tropical fruits -	Tropical & sub-tropical fruits - edible peel	N/A	N/A
Peaches	Canned	CC	Fruits and Vegetables	Stone fruits	Stone fruits	Non-fatty	Med. Sugar
	Fruit	PC					
	Single serving	CX					
	Baby Food	IH			Manufactured food single ingredient		
Papaya		YA	Fruits and Vegetables	Miscellaneous	Assorted tropical & sub-tropical fruits - inedible peel	Non-fatty	Med. Sugar
Peanut Butter		PB	Cereal Grains (High Oil)	Miscellaneous	Manufactured food single ingredient	Fatty	Med. Sugar
Pears	Fruit	PE	Fruits and Vegetables	Pome fruits	Pome fruits	Non-fatty	Med. Sugar
	Juice	PJ			Derived edible plant products	N/A	
	Canned	CP			Pome fruits	Non-fatty	Med. Sugar
	Single serving	PX					
	Baby Food	IP			Manufactured food single ingredient		
Peas	Frozen	PS	Fruits and Vegetables	Legume vegs.	Legume vegs.	Non-fatty	Low Sugar
	Canned	SD					
	Baby Food	IE			Manufactured food single ingredient		
Peppers	Bell	PP	Fruits and Vegetables	Fruiting vegs.	Fruiting vegs.	Non-fatty	Low Sugar
	Hot	HP					
Pineapples	Fruit	PN	Fruits and Vegetables	Tropical & sub-tropical fruits - inedible peel	Assorted tropical & sub-tropical fruits - inedible peel	Non-fatty	Med. Sugar
	Canned	NC					
Plums	Dried	PD	Fruits and Vegetables	Stone fruits	Stone fruits	Non-fatty	Med. Sugar
	Fruit	PU					
Pork	Adipose	KA	Animal Tissue/High Protein	Meat	Meat	N/A	
	Muscle	KM					
Potatoes		PO	Fruits and Vegetables	Root & tuber vegs.	Root & tuber vegs.	Non-fatty	Low Sugar
Poultry	Adipose	PA	Animal Tissue/High Protein	Meat	Poultry meat	N/A	
	Breast	PR					
	Liver	PL					
	Muscle	PM					
	Thigh	PT					
Radish		RD	Fruits and Vegetables	Root & tuber vegs.	Root & tuber vegs.	Non-fatty	Low Sugar
Raspberries	Fresh	RS	Fruits and Vegetables	Berry & Small Fruit	Berries & other small fruits	Non-fatty	N/A
	Frozen	RZ					
Raisins		RA	Single Commodities	Berry & Small Fruit	Dried fruits	Non-fatty	High sugar
Rice		RI	Cereal Grains (Low Oil)	Cereal grains	Cereal grains	Non-fatty	Low Sugar

EPA, Codex, and Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) Commodity Groupings

Commodity		PDP Code	PDP Commodity Group	EPA	Codex	PAM	
Snap Peas		SN	Fruits and Vegetables	Legume vegs.	Legume vegs.	Non-fatty	Low Sugar
Soybeans, Grain		SY	Cereal Grains (High Oil)	Legume vegs.	Legume vegs.	Fatty	Med Sugar
Spinach	Leafy	SP	Fruits and Vegetables	Leafy vegs.	Leafy vegs.	Non-fatty	Low Sugar
	Frozen	SF					
	Canned	SC			Manufactured food single ingredient		
Squash	Summer	SS	Fruits and Vegetables	Cucurbits	Cucurbits	Non-fatty	Low Sugar
	Winter	WS					
	Winter, frozen	WZ					
Strawberries	Fresh	ST	Fruits and Vegetables	Berry & Small Fruit	Berries & other small fruits	Non-fatty	Med. Sugar
	Frozen	SZ					
Sweet Potatoes	Raw, fresh	SW	Fruits and Vegetables	Root & tuber vegs.	Root & tuber vegs.	Non-fatty	Med. Sugar
	Baby Food	IS			Manufactured food single ingredient	Non-fatty	Med. Sugar
Tangerines		TA	Fruits and Vegetables	Citrus fruits	Citrus fruits		
Tomatoes	Cherry/Grape	CT	Fruits and Vegetables	Fruiting vegs.	Fruiting vegs.	Non-fatty	Low Sugar
	Fresh	TO					
	Canned	TC					
	Paste	TP	Single Commodities		Manufactured food single ingredient	N/A	
Water	Bottled	WB	Water	Miscellaneous	N/A	N/A	N/A
	Drinking	WR					
	Ground	WG					
	Untreated	WU					
Watermelon		WM	Fruits and Vegetables	Cucurbits	Cucurbits	Non-fatty	Med. Sugar
Wheat	Grain	WH	Cereal Grains (Low Oil)	Cereal grains	Cereal grains	N/A	
	Flour	WF			Cereal grains, milling fraction		

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

Food and Drug Administration Information for Percent Fat, Water, Sugar and pH content for Commodity Groupings

Commodity		% Fat[1]	% Water[1]	% Sugar[1]	pH[2]
Almonds					
Apples	Fruit	0.36	83.93	11.5	3.30 -4.00
	Juice	0.11	87.93	10.9	3.35-4.00
	Sauce	0.18	79.58	16.5	3.10-3.60
Asparagus		0.22	92.25	2.1	6.00-6.70
Avocado		8.87-17.33	72.56-79.73	0.9	6.27-6.58
Bananas		0.48	74.26	18.4	4.50-5.20
Barley		1.16	10.09		5.19-5.32
Basil					
Beans	Black	1.42	11.02		5.78-6.02
	Garbanzo (Chick pea)	6.04	11.53	3.8	6.48-6.80
	Kidney	1.06	11.75		5.40-6.00
	Pinto	1.13	10.95		
	Baby Food	0.1	92.5		
Beef					
Beets		0.06	92.15		5.30-6.60
Blueberry		0.38	84.61	7.3	3.12-3.33
Broccoli		0.35	90.69	1.6	6.30-6.52
Butter		81.11	17.94		
Cabbage		0.18	92.52	2.7	5.20-6.80
Cantaloupe		0.28	89.78	8.1	6.13-6.58
Carrots		0.19	87.79	6.6	5.88-6.40
Cauliflower		0.18	92.26	2.2	5.60
Celery		0.14	94.64	1	5.70-6.00
Cherries, sweet		0.96	80.76	14.6	4.01-4.54
Cilantro					
Corn	Grain	2.08	10		
	Sweet	1.18	75.96	5.4	5.90-7.30
	Syrup				
Cranberry		0.2	86.54		
Cream, heavy		37	57.71	2.8	6.50-6.68
Cucumbers		0.13	96.05	2.3	5.12-5.78
Eggplant		0.1	91.93	3.4	5.50-6.50
Fish, catfish		4.26	76.39	0	
Fish, salmon		3.4-10.44	68.5-76.35	0	
Grapefruit		0.1	90.89	6.2	3.00-3.75
Grapes	Fruit	0.35	81.3	16.4	2.90-3.82
	Juice	0.08	84.12	14.2	
Green Beans		0.12	90.27	2.6	5.60
Greens	Collard	0.22	90.55		
	Kale	0.7	84.46	2.2	6.36-6.80
	Mustard	0.2	90.8	0.8	
Honey		0	17.2	81.9	3.70-4.20
Honey Dew Melon		0.1	89.66		6.00 - 6.67
Infant formula, dairy-based					
Infant formula, soy-based					
Kiwi		0.44	83.05	8.9	
Lettuce		0.19	95.89	1.8	5.80-6.15
Mangoes		0.27	81.71	14.8	3.40 - 4.80
Milk, whole		3.66	87.69	4.9	6.40-6.80
Mushrooms		0.42	91.81	1.8	6.00-6.70
Nectarines		0.46	86.28	8.5	3.92-4.18
Oats		6.9	8.22	5.9	
Olives					

Food and Drug Administration Information for Percent Fat, Water, Sugar and pH content for Commodity Groupings

Commodity		% Fat[1]	% Water[1]	% Sugar[1]	pH[2]
Onions	Bulb	0.16	89.68	4.1	5.30-5.85
	Green	0.19	89.83	3.2	6.20
Oranges	Fruit	0.12	86.75	8.9	3.60-4.34
	Juice	0.2	88.3	10.2	3.30-4.19
Papaya		0.14	88.83		
Peaches		0.09	87.66	8.7	3.30-4.05
Peanut Butter		49.98	1.42	7.8	6.28
Pears	Fruit	0.4	83.81	10.5	3.50-4.60
	Juice				
Peas		0.4	78.86	4.5	5.70-6.70
Peppers	Bell	0.19	92.19	2.5	5.20-5.93
	Hot	0.2	87.74		4.65 - 5.45
Pineapples		0.43	86.5	11.9	3.20-4.00
Plums		0.62	85.2	7.5	2.80-4.30
Pork					
Potatoes		0.1	78.96	1.0	5.40-5.90
Poultry					
Radishes		0.54	94.84	2.7	5.52-6.05
Raisins		0.46	15.42	61.7	3.80-4.10
Raspberries		0.55	86.57		3.18-3.95
Rice		0.58	12.89	0.5	6.0.-6.70
Soybeans		19.94	8.54	6.6	
Spinach		0.35	91.58	0.4	5.50-6.80
Squash	Summer	0.21	93.68	2.2	5.79-6.10
	Winter	0.23	88.72	2.2	5.18-6.49
Strawberries		0.37	91.57	5.7	3.00-3.90
Sweet Potatoes		0.3	72.84	5.0	5.30-5.60
Tangerines		0.19	87.6		
Tomatoes	Fresh	0.33	93.76	3.0	4.30-4.90
	Paste				3.50-4.70
Watermelon		0.43	91.51	9	5.18-5.60
Wheat	Grain				
	Flour				

1 = Pesticide Analytical Manual (PAM) data
2 = Center for Food Safety and Applied Nutrition data

Data not avalilable
Fatty (>2% fat)
Non-fatty (<2% fat)
Low H_2O (<75%)
Low sugar (<5%)
Med sugar (5-15%)
High sugar (>15%)

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

USDA, AMS Pesticide Data Program
Verification of Limits of Detection (LODs)

Commodity: ____

Date: ____

Lab: ____

Note: During method validation, two spikes are required; if this form is used to record annual spike verification, only one spike is required.

Pesticide/Compound	Amt Spk Units =	LOD Spike Recovered (yes/no or +/-)	
		Spike 1	Spike 2

USDA, AMS Pesticide Data Program
Determination of Method Range

Commodity: _____ **Instrument/Detector:** _____

Date: _____ **Column:** _____

Lab: _____

Pesticide/Compound			1 X LOQ					5 X LOQ					10 X LOQ				
	LOD	LOQ	Rep 1	Rep 2	Rep 3	Mean	%CV	Rep 1	Rep 2	Rep 3	Mean	%CV	Rep 1	Rep 2	Rep 3	Mean	%CV
	Units=		Percent Recovery					Percent Recovery					Percent Recovery				

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USDA, AMS Pesticide Data Program
Precision and Accuracy Data Collection

Commodity: _____ **Instrument/Detector:** _____

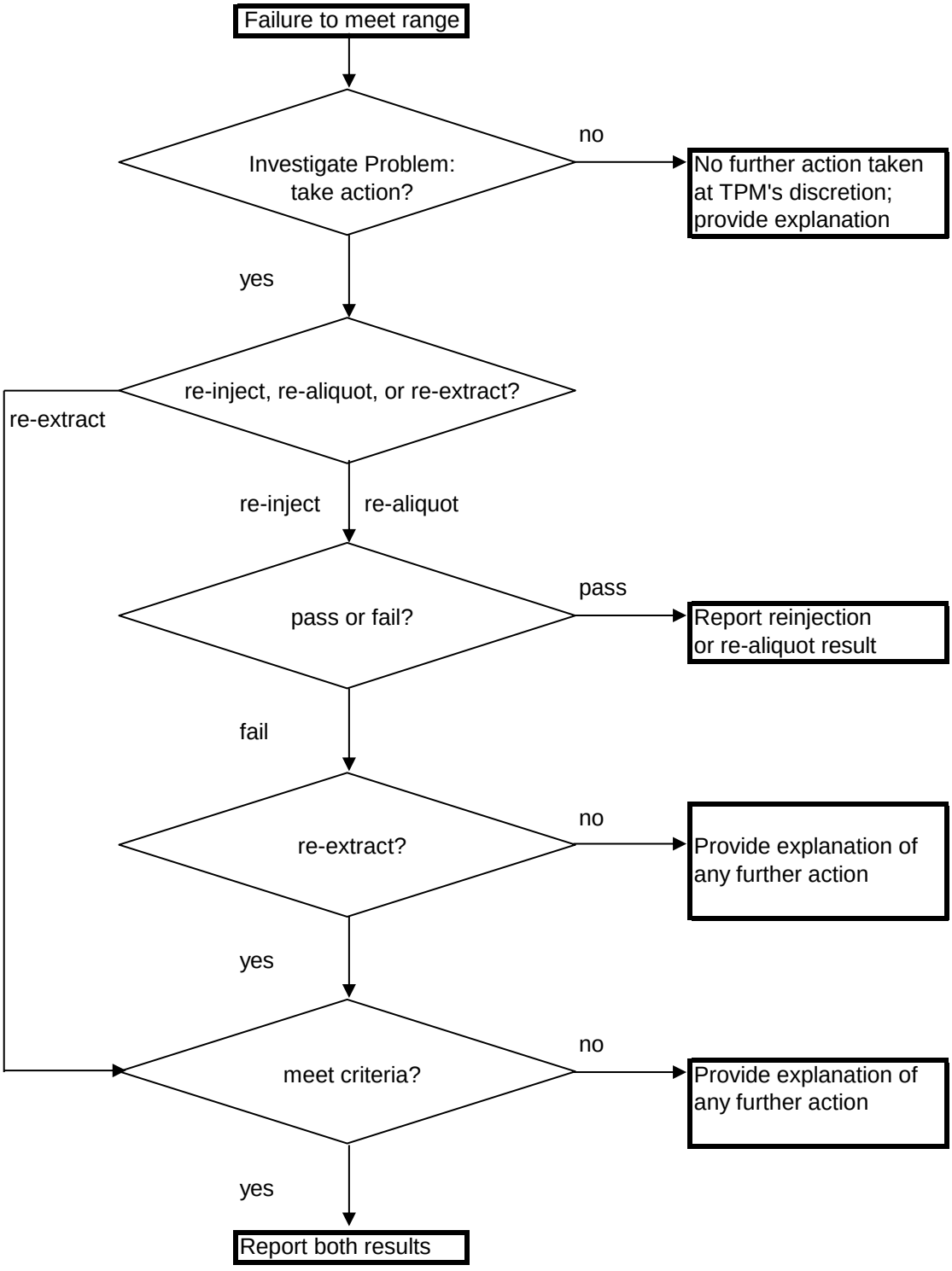
Date: _____ **Column:** _____

Lab: _____

Pesticide/Compound			2xLOQ Matrix Spikes							Mean	%CV	Comments
	LOD	LOQ	1	2	3	4	5	6	7			
	Units=		Percent Recovery							%R		

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

Process Control and Spike Recovery Acceptability Flowchart



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

SOP No.: PDP-LABOP		Page 1 of 34
Title: Sample Processing and Analysis		
Revision: 10	Replaces: 07/01/2017	Effective: 07/01/2018

1. Purpose:

To provide standard procedures for:

- the receipt, storage, archiving, and disposal of USDA/AMS Pesticide Data Program (PDP) samples and sample portions
- the preparation of USDA/AMS PDP samples
- handling sample homogenates that are shipped to another laboratory for analysis

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 Procedure:

4. References:

- *Memorandum*, Martha Lamont, PDP Technical Director, to Ed Zager, Chief, EPA/HED, August 24, 2000
- *Memorandum*, OPs in Meat and Poultry, Martha Lamont, EPA/HED, June 8, 1998
- *PCNG [Pesticide Chemical News Guide]*, annual release, CRC Press, LLC, 1725 K St NW,0Washington DC 20006
- U.S. EPA, *Maintenance and calibration of equipment,* 40 CFR 160.63
- U.S. EPA, *Maintenance and calibration of equipment,* 40 CFR 160.63
- U.S. EPA, *Standard operating procedures,* 40 CFR 160.81
- U.S. FDA, *Instructions for the Items Prepared by Contract Kitchen, Standard Operating Procedure for the Total Diet Study KCX-1*, Appendix F, January 19, 1993
- U.S. FDA, *Final Preparation Procedures, Standard Operating Procedure for the Total Diet Study KCX-1*, Appendix E, January 19, 1993
- *Memorandum to State PDP Laboratories from Dr. Robert Epstein*, Science Division, AMS, April 25, 1991
- *Memorandum to State PDP Laboratories from Dr. Robert Epstein*, Science Division, AMS, May 22, 1991
- U.S. EPA, *Good Laboratory Practice Standards*, 40 CFR Parts 160.47 and 160.51, August 17, 1989

5. Sample Processing, Storage, and Disposal

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. These instructions shall include specific practices for minimizing cross-contamination during preparation of multiple samples (e.g., cleaning of equipment and utensils between samples). Both the USDA/AMS SOPs and the laboratories' internal SOPs and work instructions will be used as the measure of compliance in the event of a USDA/AMS laboratory review. Each sample shall be analyzed for identified compounds (refer to applicable commodity-specific compound list memorandum).

5.1 Sample Receipt

Information required to be recorded in RDE/SIF

Required Information	Detailed instructions	RDE Field	SOP Section	Required/ Optional
Variety information	If not recorded by sampler	Variety Field	5.1.1	Required if available
Lot information	If not recorded by sampler	Lot Field (or Sample Comments Field if more space required	5.1.1	Required if available
Product preparation prior to sample collection	Received as washed, chopped, snipped – needs to meet Fact Sheet requirements	Sample Comments	5.1.2	Required if relevant
Container integrity, adequacy, and/or custody seals	Container integrity Container adequacy Custody seals	Lab Comments	5.1.3	Required if there are problems
Amount/weight received if different from required	Overweight/underweight	Lab Comments	5.1.5	Required if there are problems
Sample unable to be analyzed by lab	Specify reason sample not analyzed	Reason NOT Analyzed	5.1.4 5.1.7	Required if not analyzed
Received sample	Record date sample received	Date/Time Received	5.1.1	Required
Received sample	Record person that received the sample	Received By	5.1.1	Required

Required Information	Detailed instructions	RDE Field	SOP Section	Required/ Optional
Sample weight homogenized	Weight of sample homogenized	Sample Size	5.1.5	Optional
Unique laboratory number	Lab assigned number	Internal Lab Identifier	5.1.11	Required
Units	Record number of units received	# of Units	5.1.12	Required if unit counting is specified

5.1.1 Sample Inspection at Receipt

5.1.1.1 Record the person who received the sample and the date received in the RDE sample information.

5.1.1.2 Samples shall be inspected upon arrival to verify that the sample is suitable for analysis based on commodity requirements (refer to current Monitoring Programs Division (MPD) Commodity Fact Sheet). Ensure that lot numbers on all units are the same, unless a specific Commodity Fact Sheet allows multiple lots to achieve required weight. Check that required information (variety, lot numbers, etc.) that can be determined is recorded in the RDE sample information (if not already recorded by sampler), and that the information in RDE and sample identification match each other. This may be done either directly in RDE or noted on a printed Sample Information Form (SIF) and entered into RDE before or during reporting.

5.1.1.3 The laboratory shall establish procedures for ensuring the single sample label information is retained (e.g., attaching to printed eSIFs, in a sample receipt logbook, etc.)

5.1.2 Prepared Fresh Commodity Acceptability

Prepared fresh product (e.g., snipped green beans, chopped packaged lettuce) is acceptable as long as the commodity requirements on the fact sheet are met. The laboratory shall note that the product is prepared (e.g., washed, chopped, snipped) in the "Sample Comments" section of the RDE sample information if it is not already noted by the sampler.

5.1.3 Sample Containers

If the sample container integrity is compromised or inadequate, document this in the "Lab Comment" section of the RDE sample information. The laboratory shall contact the MPD Sampling Manager if there are questions as to the sample's viability.

5.1.3.1 Fresh fruit and vegetable containers shall be inspected upon arrival for any deteriorating condition (e.g., leaking sample container) which would make the sample inedible or compromise sample integrity (e.g., cross contamination).

5.1.3.2 Canned commodities shall be free of large dents or punctures.

5.1.3.3 Frozen commodities shall be inspected to determine the extent of thawing during transit.

5.1.3.4 The plastic bags sealed by the collectors shall be opened only if absolutely necessary to determine the condition of the sample(s). If the sample bag is packed too tightly to accurately count the units (for required commodities), a rough count (i.e., 13-14 units) may be recorded at the time of receipt. The unit count can then be performed after the bag is opened prior to homogenization.

5.1.3.5 If the sample integrity is compromised (e.g., frozen samples that have completely thawed, bags that are not sealed, cans with dents, compromised custody seals, etc.), the laboratory shall contact the MPD Sampling Manager to determine if analysis should be performed or if the sample should be re-collected.

5.1.4 Damaged Animal Tissue

Animal tissue samples (e.g., fish), or portions thereof, received in a damaged condition (e.g., warm to the touch, spoiled, or leaking) shall be discarded and not analyzed. Condition and disposal shall be recorded on all applicable documentation. If a sample must be discarded, the laboratory shall immediately notify MPD.

5.1.5 Sample Weight Acceptability Criteria

The sample is defined as the portion that the collector provides to the laboratory, usually between one and seven pounds. The acceptable weight range is ± 20% of the target weight (e.g., for 5 lb. samples: 4-6 lbs). ***Note:*** *Determination of the weight of the sample being homogenized is optional; however, if the weight is determined, it shall be entered in the "Sample Size" field of the RDE sample information.*

5.1.5.1 Samples that weigh less than 70% of the target weight are not acceptable (e.g., < 3.5 lbs. for 5 lb. samples). If the sample weighs between 70% and 80% of the target weight, it is left to the discretion of the receiving laboratory, based on their best professional judgment, whether or not to request resampling. Alternatively, the laboratory may contact MPD for further guidance.

5.1.5.2 If the laboratory receives an unusually large sample (e.g., more than ten pounds), the laboratory may randomly select the targeted weight of product (e.g., 5 pounds for oranges) to homogenize, as long as units or bunches are not broken (e.g., do not halve cantaloupes or split grape bunches, etc.). The laboratory shall record any weight related issues in the "Lab Comments" field of the RDE sample information (e.g., received weight between 70 and 80% of target, laboratory randomly selected target weight due to large sample size received, etc.).

5.1.6 Sample Viability

5.1.6.1 For a sample to be considered viable, a minimum of 70% of the sample, by weight or count, should be available for analysis after any damaged/deteriorated portions (e.g., wilted, mushy, moldy, etc.) are discarded.

5.1.6.2 For processed commodities (e.g., cans, jars, boxes, etc.) submitted as multiple subsamples, the lot numbers must match.

Note: *Some commodities use lot numbers that include a time stamp. For example, if three jars are labeled 15502B1130, 15502B1132, and 15502B1133, the lot number is 15502B and the last four digits are the time stamp. Times should be within a three-hour window. Lot number formats differ widely among commodities and companies. Contact MPD for guidance if there are questions regarding viability.*

5.1.7 Documentation for Samples Not Analyzed

If sample condition upon arrival prevents analysis (e.g., entire sample mushy), the condition shall be documented in the "Reason NOT Analyzed" field of the RDE sample information.

5.1.8 Missing/Late/Unacceptable Samples or eSIFs

5.1.8.1 If a sample is not received within five working days from collection or is unacceptable, the laboratory shall contact the appropriate State Sampling Manager or USDA, GIPSA designee(s) to arrange for recollection (notification of the PDP Sampling Manager is encouraged, but not required). Recollection should occur within the same month if possible (except December recollections must be within the calendar year).

5.1.8.2 If a sample arrives without a corresponding RDE SIF, the laboratory shall contact the appropriate State Sampling Manager within 24 hours and copy MPD at amsmpo.data@ams.usda.gov

5.1.8.3 If an eSIF contains an error that cannot be resolved with the Sampling State contact MPD at amsmpo.data@ams.usda.gov

5.1.8.4 If an RDE SIF arrives for a non-collected sample, the SIF shall be attached to a group/set and submitted in RDE by the laboratory. The non-collected sample can be attached to a group that contains routine analyzed samples or to a group that contains just non-collected samples. This allows MPD to track the number of missing samples and the reasons why the sample was not collected.

5.1.9 Unresolved Sampling Issues

The receiving laboratory shall notify MPD of any continuing unresolved sampling issues monthly.

5.1.10 Paper SIFs

If a paper SIF is received, the laboratory shall fax a copy (or scan and email) to MPD within 24 hours if data is not available in RDE. Grain samples collected by GIPSA are exempt from this SIF requirement (see *Grain Sample Ticket Form, FGIS-920).*
http://archive.gipsa.usda.gov/reference-library/handbooks/grain-insp/grbook4/gr-bk4-ch2.pdf.

5.1.11 Unique Laboratory Sample ID

Each sample shall be assigned a unique laboratory identification number. The identification number shall be recorded on or affixed to samples and sample aliquots in a manner to ensure its legibility during handling and storage. This number shall also be recorded on the accompanying paperwork and in the "Internal Lab ID" field in the RDE sample information.

5.1.12 Unit Counting

For all large non-clustered commodities (e.g., apples, cantaloupe, onions, pears, sweet potatoes, etc.) the counting of homogenized units (i.e., pieces of individual produce) is required for EPA to perform some of their risk assessment calculations. For any given commodity, whether or not units shall be counted, is stipulated in Section 5.3 of this SOP. Units shall be examined prior to homogenization and damaged units discarded. If more than one-third of the edible portion is damaged, discard the entire unit, then count the remaining units. Record the count in the "# of Units" field in the RDE sample information. Refer to Section 5.1.3.4 if the sample bag is so tightly packed that accurate unit counting cannot be performed.

5.1.13 Sample Receipt Log

Each laboratory shall maintain a log of samples received. Suggested methods are either in a bound notebook with ink or a computer log as long as the electronic storage of data follows acceptable practices. Refer to SOP PDP-DATA. Minimum information recorded includes sample numbers, date and time received (unless documented on the SIF), and who received the sample. Other information may include commodity type, reference to analytical method, results, and date when results were reported.

5.2 Sample Storage Prior to Homogenization

5.2.1 All refrigerators and freezers used for PDP samples shall have controlled access. Each laboratory shall have a system in place to monitor and document temperatures and sample traffic. The temperature checks shall be made each working day, or the laboratory may use automatic temperature recording devices. Checks shall be recorded.

5.2.2 Samples shall be stored in refrigerators and freezers separate from standards.

5.2.3 Fresh fruits and vegetables still sealed in bags shall be refrigerated for a period not to exceed 72 hours, or 120 hours, depending on whether it's perishable, from the time of arrival until the sample is homogenized. Section 5.3 of this SOP lists the maximum holding hours prior to homogenization for each PDP commodity.

5.2.4 Commodities normally stored by consumers at room temperature (e.g., in cans, jars, shelf-stable boxes, etc.) shall be stored in a clean, dry area at room temperature (approximately 22°C) or lower until the sample is homogenized.

5.2.5 Frozen commodities that have not thawed in transit (still cold to the touch) shall be held in the freezer at approximately 0°C or lower until the sample is homogenized.

5.2.6 Frozen commodities that thawed in transit (not cold to the touch) shall be refrigerated. If possible the sample should be homogenized within 24 hours (from the time of arrival); however refrigeration of the thawed commodity for a period not to exceed 72 hours (from the time of arrival) is acceptable.

5.3 Preparation and Homogenization of Fresh Produce, Animal Tissue, Nuts, and Grains

For all commodities, the entire sample shall be prepared for homogenization according to the commodity-specific instructions in this section. If the entire sample does not fit into the homogenizer/chopper at one time, then the sample may be homogenized in portions. All portions shall be mixed together in a clean container to assure an evenly mixed sample.

If the laboratory receives a sample that weighs significantly more than the targeted weight (e.g., 10 pounds when target weight is 5 pounds), follow the instructions in Sections 5.1.5.2 and 5.1.6 of this SOP.

Commodity		**PDP Code**	**SOP Preparation and Homogenization Section**
Almonds		AL	5.3.1
Apples	Fruit	AP	5.3.3
	Juice	AJ	5.4.3 (Juices/Concentrates)
	Sauce	AC	5.4.5 (Other Processed, Semi-processed, Packaged)
Asparagus		AS	5.3.4
Avocados		AV	5.3.5
Baby foods	Applesauce	IA	5.4.5 (Other Processed, Semi-processed, Packaged)
	Carrots	IC	
	Green Beans	IG	
Baby foods	Peaches	IH	
	Pears	IP	5.4.5 (Other Processed, Semi-processed, Packaged)
	Peas	IE	
	Sweet potatoes	IS	
Bananas		BN	5.3.6
Barley		BY	5.3.20 (Grains)

Commodity			**PDP Code**	**SOP Preparation and Homogenization Section**
Beans	Canned	Black	BC	5.4.1 (Canned Commodities)
		Kidney		
		Pinto		
		Garbanzo (Chick pea)	ZB	
	Green		GB	5.3.7
Beets, canned			BT	5.4.1 (Canned Commodities)
Blueberry		Fresh	BB	5.3.8
		Frozen	BZ*	5.4.2 (Frozen Commodities)
Broccoli			BR	5.3.9
Butter			BU	5.4.5 (Other Processed, Semi-processed, Packaged)
Cabbage			CG	5.3.10
Cantaloupe			CN	5.3.11
Carrots			CR	5.3.12
Cauliflower			CF	5.3.13
Celery			CE	5.3.14
Cherries, Sweet		Fresh	CH	5.3.15
		Frozen	CZ*	5.4.2 (Frozen Commodities)
Cilantro			CL	5.3.16
Corn		Grain	CO	5.3.20 (Grains)
		Sweet	CS	5.3.17
Corn		Syrup	CY	5.4.3 (Juices/Concentrates)
Cranberry		Fresh	CA	5.3.8
		Frozen	AZ	5.4.2 (Frozen Commodities)

Commodity		**PDP Code**	**SOP Preparation and Homogenization Section**
Cream, heavy		CM	5.4.5 (Other Processed, Semi-processed, Packaged)
Cucumbers		CU	5.3.18
Egg		EG	5.4.5 (Other Processed, Semi-processed, Packaged)
Eggplant		EP	5.3.19
Fish	Catfish	FC	5.3.2 (Animal Tissue-Fish)
	Salmon	FS	
Grapefruit		GF	5.3.31
Grapes	Fruit	GR	5.3.21
	Juice	GJ	5.4.3 (Juices/Concentrates)
Greens	Collard	GS	5.3.22
	Kale	GK	
Honey		HY	5.4.5 (Other Processed, Semi-processed, Packaged)
Honey Dew Melon		HD	5.3.23
Infant formula	Dairy-based	DF	5.4.5 (Other Processed, Semi-processed, Packaged)
	Soy-based	YF	
Kiwi		KW	5.3.24
Lettuce	Head	LT	5.3.25
	Leaf	LT	5.3.26
	Bagged	LB	5.4.5 (Other Processed, Semi-processed, Packaged)
Mangoes		MA	5.3.27

Commodity		**PDP Code**	**SOP Preparation and Homogenization Section**
Milk, whole		MK	5.4.5 (Other Processed, Semi-processed, Packaged)
Mushrooms		MU	5.3.28
Nectarines		NE	5.3.33
Oats		OA	5.3.20 (Grains)
Olives	Canned	OL	5.4.1 (Canned Commodities)
Onions	Bulb	ON	5.3.29
	Green	GO	5.3.30
Orange	Fruit	OG	5.3.31
	Juice	OJ	5.4.3 (Juices/Concentrates)
Papaya		YA	5.3.32
Peaches	Fruit	PC	5.3.33
	Canned	CC	5.4.1 (Canned Commodities)
Peanut Butter		PB	5.4.5 (Other Processed, Semi-processed, Packaged)
Pears	Fruit	PE	5.3.34
	Juice	PJ	5.4.3 (Juices/Concentrates)
	Canned	CP	5.4.1 (Canned Commodities)
Peas	Green	PS	5.3.35
	Snap	SN	5.3.36
Peppers	Bell, Sweet	PP	5.3.37
	Hot	HP	5.3.38
Pineapples	Fresh	PN	5.3.39
	Canned	NC	5.4.1 (Canned Commodities)
Plums	Fresh	PU	5.3.40
	Dried	PD	5.4.4

Commodity		**PDP Code**	**SOP Preparation and Homogenization Section**
Potatoes	Fresh	PO	5.3.41
	Frozen	PZ*	5.4.2 (Frozen Commodities)
	Sweet	SW	5.3.42
Raisins		RA	5.4.4
Raspberries	Fresh	RS	5.3.8
	Frozen	RZ*	5.4.2 (Frozen Commodities)
Rice		RI	5.3.20 (Grains)
Soybeans, Grain		SY	5.3.20 (Grains)
Spinach	Leafy	SP	5.3.43
	Canned	SC	5.4.1 (Canned Commodities)
	Frozen	SP	5.4.2 (Frozen Commodities)
Squash	Summer	SS	5.3.44
	Winter	WS	5.3.45
	Winter, frozen	WZ	5.4.2 (Frozen Commodities)
Strawberries	Fresh	ST	5.3.46
	Frozen	SZ*	5.4.2 (Frozen Commodities)
Tangerines		TA	5.3.31
Tomatoes	Cherry	CT	5.3.47
	Fresh	TO	5.3.48
	Canned	TC	5.4.1 (Canned Commodities)
	Paste	TP	5.4.5 (Other Processed, Semi-processed, Packaged)
Watermelon		WM	5.3.49
Wheat	Grain	WH	5.3.20 (Grains)
	Flour	WF	5.4.5 (Other Processed, Semi-processed, Packaged)

* For USDA use only

5.3.1 Almonds

Grind the entire sample using an appropriate device (e.g., centrifugal mill, Wiley mill, etc.) just until a visually homogeneous mixture is attained. Unit counting is not required.

5.3.2 Animal Tissue-Fish

Remove the skin and bones and mechanically homogenize the entire submitted tissue sample until a visually homogeneous mixture is attained. The laboratory shall use its discretion in the utilization of dry ice during the homogenization procedure. Unit counting is not required.

5.3.3 Apples

Wash each apple under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the apple have been rinsed. Allow to drain for at least 2 minutes. Do not peel. Remove the stem, if present. With a commercially available apple corer remove core or, using a clean, dry knife, cut each apple in half or quarters and remove the core portion. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.4 Asparagus

Remove an inch or two of the woody stem, if inedible. Wash asparagus spears under cold running tap water for approximately 15-20 seconds to assure that the water has rinsed all portions of the sample. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.5 Avocados

If necessary, avocado samples may be stored in a secure location at room temperature for up to 72 hours for ripening purposes. Wash avocados under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, cut the avocado around the pit (i.e., without cutting through the pit).

Remove the pit and skin, being careful to keep as much of the meat as possible. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours for ripe avocados and 120 hours for unripe avocados from the arrival time until the sample is homogenized.

5.3.6 Bananas

If necessary, banana samples may be stored in a secure location at room temperature for up to 72 hours for ripening purposes. Peel each fruit. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours for ripe bananas and 120 hours for green bananas from the arrival time until the sample is homogenized.

5.3.7 Beans, Green

Wash fresh beans under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Do not peel. Using a clean, dry knife, remove any stems that are present. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.8 Blueberries/Cranberries/Raspberries

Wash blueberries/cranberries/raspberries by the handful or by using a colander under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.9 Broccoli

Visually examine and discard any damaged portion or wilted florets. Do not discard leaves unless they are wilted. Trim away inedible portions of stems as described and illustrated in Laboratory Work Instruction – Broccoli. Wash the sample under cold running tap water for approximately 15-20 seconds to assure that the water has rinsed all portions of the sample. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.10 Cabbage

Visually examine the head, remove wrapper, damaged or wilted leaves, and the core. Rinse the head under cold running tap water for approximately 15-20 seconds. Turn the head top side down. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.11 Cantaloupes

Using a clean, dry knife, cut each cantaloupe in half and remove seeds and rind. Halves may be further divided at this point to facilitate removal of the rind. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.12 Carrots

If carrots have any visible dirt, hold each carrot under cold running tap water and gently scrub the entire surface with a clean vegetable brush to remove any loose soil and grit. Rinse each scrubbed carrot under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the carrot have been rinsed. Allow to drain for at least 2 minutes. With a clean, dry knife, remove stem cap portion from each carrot. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.13 Cauliflower

Visually examine the head and remove wrapper leaves and any damaged portions. Rinse the head under cold running tap water for approximately 15-20 seconds. Turn the head top side up. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogenous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.14 Celery

Using a clean, dry knife, remove the inedible portion of the stalk (i.e., the woody part at the base of the stalk) to allow the stems to separate. Do not remove the leaves unless discolored or

damaged. Wash the stems under cold running water for approximately 15-20 seconds to assure that all surfaces have been rinsed and that all extraneous matter (e.g., soil) is removed. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.15 Cherries, Sweet

Remove the stem from each cherry. Wash cherries under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Remove the pit, being careful to remove as little of the meat as possible. A commercial cherry pitter is recommended. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.16 Cilantro

Using a clean, dry knife trim the ends. Remove the discolored or damaged leaves. Wash the stems with the leaves under cold running water for approximately 15-20 seconds to assure that all surfaces have been rinsed and that all extraneous matter (e.g., soil) is removed. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.17 Corn

Remove husk and silk from each ear. Wash each ear under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean dry knife or other appropriate utensil, remove the kernels from cob. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.18 Cucumbers

Wash each cucumber under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the cucumber are rinsed. Allow to drain for at least 2 minutes. Cucumbers

may be halved or quartered at this point to facilitate homogenization. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.19 Eggplant

Wash each eggplant under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, remove the end pieces. Mechanically chop just until a visually homogenous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.20 Grains

Pour entire grain sample into a Boerner Divider and use one of the two resulting 500 gram sub-samples for homogenization (the remaining 500 gram sub-sample can be stored). Grind the 500 gram subsample using an appropriate device (e.g., Falling 3300 laboratory mill, Jacobsen grinder, UDY). Tumble the resulting powder homogenate to obtain a homogeneous mixture. Unit counting is not required.

5.3.21 Grapes

Wash each sample under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Remove all stems and extraneous matter. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.22 Greens

Visually examine the sample and remove only the damaged or wilted leaves and any woody stems. Wash remaining sample under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. **Note:** *Bagged pre-washed greens do not require washing by the laboratory.* Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.23 Honeydew Melons

Using a clean, dry knife, cut each melon in half and remove seeds and rind. Halves may be further divided at this point to facilitate removal of the rind. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.24 Kiwi

Wash each kiwi under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Do not peel. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.25 Lettuce, Head

Visually examine the head and remove wrapper and damaged or wilted leaves. Rinse the head under cold running tap water for approximately 15-20 seconds. Turn the head top side down. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.26 Lettuce, Leaf

Visually examine the sample and remove only the damaged or wilted leaves and any woody stems. Wash remaining sample under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required if discrete bunches are received. Unit counting is not required if loose leaves are received. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.27 Mangoes

Mango skin contains small amounts of urushiol (the same chemical as in poison ivy) and may cause an allergic reaction or induced contact dermatitis. Sensitive persons should handle mangoes with gloves. Wash each mango under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the fruit are rinsed. Allow to drain for at least 2 minutes.

Do not peel. Remove stem if present. Using a clean, dry knife, cut the mango around the pit (i.e., without cutting through the pit). Remove the pit, being careful to remove as little of the meat as possible. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours for ripe mangoes and 120 hours for green mangoes from the arrival time until the sample is homogenized.

5.3.28 Mushrooms

Wash mushrooms under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, slightly trim end pieces to remove any inedible/woody portions. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.29 Onions, Bulb

Using a clean knife, remove onion top, outer layer, first white layer and membrane, and any other inedible portions. Remove root portion last to minimize fumes. Preparation procedures may be performed with onions immersed in cold tap water, with total immersion time for each unit not to exceed 10 minutes. Allow onions to drain at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.30 Onions, Green

Wash green onions under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, trim the bulb end of any roots/inedible material and trim the tops if damaged or wilted. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.31 Oranges/Tangerines/Grapefruit

Peel each fruit and remove any excess white membrane. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.32 Papaya

If necessary, papaya samples may be stored in a secure location at room temperature for up to 72 hour for ripening purposes. Wash each papaya under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the fruit are rinsed. Allow to drain for at least 2 minutes. Cut in half. Scoop out and discard seeds. Scoop out flesh for homogenization. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Ideally, the fruit should be ripe enough to deal with. Refrigeration may not exceed 72 hours for ripe papayas and 120 hours for green papayas from the arrival time until the sample is homogenized.

5.3.33 Peaches/Nectarines

Wash each peach/nectarine under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the peach are rinsed. Allow to drain for at least 2 minutes. Do not peel. Remove stem and leaves if present. Using a clean, dry knife, cut the peach around the pit (i.e., without cutting through the pit). Remove the pit, being careful to remove as little of the meat as possible. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.34 Pears

Wash each pear under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the pear have been rinsed. Allow to drain for at least 2 minutes. Do not peel. Remove stem, if present. Using a clean, dry knife, cut each pear in half or quarters and remove the core portion. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.35 Peas, Green

For each sample, shell enough peas to comprise at least one cup. Discard pods. Rinse peas under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.36 Peas, Snap

For each sample, rinse snap peas under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Remove inedible portion(s). **Note:** *Bagged pre-washed (including ready-to-eat and steam in bag) snap peas do not require washing and may be processed as-is by the laboratory.* Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.37 Peppers, Bell Sweet

Wash each pepper under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, remove stem, core, and seeds. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.38 Peppers, Hot

Wash each pepper under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, remove stem. For Anaheim variety only, also remove core and seeds. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.39 Pineapples

Wash each pineapple under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the fruit have been rinsed. Allow to drain for at least 2 minutes. Remove the top of each pineapple. Using a clean, dry knife, cut in half and remove core and shell. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is

required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.40 Plums

Wash each plum under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Do not peel. Remove stem and leaves if present. Using a clean, dry knife, cut the plum around the pit (i.e., without cutting through the pit). Remove the pit, being careful to remove as little of the meat as possible. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.41 Potatoes

Hold each potato under cold running tap water and gently scrub the entire surface with a clean vegetable brush to remove any loose soil and grit. Rinse each scrubbed potato under cold running tap for approximately 15-20 seconds to assure that all surfaces of the potato have been rinsed and allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.42 Potatoes, Sweet

Hold each sweet potato under cold running tap water and gently scrub the entire surface with a clean vegetable brush to remove any loose soil and grit (remove any woody stems if present). Rinse each scrubbed sweet potato under cold running tap for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.43 Spinach

Visually examine the sample and remove only the damaged or wilted leaves and any woody stems. Wash remaining sample under cold running tap water for approximately 15-20 seconds to assure that all surfaces have been rinsed. Allow to drain for at least 2 minutes. **Note:** *Bagged pre-washed spinach does not require washing by the laboratory.* Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.44 Squash, Summer

Wash each squash under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, remove end pieces. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.45 Squash, Winter

Wash each squash under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. When possible, using a clean, dry knife, remove stem and/or end pieces. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.46 Strawberries

Wash strawberries by the handful or by using a colander under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Remove stems and leaves if present. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.47 Tomatoes, Cherry

Wash tomatoes under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the tomatoes are rinsed. Allow to drain for at least 2 minutes. Do not peel. Remove any present stems. Unit counting is not required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.48 Tomatoes, Fresh

Wash each tomato under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the tomato are rinsed. Allow to drain for at least 2 minutes. Do not peel. Using a

clean, dry knife, cut the tomato around the stem area. Remove any stem, being careful to remove as little of the meat as possible. The tomatoes may be quartered prior to homogenization. Mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.3.49 Watermelon

Wash each melon under cold running tap water for approximately 15-20 seconds to assure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, cut each watermelon into quarters, and remove the rind. For large watermelons, take alternate quarters of each fruit and mechanically chop just until a visually homogeneous mixture is attained. For small watermelons, take the entire sample and mechanically chop just until a visually homogeneous mixture is attained. Unit counting is required. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.4 Preparation and Homogenization of Processed Commodities

The sample is defined as the portion that the collector sends to the laboratory, usually between one and seven pounds. For all commodities except dried fruits, the entire sample shall be homogenized. For dried fruits, the entire sample is mixed to obtain a representative analytical portion prior to hydration and analysis. If the entire sample does not fit into the homogenizer/chopper at one time, then the sample may be homogenized in portions. All portions shall be mixed together in a clean container to assure an evenly mixed sample.

5.4.1 Canned Commodities

If the lid of the can has visible dirt or dust, rinse the lid under cold running tap water for 5 to 10 seconds. Dry the lid with a paper towel. Open each can and pour the entire contents of each can including the liquid into a blender/homogenizer. Blend just until a visually homogeneous mixture is attained.

5.4.2 Frozen Commodities

The samples may be chopped while frozen, or to prevent damage to the chopper/homogenizer blades, the sample may be thawed in a refrigerator or in a room temperature water bath. Open

the containers and pour the entire contents into the chopper/homogenizer. Mechanically chop just until a visually homogeneous mixture is attained.

5.4.3 Juices/Concentrates

For fresh and reconstituted juices, ensure that the sample is evenly mixed to obtain a homogeneous mixture. For concentrates, dilute juice in a dry, clean container with cold running tap water, according to label directions. Mix well to ensure a homogeneous mixture.

Canned product concentrates (e.g., tomato paste) may be considered homogeneous and do not need to be mixed prior to weighing of analytical portion. Dilute appropriate analytical portion with sufficient water to facilitate sample extraction. Report results based on undiluted concentrated product.

5.4.4 Dried Fruits

Open all of the dried fruit package(s) into a container and mix or shake to obtain a representative analytical portion. Add enough water to cover the analytical portion and soak with water until re-hydrated. Prepare the analytical portion for extraction and analysis. Unit counting is not required.

5.4.5 Other Processed, Semi-Processed, Packaged Commodities

5.4.5.1 For other processed, packaged products that are homogenous, (e.g., corn syrup, peanut butter, baby food) proceed as follows:

- If the sample is comprised of a single container, simply weigh appropriate analytical portion.

- If the sample is comprised of multiple containers, combine and mix enough containers to achieve the commodity's specified sampling size (e.g., 16 ounces for baby foods) and weigh appropriate analytical portion.

5.4.5.2 If a processed, packaged product appears non-homogeneous (e.g., separation of oil from peanut butter), ensure that the sample is evenly mixed prior to weighing of analytical portion.

5.4.5.3 For semi-processed raw commodities (e.g., pre-washed, bagged spinach; peeled, cut carrots; etc.) refer to instructions in Section 5.3. Washing is not required.

5.4.5.4 For infant formula:

- For ready-to-eat samples, ensure that the sample is evenly mixed to obtain a homogeneous mixture.
- For concentrated liquid samples, dilute in a dry, clean container with reagent water, according to label directions and mix well to ensure a homogeneous mixture.
- For powdered samples, reconstitute in a dry, clean container with reagent water according to label directions and mix well to ensure a homogeneous mixture.

5.4.5.5 For Eggs:

- Crack open the eggs that make up the sample unit (minimum 10 eggs) into a clean blender/homogenizer. Discard the egg shells. Homogenize the eggs until a visually homogeneous mixture is attained. Refrigeration may not exceed 72 hours from the arrival time until the sample is homogenized.

5.5 Weighing of Analytical Portion

The laboratory internal SOP shall define the weight required for the analytical portion. If the precision is $\pm$1% or less, the laboratory may use the nominal target weight in further calculations.

5.6 Transshipment of Homogenate Subsamples

Specific details not addressed here may be worked out between the shipping laboratory, the testing laboratory, and/or MPD.

5.6.1 MPD designates which commodity homogenates shall be transshipped from one laboratory to another. Transshipments occur when required analyses (typically special procedures for single analytes or analyte classes) are not performed by the laboratory that receives the original collected sample. Rather than having PDP sampling staff split portions at the point of sample collection, laboratories split the sample at the point of homogenization. PDP and laboratory SOPs for handling, preparation, and custody shall apply to the subsamples destined for transshipping.

5.6.2 The testing laboratory designates the analytical portion size (e.g. by weight, volume, etc) and the number of replicates necessary to perform their testing. The laboratories shall agree upon suitable containers and what paperwork needs to accompany the shipment. The shipping laboratory shall ensure that adequate analytical portions are provided to the testing laboratory by verifying that the agreed upon containers and fill volumes provide the minimum amounts needed for analysis.

5.6.3 At the time of sample homogenization, the specified analytical portion is placed into the sample container and the container is labeled with the internal laboratory identification number. This information shall be recorded in permanent non-smearing ink or on waterproof, freezer-proof stickers. The homogenate subsamples shall then be stored at approximately -40°C, or lower, at least overnight, until shipment.

5.6.4 On the day of shipment, homogenates and applicable paperwork are packaged tightly into shipping coolers with adequate blue ice and packing material to ensure they are received in satisfactory condition by the testing laboratory.

5.6.5 At a minimum, all samples shall be identified with both the PDP sample identification number and the internal laboratory identification number either directly (on the sample container) or indirectly (e.g., logsheets/worksheets). Appropriate chain-of-custody forms and sample identification logsheets/worksheets (if used) shall be placed in a resealable plastic bag and included with the samples. If shipping to a non-PDP laboratory, the PDP Sample Information Form (SIF) shall not be included because it contains proprietary program site information.

5.6.6 Homogenates shall be shipped by overnight courier so that they arrive at the testing laboratory on a workday unless a weekend delivery has been agreed upon by the laboratory and MPD. The shipping laboratory shall notify the testing laboratory of the shipment. The shipping laboratory bears the cost of shipping. If the shipping laboratory requests the return of empty shipping coolers, the testing laboratory bears the cost of return.

5.7 Storage of Homogenate Subsamples

5.7.1 If it is not possible to extract the sample after homogenization, then the homogenized samples may be held for a period not to exceed 72 hours at approximately -20°C or lower, or the homogenized sample may be held for longer periods of time at approximately -40°C or lower.

5.7.2 One or more adequate portions of homogenized sample shall be held in reserve for reanalysis and/or confirmation as needed. The laboratory internal SOP shall define "adequate portion" and the distribution.

5.7.3 The reserve portions of violative samples shall be retained at approximately -40°C or lower until final QA review and successful RDE transmission.

5.7.4 The reserve portion of all other samples shall be stored at approximately -40°C or lower until final QA review and successful RDE transmission. An exception to this is allowed if blank homogenates need to be released early to serve as QC matrix. Also, if freezer space is limited, non-violative homogenates may be transferred to other freezers prior to final disposition.

5.8 Storage of Extracts

Extracts shall be stored in appropriate containers (e.g., bottles, tubes, injection vials, etc.) and at appropriate temperature (approximately 4°C or lower) to protect them from degradation and solvent evaporation. **Note:** *Vials held in active autosampler trays during instrumental analysis do not require refrigeration.*

5.9 Disposal of Reserve Samples

The reserve sample may be discarded after time period(s) specified in Section 5.7 have elapsed. Each laboratory shall establish the proper procedures for disposal of its reserve samples in an internal SOP.

5.10 Disposal of Extracts

The extracts may be discarded after time period(s) specified in the laboratory's internal SOP have elapsed. Disposal shall be documented (e.g. in the refrigerator/freezer log) and shall contain, at minimum, the date of disposal, initials of the individual who discarded the sample, and sample number(s) or set number(s). Each laboratory shall establish the proper procedures for disposal (e.g., disposal by a licensed contractor) of its extracts in an internal SOP.

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Revision 10 July 2018 Monitoring Programs Division

- Added preparation instructions for kiwi (section 5.3.24)
- Rearranged and renumbered commodities in section 5.3 to correspond to the table
- In revision 9 history section, reversed snap peas and greens to correspond to sections 5.3.22 and 5.3.35
- Added requirement for a procedure to dispose reserve samples in section 5.9
- Updated section 5.1.6.1 by changing the sample viability requirement for analysis

Revision 9 July 2017 Monitoring Programs Division

- Updated section 5.1.1.3 to remove requirement for duplicate labels – only one label required
- Added section 5.1.8.4 on capturing sample identity information for non-collected samples
- Updated table in section 5.3 by adding canned peaches and dried plums
- Updated table in section 5.3 by changing raisins from section 5.4.4.1 to section 5.4.4
- Updated table in section 5.3 by adding codes for frozen cranberries and garbanzo (chick peas)
- Updated preparation instructions for greens and snap peas (sections 5.3.22 and 5.3.35)
- Removed section 5.4.4.1 and updated procedure from raisins to dried fruits
- Added preparation instructions for eggs (section 5.4.5.5)

Revision 8 July 2016 Monitoring Programs Division

- Updated section 5.1.8.3
- Added requirements for mixed lot numbers to section 5.1.1.2
- Added custody seals to section 5.1 and 5.1.3.5
- Updated section 5.3.2
- Updated table in section 5.3 by adding canned olives, canned pineapples and frozen cranberries
- Removed 90 day storage requirement for violative samples in section 5.7.3

Revision 7 July 2015 Monitoring Programs Division

- Removed sections that referenced FSIS meat collection and water (no longer part of PDP collection programs
- Updated section numbering throughout
- Updated title to section 5.1.8
- Added reference to work instructions in section 5
- Added requirement for laboratory to establish procedures to capture information from duplicate sample labels to section 5.1.1.3
- Added requirement for laboratory to contact MPD Sampling Manager if sample viability is in question to section 5.1.3
- Added option for estimated unit counting to section 5.1.3.4
- Included additional examples to section 5.1.3.5

- Updated section 5.1.8
- Removed references to commodities not tested by PDP from table in section 5.3

Revision 6 June 2014 Monitoring Programs Division

- Updated the document by replacing references of MP to MPD
- Added section 5.1.3.5 specifying MPD notification for samples with compromised integrity at receipt
- Updated table in section 5.3 by adding dairy-based and soy-based infant formula; salmon; and frozen blueberries, cherries, and potatoes
- Changed requirement throughout section 5.3 from "…allow to drain for at least 2 minutes on paper towels and a flat surface." to "…allow to drain for at least 2 minutes."
- Clarified broccoli preparation and homogenization requirement in section 5.3.9
- Added infant formula to section 5.4.5.4
- Changed reserve homogenate storage requirements in section 5.7.2
- Removed "-40" from section 5.7.4
- Corrected cross-reference in section 5.9

Revision 5 October 2012 Monitoring Programs Division

- Updated table in section 5.3 by adding avocados and raspberries
- Added avocados to Section 5.3.7 and renumbered remaining 5.3 sections
- Added raspberry to Section 5.3.8
- Renamed section 5.7 and added information on homogenate storage that was moved from section 5.3

Revision 4 July 2012 Monitoring Programs Division

- Updated purpose (section 1)
- Increased information presented in outline (section 3)
- Reorganized subsections in section 5.1 to reflect sample flow through the laboratory
- Updated RDE/SIF table in section 5.1 by adding "Required/Optional" column, clarifying specifications for required information
- Clarified requirements for samples' weights (section 5.1.6)
- Updated table in section 5.3 by adding codes for baby food applesauce, carrots, peaches, and peas
- Clarified preparation and homogenization requirements for homogenous processed commodities in section 5.4.5.1
- Reordered sections 5.5, 5.6, and 5.7
- Renamed section 5.7 from "Storage of Reserve Homogenate Subsamples" to "Storage of Homogenate Subsamples"

Revision 3 July 2011 Monitoring Programs Division

- Updated the document by changing the MPO name with Monitoring Programs Division or Monitoring Programs (MP)

- Updated requirements for samples' weights (section 5.1.8)
- Updated table in section 5.3 by adding papaya and renumbering the homogenization procedures
- Added section 5.3.30 as Papaya homogenization and renumbered all subsequent subsections
- Removed **Note** in section 5.5.3 referring to cap labeling

Revision 2 January 2011 Monitoring Programs Office

- Added the information required to be recorded in RDE/SIF as a table (in section 5.1)
- Updated requirements for recollection of samples (section 5.1.1)
- Eliminated the codes for animal tissue commodities and updated the example (section 5.1.4)
- Updated section 5.1.5 as to record in "Sample Comment" of RDE the preparation state of the fresh sample
- Updated section 5.1.6 as to record in "Lab Comment" of RDE the inadequacy of the received sample
- Updated section 5.1.7 as to record in "Reason NOT Analyzed" of RDE the lab's inability to analyze the sample
- Updated section 5.1.8 as to record in the RDE sample information the date and person that received the sample
- Updated section 5.1.10 as to record in "# of Units" of RDE the number of units for non-clustered commodities
- Added centralized table with all commodities, their PDP codes and the SOP corresponding sections to their preparation and homogenization (section 5.3)
- Rearranged and renumbered the commodities in section 5.3 to correspond to the table
- Added Tangerines to citrus group (section 5.3.29)
- Added preparation instruction for Snap Peas (section 5.3.33)
- Updated preparation instructions for Anaheim Hot Peppers to have the core and seeds removed (section 5.3.35).
- Added instruction for cherry tomatoes preparation (section 5.3.44)
- Added baby food as another example in the processed foods section (5.4.5)
- Eliminated the transshipping amounts from section 5.5.2
- Updated homogenized sample container labeling (section 5.5.3)
- Updated the retention time of violative samples from 6 months to 90 days (section 5.6.2)
- Updated requirement for -40°C homogenate subsamples' storage (section 5.6.3)
- Updated the weight and precision of analytical sample (section 5.7)

Revision 1 September 2010 Monitoring Programs Office

- Added preparation instructions for Hot Peppers (section 5.3.25).
- Added instruction for soybean transshipping (section 5.5.2).

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

SOP No.: PDP-DATA		Page 1 of 29
Title: Data and Instrumentation		
Revision: 6	Replaces: 04/01/17	Effective: 04/01/18

1. Purpose:

To provide standard procedures for:

- instruments, equipment and injection sequence used in the USDA/AMS Pesticide Data Program (PDP). See SOP PDP-ADMIN for administrative requirements, (e.g., purchase approval, PDP Equipment Inventory System, Instructions for Permission to Salvage, Transfer or Dispose of Equipment, etc.).
- quantitative and qualitative analysis of pesticide residues determined for the USDA/AMS PDP.
- data reduction, reporting, and submission by participating laboratories.

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:

4. References:

- US EPA, *Maintenance and calibration of equipment,* 40 CFR 160.63.
- US EPA, *Standard operating procedures,* 40 CFR 160.81.

- US EPA, *Conduct of a study,* 40 CFR Part 160.130
- US EPA, *Appendix A,* 40 CFR Part 136
- US EPA, *Reporting of study results,* 40 CFR Part 160.185
- US EPA, *Tolerances and Exemptions from Tolerances for Pesticide Chemicals in Food,* 40 CFR Subchapter E, Part 180
- US EPA/OPPTS, *Processed Food/Feed*, 860.1520
- FIFRA, *Pesticide Emergency Exemptions*, Section 18, 40 CFR Part 160
- FDACS, *QA/QC Guideline Document,* Section 14
- USDA/FDA, *Food and Drugs*, 21 CFR Part 175.105
- FDA Center for Veterinary Medicine, *Guidance for Industry: Mass Spectrometry for Confirmation of the Identity of Animal Drug Residues, Final Guidance,* US Department of Health and Human Services, Rockville, MD, Guide #118, May 1, 2003. http://www/fda/gov/cvm/guidance/guide118.pdf
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5. Instrumentation

5.1 SOPs and Manuals

Each laboratory shall develop SOPs for PDP equipment operation. The SOPs shall set forth in sufficient detail the methods, materials, and schedules to be used in the routine inspection, cleaning, maintenance, testing, calibration, and/or performance verification of equipment used, and shall, when appropriate, specify remedial action to be taken in the event of failure or malfunction of equipment. SOPs and operator manuals shall be readily accessible to applicable laboratory staff. Manufacturer's manuals or published literature may be used as a supplement to SOPs.

5.2 Maintenance

All instruments and other equipment used in the analysis of PDP samples shall be inspected, cleaned, and maintained in proper working condition so that the accuracy, precision, and sensitivity requirements specified in this SOP and SOP PDP-QC are met.

5.3 Performance Verification

Before being placed into service, an instrument shall undergo appropriate checks to establish that all requirements are met. See SOP PDP-QC.

5.4 Records

5.4.1 Records (e.g., logbooks) shall be maintained for all critical equipment and instruments. These records shall be used to document all routine and non-routine inspection, maintenance, and calibration activities, including the date, the identity of the personnel performing the activities, and any maintenance (routine or otherwise), repairs, or remedial actions.

5.4.2 Data packages shall reflect the specific instruments and equipment that were used to generate, measure, or assess the data. Data on the performance verification of instruments (e.g., gas chromatograph-mass selective detector (GC-MSD), etc.) utilized in the analysis of a data set are to be maintained by the laboratory. See Section 8 of this SOP for hardcopy data package requirements. See Section 7 of this SOP for mass spectrometry (MS) documentation requirements.

5.4.3 Calibration and/or performance verification data for balances, refrigerators, and other peripheral equipment do not need to be included in the hardcopy data packages, but shall be maintained by the laboratory.

5.4.4 See SOP PDP-ADMIN for records storage and archival requirements.

6. Calibration

Instruments and equipment that have significant effects on test results shall be calibrated at the minimum frequency specified in the laboratory's internal SOPs.

6.1 Calibration Integrity

6.1.1 Calibration integrity is defined as steady instrument response to a given amount of analyte over the duration of a sample run. Calibration integrity shall be determined by injecting standards at the beginning and end of a run to evaluate the variability in instrument response and any changes in retention time (see 6.1.2). Injection of a standard(s) between the beginning and end of a run also may be required. Calibration integrity shall be calculated in terms of relative percent difference (RPD), percent difference (%D), or percent relative standard deviation (%RSD) using the following equations:

$$RPD = \frac{|X_1 - X_2|}{\left[\frac{X_1 + X_2}{2}\right]} \times 100$$

where X_1 is the response of the first analytical standard injected and X_2 is the response of the second standard injected;

$$\%D = \frac{C_1 - C_2}{C_1} \times 100$$

where C_1 is the known concentration of the standard used for quantification and C_2 is the concentration of that standard calculated using the calibration curve;

$$\%RSD = \frac{SD}{avg.\ RF} \times 100$$

where SD is standard deviation:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

and RF is response factor, or the area or height of each standard divided by the concentration of that standard.

6.1.2 Standard response drift greater than 20% RPD, %D, or RSD indicate that additional standards within the run may be injected in order to attempt to meet the required 20% calibration integrity requirement. Each laboratory shall document exceptions in internal SOPs and shall determine the number of intermediate standards required throughout the run to maintain calibration integrity.

6.1.3 For cases where no residues were detected in samples and only the spike recovery is being quantitated, the requirement for calibration integrity shall be 30%.

6.2 Quantification Using Calibration Curves

6.2.1 If calibration curves are used for quantification, they shall be constructed using standards which bracket the expected range of residue concentration. A suggested range

is 1xLOQ to 10xLOQ. Second-order curves (i.e., quadratic) may be employed, providing that a sufficient number of points (i.e., minimum of five) is used to define the curve.

6.2.2 For any analyte that is quantitated using a calibration curve, the fitness of curve, whether first- or second-order, **shall** be demonstrated in the same injection sequence used to report the data by one of the following accepted methods:

- correlation coefficient (where $R \geq 0.995$ / $R^2 \geq 0.990$),
- percent relative standard deviation (where %RSD ≤ 20), or
- percent difference of calculated vs. known standard concentration in the curve (where %D is within 20%).

6.2.3 The laboratory shall specify in an internal SOP the method/parameter(s) used to demonstrate fitness of curve.

6.2.4 Results obtained using a calibration curve shall lay within the range of the calibration curve. If results fall outside the calibration curve, the sample must be diluted, the calibration curve extended, or the procedures for single point comparisons followed. The procedure for extending the range of the calibration curve shall be documented in internal laboratory procedures. Data generated to support extension of the calibration curve shall be maintained and housed with the QAU.

If method range has been extended beyond the highest validated level, then samples may be diluted for quantitation purposes; however, dilutions must be done proportionally with matrix so that the matrix concentration of the sample is similar to that of the analytical standards used to prepare the calibration curve.

6.3 Quantification Using Single Point Comparisons

Quantification using a single standard is permitted if the sample response is within 30% of the standard response for samples greater than LOQ; if it is not, dilution of the sample or injection of a different standard concentration shall be required. This difference shall be calculated using the following equation:

$$\frac{X_{standard} - X_{sample}}{X_{standard}} \times 100$$

where $X_{standard}$ is the response of the standard and X_{sample} is the response of the sample.

6.4 Quantification of Multi-Peak Compounds

Quantification of multi-peak compounds may be based on the largest peak or the sum of all of the peaks. When reporting multi-peak compounds as total (combined) values and one or more peaks are Below Quantifiable Level (BQL), determine and report the value(s) for the BQL peak(s) using either single point quantification or the value calculated by the data station based on the calibration table. If one or more peaks are less than the Limit of Detection (LOD), or LOQ where LOD=LOQ, do not include them in calculating the total (combined) value. In either case, code the reported value as an estimate "E" in the quantification field of the analytical results section.

6.5 Quantification of Spikes

6.5.1 Incurred residue levels may be subtracted from spike recovered prior to calculating the percent recovery. A laboratory may elect to subtract incurred residue levels if the following conditions are met:

- Blank matrix cannot be obtained. The laboratory shall make every effort to obtain blank matrix such as purchasing organic produce, saving analyzed samples that are pesticide free, etc.
- The incurred residue level is less than 2xLOQ.
- The laboratory shall report blank subtracted spike recovery data by entering the amount subtracted into the comments field and entering an "S" (Incurred Subtracted) code in the Exception field for that compound on the QA/QC Recovery section of the RDE.
- If a laboratory elects to subtract incurred residues, they shall have internal procedures on how to handle the subtraction process.

6.5.2 When a 2xLOQ spike recovery value falls below 50%, by definition, these spikes are quantitated using responses less than the LOQ. This is an acceptable PDP practice.

6.5.3 Incurred residues, as determined using the matrix blank, shall not be subtracted from the spike when the residue in the matrix blank exceeds 2xLOQ. If an incurred residue is greater than 2xLOQ or otherwise prevents reporting of an associated QA/QC recovery, an "I" (Incurred Residue) code shall be entered into the Exception field for that compound on the QA/QC Recovery section of the RDE for recoveries that are reported.

6.5.4 Pesticides not recovered shall be reported using an "N" (Not Recovered) code in the Exception field for that compound on the QA/QC Recovery section of the RDE for the spiked pesticide.

6.5.5 Pesticides reported as estimates shall be coded as "E" (Estimate) in the Exception field for that compound on the QA/QC Recovery section of the RDE for recoveries that are reported.

6.5.6 Pesticides reported as having matrix interference shall be coded as "M" in the Exception field for that compound on the QA/QC Recovery section of the RDE.

6.5.7 Pesticides reported as Marginal Performing Analytes shall be coded as "P" in the Exception field for that compound on the QA/QC Recovery section of the RDE.

6.5.8 Pesticides reported as unvalidated shall be coded as "U" in the Exception field for that compound on the QA/QC Recovery section of the RDE (refer to *Attachment 2, Flowchart for reporting codes).*

7. Generating Raw Data

7.1 Injection sequence description

7.1.1 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% RPD, %D, or RSD between calibration responses (refer to Section 6.1 of this) or 30% if a residue was not detected and only the spike is being quantitated.

7.1.2 Standards for each compound analyzed shall be included with every injection sequence. It is recommended that standards spanning the expected range of residue concentrations, such as 1xLOQ to 10xLOQ, be included in the sequence to allow construction of a calibration curve; however, construction of a calibration curve is not required unless a curve is used for quantification.

7.1.3 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.

7.1.4 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).

7.1.5 A non-extracted 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 (s/n), 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.

7.2 Retention Time Criteria (Selective Detection and MS Systems)

7.2.1 GC Retention Time

7.2.1.1 If an external standard is used, the retention time (RT) of the compound of interest in the standard and the RT of the same compound in the sample shall be within 0.1[1] minutes.

[1] The laboratory may perform instrument-specific retention time studies to verify stipulation of different retention time window criteria than those specified in this SOP. It is expected that a generally accepted method of retention time window calculation be used and documented to establish these criteria.

7.2.1.2 If an internal standard is used, the relative retention time (RRT) of the compound of interest to the internal standard within the reference standard and the RRT of the compound of interest to the internal standard within the sample shall be within 0.01 minutes.

7.2.2 LC Retention Time

7.2.2.1 If an external standard is used, the RT of the compound of interest in the standard and the RT of the same compound in the sample shall be within 0.1[1] minutes.

7.2.2.2 If an internal standard is used, the RRT of the compound of interest to the internal standard within the reference standard and the RRT of the compound of interest to the internal standard within the sample shall be within 0.1 minutes.

7.2.3 MS Screening for Identification

In order to maximize the number of compounds screened by MS systems while maximizing the number of scans per second and dwell times, it may be desirable to perform the initial identification and quantification using fewer than three ions for some or all of the compounds. Presumptive-positive samples shall be re-injected or data reprocessed to meet all MS confirmation criteria.

7.3 Confirmation Procedures for Selective Detection Systems

7.3.1 Where possible, mass spectral confirmation is preferred. All residues detected at concentrations that are equal to or greater than the established and verified LOD for a given analyte shall be confirmed. The method available for confirmation shall be capable of detecting the desired residue at a concentration that is equal to or less than the concentration quantitated by the primary instrument. All residues that cannot be confirmed shall be reported as non-detects. The confirmation method shall be reported (refer to *Attachment 1, PDP Laboratory Information Form (LIF) Codes*).

7.3.2 When more than one confirmation method has been utilized, the method with the higher level of confidence shall be entered in the Confirmation Method 1 field and the

method with the next highest level of confidence should be entered in the Confirmation Method 2 field. For example, if a residue is confirmed using an alternate column and mass selective detector: the laboratory would most likely enter "M" in the Confirmation Method 1 field and "C" in the Confirmation Method 2 field of the RDE. The decision regarding the level of confidence of a particular confirmation is left to the discretion of the Technical Program Manager.

Note: The Ident Points (identification points) field of the RDE is an optional field that may be used to record the degree of confirmation.

7.3.3 Acceptable confirmation methods for GC and LC analyses (element specific/selective detectors) are:

- Alternate detector (element specific/selective detectors, including various forms of mass spectrometry). All applicable confirmation criteria for that detector must be met.
- Alternate column, provided the alternate column changes the elution order or significantly changes (i.e., 2 or 3 peak widths) the retention time (RT) of the detected pesticides.
- Alternate mobile phase, provided the alternate mobile phase changes the elution order or significantly changes (i.e., 2 or 3 peak widths) the RT of the detected pesticides is an acceptable confirmation method only for LC analysis (element specific/selective detectors).

7.4 MS Confirmation Criteria

7.4.1 GC/MS and LC/MS Confirmation Criteria

7.4.1.1 A minimum of three structurally significant ions (meeting the 3:1 s/n ratio) are required for confirmation. For GC/MS, because the molecular ion is the most structurally significant ion in a mass spectrum, if it is present and meets the 3:1 s/n ratio, it is preferable that it be included as one of the three ions.

Note: If instrument conditions and/or ionization techniques limit the number of ions available, the laboratory shall request a deviation from MPD in order to report results under these conditions.

7.4.1.2 A pair of isotopic cluster ions may be used as two of the three structurally significant ions required for confirmation.

7.4.1.3 Use of fragment ions resulting from water loss to meet the three structurally significant ions requirement is discouraged.

7.4.1.4 The confidence limits of the relative abundance of structurally significant ions used for SIM and/or full scan identification shall be ± 30% (relative) when compared to the same relative abundances observed from a standard solution injection made during the same analytical run.

7.4.1.5 MS spectra produced by "soft" ionization techniques (e.g., GC/MS - chemical ionization and for LC/MS – APCI, APPI, ESI, etc.) may require additional evidence for confirmation. If the isotope ratio of the ion(s) or the chromatographic profile of isomers of the analyte is highly characteristic, there may be sufficient information for confirmation. Additional evidence may consist of MS/MS data, use of a different ionization technique, use of a different chromatographic separation system, and for LC/MS systems, altering fragmentation by changing ionization conditions.

7.4.1.6 GC/MS: Fragmentation that results from "soft" ionization techniques is highly dependent on instrument design and the conditions applied (i.e., the obtained spectra can widely differ). Commercially available spectral libraries bundled with GC/MS instruments may contain spectra generated under standard 70eV EI conditions; therefore, the use of library search software for spectra from "soft" ionization techniques could result in identification errors and is discouraged.

7.4.2 GC/MS/MS and LC/MS/MS Confirmation Criteria

7.4.2.1 Target analyte confirmation shall be performed by either (1) monitoring the transition of one precursor ion to at least two product ions, OR (2) monitoring at least two precursor-to-product ion transitions.

Multipeak compound confirmation may be based on the largest peak or the sum of all the peaks. If it is based on the sum of all the peaks, one or two of the constituents can be used for both transitions.

Note: If instrument conditions and/or ionization techniques limit the number of transitions available, the laboratory shall request a deviation from USDA/AMS in order to report results under these conditions.

7.4.2.2 The abundance of the signal from the precursor-to-product ion transition shall meet the 3:1 s/n ratio requirement.

7.4.2.3 The relative abundances of ion transitions used for compound identification in the sample shall be ± 30% (relative) when compared to the same relative abundances observed from a standard solution analyzed during the same analytical run if more than one precursor-to-product ion transition is monitored. The ion ratio tolerance shall be calculated using the following example: If the ion ratio (qualifier area count/target area count) is 15%, the acceptable range will be 15%+/-4.5 or 10.5% to19.5%.

7.4.2.4 Use of product ions resulting from water loss for identification is discouraged.

Note: Any information that provides a contraindication of identity of the residue will be addressed in the internal SOP by the laboratory.

7.5 MS Documentation Criteria

Structurally significant ions and/or precursor-to-product ion transitions used for confirmation shall be documented.

8. Data Handling

8.1 Raw Data Handling

8.1.1. Hardcopy raw data are defined as any laboratory worksheets, logbooks, records, notes, chromatograms, calculations, instrument printouts, and any other data, which are the result of original observations and activities. Electronic raw data are the files generated by the instrument system.

8.1.2 For manual entry, hardcopy raw data shall be recorded directly, promptly, and legibly in permanent ink. Pencil or erasable pen is not acceptable. All data entries shall be dated on the date of entry and signed or initialed by the person entering the data. Each individual error shall be corrected using a single-line cross out (no white-out). It is recommended, but not required, that the reason for the correction be indicated. Each correction shall be dated and initialed. Documented error codes may be used to explain errors. Correction of multiple errors may be accomplished in the following manner:

- On first occurrence of the error, or on a summary sheet, make/indicate the appropriate correction, including date, initials, explanation of error/error code, and all affected subsequent entries.
- Each subsequent occurrence of the error must then be corrected, dated, and initialed.

8.1.3 Each participating laboratory shall ensure sample and data traceability for raw and electronic data collection and processing. Chromatograms that have been reprocessed through the data system shall be clearly labeled.

8.1.4 Each participating laboratory shall maintain a log of names, initials, and signatures for all individuals who are responsible for signing or initialing any laboratory record.

8.2 Hardcopy Data Package Requirements

8.2.1 Routine sample data packages and method validation data packages retained by the participant laboratory shall consist of laboratory records (i.e., worksheets and/or completed forms), USDA collection and report forms (where applicable), and supporting technical data in the form of chromatograms and integration reports, calculations, and derived data. Data requirements consist of two types, instrument and chromatographic. The following information shall be included in the data package.

8.2.1.1 The instrument method shall be included or referenced. Instrument information shall be traceable. Examples may consist of instrument type and identifier, detector type, injection volume, temperature parameters (injector, detector, oven), analytical column parameters (phase, film thickness, diameter, length), and instrument parameters (integration threshold, attenuation, timed events).

8.2.1.2 Chromatographic information shall be traceable. Examples may consist of sample ID, analyst name, dilution information, and date and time of injection.

8.2.2 At a minimum, hardcopies of data sets shall include the following:

- Instrument methods, or references to them (data acquisition, calibration/standardization, and data analysis parameters)
- Injection sequences
- Chromatograms and/or instrument reports of samples, standards, reagent blanks, matrix blanks, and matrix spikes
- PDP Sample Information Forms (SIFs) [if paper SIFs were submitted by the Sample Collector]
- Matrix blank, reagent blank, matrix spike, and sample results
- Documentation of technical and QA review

Note: Laboratories that choose to retain electronic data sets as.pdf or Excel files shall ensure all requirements for QA, traceability, etc. are met. Nothing shall be lost in the electronic domain that would normally be captured on paper, and all markups of the original chromatogram shall also be retained.

8.2.3 Hardcopies of method validation data packages submitted to USDA/AMS shall include copies of the summary reporting forms, narrative describing the method, and cover memo submitted to the PDP Technical Director, Method Validation Coordinator, and liaison chemist (refer to SOP PDP-QC).

9. Data Reporting

9.1 Calculations and Significant Figures

9.1.1 Each laboratory shall have an internal SOP describing the data processing steps taken to reach the final reported concentration. Data shall not be ignored without a written explanation (e.g., instrument malfunction, wrong standard used, co-eluting peak, etc.).

9.1.2 In calculations, at least one significant figure in excess of the reporting requirements shall be carried through the calculation. When rounding is required, values greater than or equal to 5 shall be rounded up.

9.1.3 Percent recoveries shall be reported to two significant figures if less than 100 or to three significant figures if greater than 100.

9.1.4 Concentrations shall be reported to at least two significant figures in parts per million (ppm), parts per billion (ppb), or parts per trillion (ppt). The laboratory may elect to report more than two significant figures. If more than two significant figures are reported, it is the laboratory's responsibility to determine the appropriate number of significant figures for each commodity/pesticide pair using a given method.

9.1.5 Individual peaks may be reported for multiple peak compounds. If separate standards are available for separate isomers, it is preferable to report the isomers separately.

9.2 Determination of Residue Concentrations for PDP Reporting Purposes

9.2.1 A laboratory may elect to set LOD = LOQ provided all of the following conditions are met:

- the analyses are completely performed via MS systems (i.e., quantification and self-confirmation) **and**
- the qualifier ions are at least 3 x s/n **and**
- the quantification ions have a response at least 10 x s/n.

The laboratory shall code the findings (both detects and non-detects) as "Z" [LOD equals LOQ] in the "Test Class" section of the RDE analytical results section.

9.2.2 Do not report residue concentrations less than the verified LOD.

9.2.3 Compounds appearing on the analytical results list for which results are not/cannot be reported shall be coded as "M" [not analyzed (e.g., compound not in standard, used as marker only)] or "UD" [unable to determine (e.g., matrix interference, method failure)] in the mean result field of the RDE analytical results section.

9.2.4 Numeric concentrations below the LOQ are considered low confidence values associated with a qualitative finding. A concentration value is not required when a pesticide is detected at or above the determined LOD and below the determined LOQ. The laboratory shall code the finding as "Q" (residue BQL) in the "Annotated Info." section of the RDE analytical results section. The concentration will be converted to ½ LOQ in the PDP database for reporting purposes.

9.2.5 All detections shall be coded as:

- "O" (detect – original extraction value);
- "A" (detect – average of original and re-extraction analyses values); or
- "R" (detect – re-extraction analysis value)

in the mean result field of the RDE analytical results section (refer to *Attachment 2, Flowchart for Reporting Codes)*.

9.2.6 Validated Pesticide/Commodity Pairs

A pesticide/commodity pair is considered validated when all applicable modules in SOP PDP-QC have been met.

9.2.6.1 Results less than the verified LOD shall be coded as "ND" (non-detect, well-recovered analyte) in the mean result field of the RDE analytical results section.

9.2.6.2 Residue concentrations greater than or equal to the LOQ shall be reported on the RDE analytical results section. If there are no qualifications of the data (i.e.,

estimate, marginal performing analyte, unvalidated compound), the quantification field shall be left blank. If the data is an estimate (e.g., has failed linearity, calibration integrity or spike recovery), then the results shall be coded as "E" (estimate) in the quantification field of the RDE analytical results section (refer to *Attachment 2, Flowchart for Reporting Codes)*.

9.2.7 Validated Marginal Performing Analytes

Marginal Performing Analytes are identified and documented during method validation or during ongoing QC.

9.2.7.1 Results less than the verified LOD shall be coded as "NP" (non-detect, marginal performing analyte) in the mean result field of the RDE analytical results section.

9.2.7.2 Residue concentrations greater than or equal to the LOQ shall be reported on the RDE analytical results section. Results shall be coded as "P" (marginal performing analyte) in the quantification field of the RDE analytical results section (refer to *Attachment 2, Flowchart for Reporting Codes)*.

9.2.8 Unvalidated Pesticide/Commodity Pairs

As a rule, unvalidated residues should not be reported. However, unvalidated residues may be reported on a case-by-case basis. For example, identification and tentative quantification of a compound not currently included in the analytical screen or preliminary results for special projects. Procedures to be followed in these instances are as follows:

9.2.8.1 Results less than the estimated LOD shall be coded as "NU" in the mean result field of the RDE analytical results section.

9.2.8.2 Residue concentrations greater than or equal to the LOQ shall be reported on the RDE analytical results section. Results shall be coded as "U" (unvalidated analyte) in the quantification field of the RDE analytical results section (refer to *Attachment 2, Flowchart for Reporting Codes)*.

9.2.9 In cases where calibration integrity exceeds 20%, the laboratory shall use best professional judgment to determine whether or not to report positive findings as follows:

- Report positive findings using quantification codes: "E" (Estimate), "P" Marginal Performing Analyte, or "U" (Unvalidated Compound). The use of code "E" does not require a deviation letter and should be determined on a set-to-set basis, using best professional judgment. It could be used when the calibration integrity, linearity, or the spike recovery fail.

- Report results that could not be quantified as non-detects using mean result code "UD" (refer to *Attachment 2, Flowchart for Reporting Codes)*.

9.3 Administrative Reporting Level

The Administrative Reporting Level is a level below which results shall be reported as not detected. For all commodities, it is 1 ppb (parts per billion). A laboratory's reported LOD may be at or above this level, but not below.

9.4 Reporting o-Phenylphenol

O-phenylphenol has multiple uses as an antimicrobial agent. It is listed in 21 CFR as an indirect food additive as a component of a sanitizing solution. O-phenylphenol also has a number of tolerances established for various food commodities. Therefore, when detected, it cannot be determined whether residues result from the application of o-phenylphenol to a given commodity or from unintended contact with o-phenylphenol via packaging or environmental sources such as typical cleaning agents. PDP's reporting policy for residues of o-phenylphenol for all commodities is as follows:

- If no tolerance is established for a given commodity, o-phenylphenol will not appear on that commodity's list of requested compounds. Do not validate or report o-phenylphenol for that commodity.
- If a tolerance is established, o-phenylphenol will appear on that commodity's list of requested compounds. Attempts shall be made to validate and report o-phenylphenol.

9.5 PDP Tolerance Table

9.5.1 USDA/AMS maintains a PDP Tolerance Table adapted for current PDP samples. The table is available to PDP participants on the USDA/AMS Extranet. It lists tolerances (and FDA action levels for compounds with revoked tolerances that are persistent and may still be found) for many but not all registered pesticides on current PDP commodities and may or may not include the same compounds as those listed in a particular Commodity Compound List Memorandum (also available on the USDA/AMS Extranet). The PDP Tolerance Table includes permanent, interim, regional, and Section 18 emergency tolerances. Blank spaces in the table indicate that no tolerance is established. Tolerances for metabolites are based on the parent unless there is a specific tolerance for the metabolite.

9.5.2 The PDP Tolerance Table is intended to be used only as a general guide and is prepared for the convenience of the participants. The tolerance information should not be used for enforcement, or domestic/international trade issues, without verifying the completeness and accuracy of this tolerance information. The information may be out-of-date because new pesticide tolerances may be promulgated by EPA at any time and existing tolerances may be revised/revoked at any time following EPA review. EPA's new/revised/revoked tolerances are published as issued in the daily Federal Register. The PCNG is a monthly subscription service that reviews tolerance information published in the Federal Register and publishes cumulative monthly updates. The PDP Tolerance Table is updated approximately quarterly to reflect any changes to pesticide-commodity tolerances. Laboratories are encouraged to notify USDA/AMS if they become aware of any newly registered pesticides or find errors in the PDP Tolerance Table.

9.6 Non-violative results

Non-violative results for PDP reporting purposes are residue determinations that do not exceed a stated tolerance. A tolerance is the maximum amount of a pesticide residue that is permitted in or on a food. All concentrations shall be reported on the RDE analytical results section.

- A detected residue concentration is considered to be non-violative if it is equal to or less than the 40 CFR 180 tolerance for the given commodity.
- If no commodity tolerance exists then the group tolerance (if available) should be used.

- If no commodity or group tolerance is established or Section 18 reference noted, the tolerance shall be considered zero.

9.7 Presumptive Tolerance Violations (PTV)

Tolerances are established for food commodities by EPA under the authority of the Federal Food, Drug, and Cosmetic Act (FFDCA) and are listed in 40 CFR 180. Tolerances are usually established for a specific commodity, however, tolerances may also be established by the commodity groupings established by EPA in 40 CFR 180 or Section 18 tolerances may apply.

9.7.1 A residue is considered to exceed the 40 CFR 180 tolerance when the reported value exceeds the tolerance by one number in the second significant figure, or in the case of a single significant figure in the tolerance expression, by one number in that significant figure. For example, if the tolerance is 20 ppm, then a "presumptive violation" would occur at 21 ppm. If the tolerance is 1.0 ppm, then a "presumptive violation" would occur at 1.1 ppm. If the tolerance is 1 ppm, then a "presumptive violation" would occur at 2 ppm.

9.7.2 If the pesticide residue exceeds the established tolerance or does not have an established tolerance, the laboratory shall report the appropriate code in the annotated information field of the RDE analytical results section (refer to *Attachment 1 – Laboratory Information Form (LIF) Codes*).

9.7.3 PTV Notification Policy

PTVs shall be transmitted via RDE during normal data submission process. USDA/AMS shall notify HQ FDA. If States have a cooperative agreement with local FDA, USDA/AMS will also send a State-specific report to the laboratories, if requested.

9.8 Tolerance Interpretation for Processed Commodities

9.8.1 USDA/AMS shall follow the guideline of the EPA Office of Prevention, Pesticides and Toxic Substances (OPPTS) 860.1520, processed food/feed, section (b), which states in part:

"If residues do concentrate in a processed commodity, a food or feed additive tolerance must be established under section 409 of the Federal Food, Drug and Cosmetic Act (FFDCA) (or a section 701 Maximum Residue Limit (MRL) in some cases). However, if residues do not concentrate in processed commodities, the tolerance for the raw agricultural commodity (RAC) itself applies to all processed food or feed derived from it."

9.8.2 When a specific tolerance for a compound is listed for a processed commodity in the Code of Federal Regulations (CFR), that tolerance will be stated in the quarterly tolerance tables. For example, 40 CFR 180.472 lists a specific tolerance for imidacloprid in tomato paste at 6.0 parts per million (ppm). This is the tolerance that will be listed for tomato paste in the tolerance table scheduled for next quarterly release.

9.8.3 If a specific tolerance for a compound is not listed in the CFR for a processed commodity, then the tolerance for the RAC will be listed in the PDP tolerance table. For example, 40 CFR 180.303 does not list a specific tolerance for oxamyl in tomato paste; however, there is a tolerance of 2 ppm for oxamyl on tomatoes, the RAC. A tolerance of 2 ppm for oxamyl will be listed for tomato paste in the next released tolerance table.

9.8.4 For juices, the tolerances for the RAC will be listed in the tolerance tables unless specific tolerances for juices are listed in the CFR. When adding water to juice concentrate, do not back-calculate for the water added. Reconstituted juices should be treated the same as ready-to-serve (RTS) juices. USDA/AMS will apply the RAC tolerance for a compound, as is, to RTS juice unless there is a specific juice tolerance in the CFR. For example, 40 CFR 180.608 lists a tolerance for spirodiclofen in grape juice at 2.4 ppm. This tolerance applies to both the RTS juice and the grape juice concentrate, after it is reconstituted. A tolerance of 2.4 ppm for spirodiclofen in grape juice is reflected in the current tolerance table. Another example is that 40 CFR 180.157 does not list a specific tolerance for mevinphos in grape juice. However, there is a specific tolerance listed at 0.5 ppm for grapes, the RAC. A tolerance of 0.5 ppm for mevinphos for the RAC is reflected for RTS grape juice and grape juice concentrate, after it is reconstituted, in the current tolerance table.

9.9 Reporting Proficiency Testing (PT) Results

Results for PT rounds issued by the CDFA-QAU and FAPAS shall be reported according to the provider's instructions. Laboratories also may report PDP PT results to USDA/AMS via RDE.

10. Data Review

10.1 Each data package shall undergo review by the technical and QA sections for accuracy and completeness, adherence to PDP criteria, and integrity of the overall quality system. The QAU shall have access to all documentation necessary to achieve this objective. Both technical and QA reviews shall be documented.

10.2 Following QAU review of a data package, that data shall not be changed by any laboratory personnel unless as a response to comments/concerns/recommendations by the QAU. Actions taken as a result of technical and/or QA findings shall be documented.

11. Remote Data Entry (RDE) System

11.1 RDE System Administration

11.1.1 Each laboratory and/or TPM shall designate an individual or individuals to administer applicable aspects of the RDE system. USDA/AMS shall create or modify the RDE account for the designated individual to grant laboratory system administrator privileges.

Note: For laboratories that do not interact extensively with the RDE (i.e. those that upload/transmit from internal LIMS) a local system administrator is optional. The lab may choose to have USDA/AMS perform occasional administrative functions.

11.1.2 The laboratory system administrator shall create RDE user accounts for laboratory personnel using the Maintain User option on the RDE System Admin menu. Each user account shall be assigned one or more roles, which serve as defined permissions to access the different RDE options, based on position requirements.

11.1.3 The laboratory system administrator may reset passwords and unlock accounts as needed using the Maintain User option in the RDE System and shall disable the RDE user account when an individual terminates employment with the organization.

Note: The RDE system will automatically deactivate any account not accessed in the past 90 days.

11.2 RDE System Access

11.2.1 The RDE system requires a Web browser and an assigned user account and password to gain access. Laboratory users shall access the secured RDE site by preceding the Web address with "https" for encrypted data communication between the central server and the user's workstation.

11.2.2 Access to the RDE system is restricted to computers in the laboratories and at USDA/AMS based on a list of acceptable internet protocol (IP) addresses that indicate the internet connection points for the computers. Laboratories shall notify USDA/AMS if access to RDE is denied on a laboratory computer.

11.3 RDE Data Entry

11.3.1 The laboratory shall create analytical sets, referred to as Groups in RDE, so that all samples related to the corresponding set's QA Recovery Data, are included under one unique Group identification number. Multiple Groups for the same commodity and month are acceptable.

11.3.2 Matrix Spike Recovery data shall be entered that are associated to all samples in the Group as specified in SOP PDP-QC.

11.3.3 Sample identity information for collected and non-collected samples shall be entered from a paper SIF or attached to the Group if an electronic SIF was submitted. Ensure that the sample identification information match the information that is recorded in RDE.

11.3.4 Analytical Results data shall be entered for each sample as specified in this SOP.

11.3.5 Process Control spike recovery data shall be entered for each sample as specified in SOP PDP-QC.

11.3.6 Data may be entered and maintained on a Laboratory Management Information System (LIMS), but shall be imported into the RDE System for sign-off and transmission to USDA/AMS.

11.3.7 Refer to the latest RDE System documentation for further information.

11.4 RDE Data Sign-off and Transmission

11.4.1 The data must go through a multi-level review and sign-off process prior to submission to USDA/AMS (the RDE system provides for up to three reviewer sign-offs for each analytical set). The first level sign-off is optional, while the TPM and Quality Assurance Officer sign-offs are required before the analytical set is allowed to be transmitted. A proxy sign-off for the TPM and/or QAO can be done for data sets that are imported from a LIMS provided that the TPM and QAO have both reviewed and approved the data. Data may be maintained on a LIMS, but must be transmitted through the web-based RDE system.

11.4.2 Data shall be electronically transmitted to USDA/AMS as described in this SOP using the Transmit option in the RDE System. Analytical data on any other media shall not be submitted without prior authorization from USDA/AMS.

11.4.3 Participating laboratories shall submit electronic results for routine data sets to USDA/AMS via RDE within 90 days of receipt of the last sample in the set according to established procedures as detailed in this SOP. If the 90 day reporting requirement is not met, the laboratory shall send the PDP Technical Director monthly updates detailing the reason for the delay and a projected schedule for data delivery.

11.4.4 USDA/AMS and the laboratory will come to a written agreement, on a case-by-case basis, regarding any changes to be made to program data after it has been reported to the PDP database. The laboratory shall be responsible for making any changes to hardcopies and their own internal database/records.

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ORIGINAL SIGNATURE PAGE MAINTAINED BY USDA, AMS, SCIENCE & TECHNOLOGY, MONITORING PROGRAMS DIVISION
ELECTRONICALLY REPRODUCED SIGNATURE

Revision 6 April 2018 Monitoring Programs Division

- Changed GC and LC ion ratio criteria to +/- 30% relative in sections 7.4.1.4 and 7.4.2.3
- Added analyte confirmation language and ion ratio tolerance example calculation to section 7.4.2
- Added electronic data retention requirement to section 8.2.2
- Updated language for PT reporting in section 9.9
- Removed sections 9.9.1 and 9.9.2
- Updated language to address restricted access to RDE in section 11.2.2
- Added new codes for determinative method to Attachment 1

Revision 5 April 2017 Monitoring Programs Division

- Added multipeak confirmation criteria to section 7.4.2.1
- Clarified sample identity information in section 11.3.3

Revision 4 February 2016 Monitoring Programs Division

- Updated language for quantification of multi-peak compounds in section 6.4
- Updated language for quantification of spikes in section 6.5
- Changed GC and LC retention time criteria to 0.1 minutes in section 7.2
- Clarified tolerances for metabolites are based on parent levels in section 9.5

Revision 3 November 2014 Monitoring Programs Division

- Updated MPD address
- Updated procedures for method validation package submission to section 8.2.3
- Updated PDP Tolerance Table procedures to section 9.5
- Updated RDE System Access procedures to section 11.2
- Added new codes for determinative method, extraction, and test class to Attachment 1

Revision 2 August 2013 Monitoring Programs Division

- Updated MPO to USDA/AMS throughout document
- Changed requirement for calibration integrity to 30% for residues not detected in routine samples in sections 6.13 and 7.1.1
- Clarified reporting exceptions in section 6.5
- Added requirement for name/initials/signature log to section 8.1.4

Revision 1 April 2011 Monitoring Programs Office

- Added paragraph regarding sample dilution in section 6.2.4.
- Added specification about incurred residues in section 6.5.3.
- Added sections 6.5.5-6.5.8 regarding spike coding in RDE.
- Updated section 7.1.1 for redundant information.

- Changed "shall" with "should" in section 7.3.2 to reflect MPO needs.
- Updated section 7.4.2.3.
- Updated section 9.1.1 by taking out "best two out of three" requirement.
- Updated section 9.8.4 with relevant pesticide example.
- Updated Attachments 1 and 2 with QA Codes

Original Revision April 2010 Monitoring Programs Office

- Combined all PDP DATA (03, 07, 09) and INSTR (04, 06) into a single document as follows:
  - PDP – INSTR 04 is section 7.1
  - PDP – INSTR 06 is section 5 (Instrumentation)
  - PDP – DATA 03 is section 6 (Calibration)
  - PDP – DATA 07 is spread over sections 8 (Data Handling), 9 (Data Reporting), 10 (Data Review) and 11 (RDE System)
  - PDP – DATA 09 is section 7 (Generating Raw Data)
- Removed requirements to check instruments performance verification before/during analysis from old PDP - INSTR 06, section 5.4.b.
- Removed requirements to include comment in SIF field when using "E" code, from old PDP – DATA 03, section 5.1.c.1, currently section 9.2.9.
- Moved and reworded section 5.1.c from old PDP – DATA 03 to chapter 9 (Data Reporting), section 9.2.9 of current PDP – DATA.
- Updated section 5.3.b from old PDP – DATA 07 (currently section 8.2.2)
- Reworded section 5.5 from old PDP – DATA 07 (currently section 9.3)
- Updated section 5.16.c from old PDP – DATA 07 (currently section 11.4.3)

Pesticide Data Program
Laboratory Information Form Codes

CONFIRMATION CODES	
CODE	CONFIRMATORY METHOD (Instrumental method used to confirm analyte identity)
A	GC/AED - Gas Chromatography with Atomic Emission Detector
C	GC or LC Alternate Column
CD	GC or LC Alternate Column and Alternate Detector
D	GC or LC Alternate Detector
F	Liquid Chromatography with Fluorescence Detector
GF	GC/TOF - Gas Chromatography with Time of Flight Mass Spectrometry
GI	GC/MS/MS - Gas Chromatography with Tandem Mass Spectrometry - ion trap
GN	GC/MSD w/ Negative Chemical Ionization (NCI)
GT	GC/MS/MS - Gas Chromatography with Tandem Mass Spectrometry - triple quadrupole
HR	GC or LC High Resolution Mass Spectrometry
I	GC/IT - Gas Chromatography with Ion Trap Mass Spectrometry - single stage
IA	Immunoassay
LF	LC/TOF - Liquid Chromatography with Time of Flight Mass Spectrometry
LI	LC/MS - Liquid Chromatography with Ion Trap Mass Spectrometry - single stage
LL	LC/MS/MS - Liquid Chromatography with Tandem Mass Spectrometry - ion trap
LS	LC/MS - Liquid Chromatography with Mass Spectrometry - single quadrupole
LU	LC/MS/MS - Liquid Chromatography with Tandem Mass Spectrometry - triple quadrupole
M	GC/MS - Gas Chromatography with Mass Spectrometry - single quadrupole
MO	Quantitation & Confirmation by GC/MS only
MR	GC or LC Mid Resolution Mass Spectrometry
P	LC-AMP - Liquid Chromatography Alternate Mobile Phase
R	LC-DAD - Liquid Chromatography with Diode Array Detector
S	GC or LC -MS Alternate Detector (see PDP-Data-03.5.7)
Z	Other

ANNOTATION CODES	
CODE	ANNOTATED INFORMATION (Additional information about analyte finding)
Q	Residue at Below Quantifiable Level (BQL)
QV	Residue at <BQL> with a Presumptive Violation - No Tolerance
QX	Residue at <BQL> with a Presumptive Violation - Exceeds Tolerance
V	Residue with a Presumptive Violation - No Tolerance
X	Residue with a Presumptive Violation - Exceeds Tolerance

QUANTITATION CODES	
CODE	QUANTITATION (Method used to calibrate, quantitate or validate analyte)
(none/blank)	No qualifications of data or non-detect
E	Estimate
P	Marginal performing analyte
U	Unvalidated compound

MEAN RESULT CODES	
CODE	MEAN RESULT (Summary of analyte findings and how they were determined)
O	Detect: original extraction value
R	Detect : re-extraction analysis value
A	Detect: average of original and re-extraction analyses values
ND	Non-detect: validated, well-recovered analyte
NP	Non-detect: marginal performing analyte
NU	Non-detect: unvalidated residue
M	Not analyzed (not in standard, used as a marker only)
UD	Unable to determine (matrix interference, method failure)

QA/QC Codes (Exception Codes)	
CODE	QA/QC RESULT (Summary of spike recoveries)
I	Incurred residue when levels>2xLOQ
N	Not recovered
S	Incurred subtracted
E	Estimate
M	Matrix interference
P	Marginal performing analyte
U	Unvalidated

Pesticide Data Program
Laboratory Information Form Codes

DETERMINATIVE CODES	
CODE	DETERMINATIVE METHOD (Instrumental method used to quantitate analyte)
01	GC/ECD - Electron Capture Detector
02	GC/FPD - Flame Photometric Detector in Phosphorus Mode
03	GC/FPD - Flame Photometric Detector in Sulfur Mode
04	GC/ELCD - Electrolytic Conductivity Detector in Nitrogen Mode
05	GC/ELCD - Electrolytic Conductivity Detector in Halogen Mode
06	GC/FID - Flame Ionization Detector
07	GC/MS - Gas Chromatography with Mass Spectrometry - single quadrupole
08	GC/IT - Gas Chromatography with Ion Trap Mass Spectrometry - single stage
09	TLC - Thin Layer Chromatography
10	LC/FL - Liquid Chromatography with Fluorescence Detector
11	LC/UV - Liquid Chromatography with UV Detector
12	Liquid Chromatography with Post-Column Derivatization & Fluorescence Detection
14	GC/NPD - Phosphorus Mode
15	GC/NPD - Nitrogen Mode
16	GC/NPD - Nitrogen/Phosphorus Detector
18	GC/FPD - Flame Photometric Detector in Nitrogen Mode
19	Liquid Chromatography with Pre-Column Derivatization & Fluorescence Detection
27	GC/AED - Atomic Emission Detector
28	AED - Element Selective GC/AED
30	GC/ELCD - Electrolytic Conductivity Detector in Sulfur Mode
35	GC/MS/MS - Gas Chromatography with Tandem Mass Spectrometry - triple quadrupole
51	LC/MS - Liquid Chromatography with Mass Spectrometry - single quadrupole
52	LC/MS/MS - Liquid Chromatography with Tandem Mass Spectrometry - triple quadrupole
58	GC - Gas Chromatography w/ Detector other than Listed
59	LC - Liquid Chromatography w/ Detector other than Listed
60	GC/XSD - Halogen Specific Detector
63	Second LC/MS
64	Second LC/MS/MS
65	GC/Micro ECD - Micro Electron Capture Detector
66	GC/PFPD - Pulsed Flame Photometric Detector
67	Third LC/MS/MS
68	Second GC/ECD
70	Fourth LC/MS/MS
71	Second GC/Micro ECD
72	GC/MSD with Negative Chemical Ionization (NCI)
73	GC/MS/MS - Gas Chromatography with Tandem Mass Spectrometry - ion trap
74	LC/MS - Liquid Chromatography with Ion Trap Mass Spectrometry - single stage
75	LC/MS/MS - Liquid Chromatography with Tandem Mass Spectrometry - ion trap
76	GC/TOF - Gas Chromatography with Time of Flight Mass Spectrometry
77	LC/TOF - Liquid Chromatography with Time of Flight Mass Spectrometry
78	Second GC/MS - single quadrupole
79	GC/HRMS-Gas Chromatography with High Resolution Mass Spectrometry
80	LC/HRMS-Liquid Chrmatography with High Resolution Mass Spectrometry
98	Immunoassay Screen
99	OTHER

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

Pesticide Data Program
Laboratory Information Form Codes

EXTRACTION CODES	
CODE	EXTRACTION METHOD (Extraction method used for this analyte)
000	No Extraction Necessary
015	Modified Luke Extraction Method without Cleanup for Multi-Residues & Carbamates
550	CDFA Lee et al C-18 Extraction Method
551	CDFA Chlorinated ACN Florisil SPE Extraction Method
552	CDFA MSD Aminopropyl Extraction Method
553	CDFA Carbamate SPE Extraction Method
554	CDFA Organophosphate Florisil SPE Extraction Method
555	CDFA Chlorinated Aminopropyl SPE Extraction Method
556	CDFA LC compounds Florisil SPE Extraction Method
800	FL-Modified CDFA C-18 Extraction Method (P-fraction)
801	FL-Modified CDFA C-18 Extraction Method Aminopropyl SPE Cleanup
802	FL-Modified CDFA C-18 Extraction Method w/ Florisil SPE Cleanup
803	GIPSA Modifed Method for Extraction of Multi-Residues in Grains
804	GIPSA Modified Method for Determination of Triazole Metabolites in Wheat Flour (SPE, LC/MS-MS)
805	Modified Quecher's Method
806	NYS Modifed SPE Method (F&V)
807	NYS Modified Method for Determination of Triazoles and Metabolites in Peaches (SPE, LC/MS-MS)
808	WSDA Modified Method for Determination of Triazoles and Metabolites in Apples (SPE, LC/MS-MS)
809	NSL Butter Extraction Method
810	Montana SPE Triazole Extraction Method for Water
811	Montana SPE Extraction Method for Polar Pesticides (Water)
812	Montana Liquid/Liquid Extraction Method for Non-Polar Pesticides
813	NSL Dairy Product Method
814	WA-Modified CDFA C-18 Extraction Method (P-fraction)
815	WA-Modified CDFA C-18 Extraction Method Aminopropyl SPE Cleanup
816	WA-Modified CDFA C-18 Extraction Method w/Florisil SPE Cleanup
817	FL Aminopropyl SPE Extraction Method
818	NSL Animal Tissue Extraction Method
819	EPA Extraction Method
820	Phenoxy Extraction Method
821	NSL Honey Extraction Method
822	CDFA-Modified QuEChERS Method
823	WSDA Animal Tissue Extraction Method
900	Liquid/Liquid Method
901	NYS Modification of USGS Method 2001/2002 (SPE, GC)
902	NYS Modification of USGS Method 9060 (SPE, LC)
903	NYS Modification of USGS Method for Chloroacetanilide Metabolites (SPE, LC)
997	OTHER Methods Used for Determination of Single Components
998	OTHER Single-Analysis Methods
999	OTHER Multi-Residue Methods

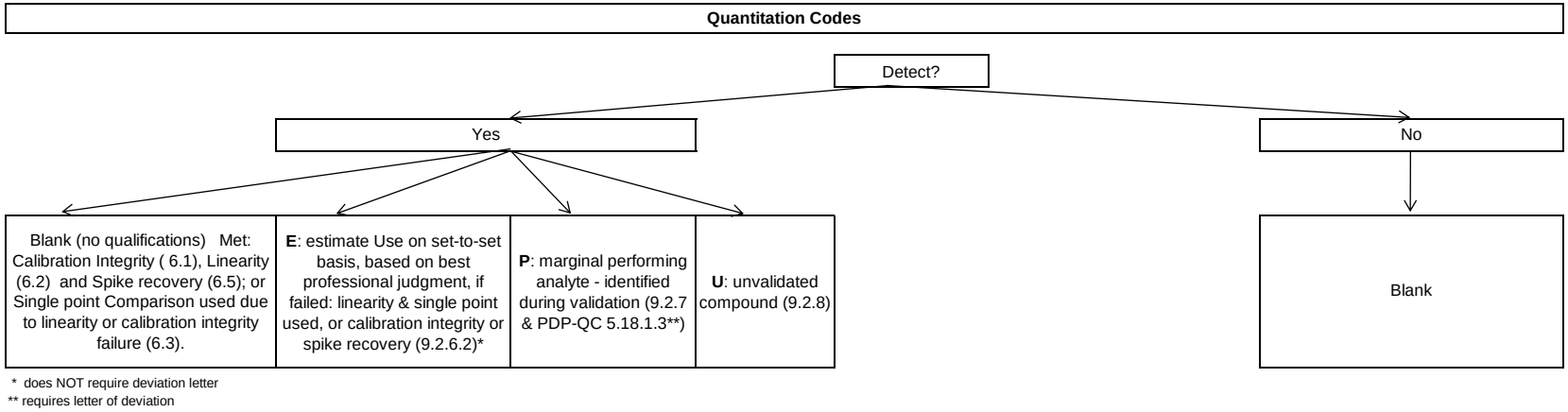
Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

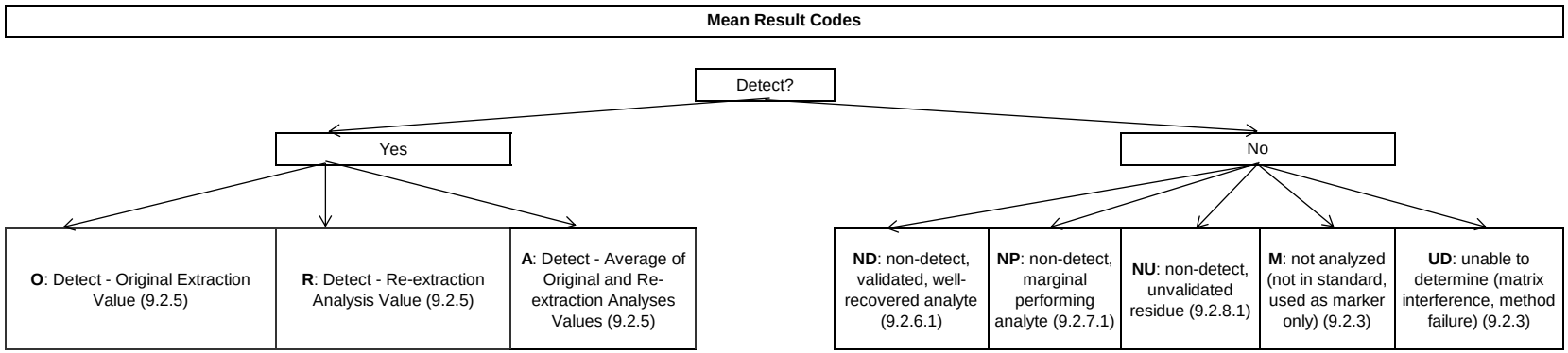
Pesticide Data Program
Laboratory Information Form Codes

TEST CLASS CODES	
CODE	TEST CLASS (Test classifications for analytes)
A	Halogenated
B	Benzimidazole
C	Organophosphorus
D	Avermectin
E	Carbamate
F	Organonitrogen
G	2,4-D / Acid Herbicides
H	Formetanate HCL
I	Other Compounds
J	Imidazolinone
K	Sulfonyl Urea Herbicides
L	Conazoles / Triazoles
M	Dithiocarbamates
N	Imidazoles
O	Pyrethroids
P	Thiocarbamates
Q	QA only (for RDE)
R	Triazines
S	Triazine, Non-Halogenated
T	Nitrile
U	Uracil
V	Pyrimidone
W	Morpholine
X	Natural Pesticides
Z	LOD equals LOQ (for RDE)

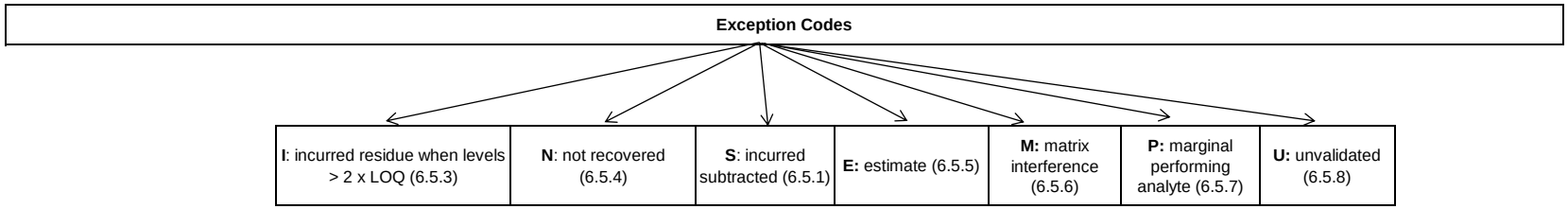
Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

1) Sample Results (LIF Codes)





2) QA/QC Results (QA/QC Codes)



United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program
Glossary of Mass Spectrometry Terms and Acronyms

Adduct ion: Ion formed by the interaction of the molecular ion and another compound or element (e.g., ammonium, hydrogen, sodium, etc.) as a result of van der Waals forces.

Atmospheric pressure chemical ionization (APCI): Ionization process where an aerosol of sample solution is sprayed at atmospheric pressure into a heated region creating a reaction between a reagent ion and a neutral molecule to create a charged ionic form of the molecule.

Atmospheric pressure ionization (API): Ionization process carried out at atmospheric pressure by any of several procedures including a radioactive source, electrical discharges, light sources, and high voltage electric fields. The main types are APCI, APPI, and ESI.

Atmospheric pressure photo ionization (APPI): Ionization process where an aerosol of sample solution is sprayed at atmospheric pressure into an area with a light source creating a reaction between photons and a neutral molecule to create a charged ionic form of the molecule.

Atomic mass unit (amu): An arbitrarily defined unit in terms of which the masses of individual atoms are expressed. One amu is exactly 1/12 of the mass of an atom of the nuclide ^{12}C (the predominant isotope of carbon).

Base peak: The ion with the most intense peak in the mass spectrum (full scan). The relative abundance of the base peak is assigned a value of 100%, and the abundance of all other ions plotted in that reference spectrum are normalized to that value.

Chemical ionization (CI): Ionization process initiated by the reaction of a reagent ion and a neutral molecule to create a charged ionic form of the molecule.

Collision induced dissociation (CID): Process by which an isolated ion is fragmented, producing an MS/MS spectrum. CID is sometimes called collision activated dissociation.

Confidence limits: The upper and lower boundaries in the range of values which includes (with a pre-assigned probability called the confidence level) the true value of a parameter.

> "Absolute" confidence limits: Confidence limits determined for relative abundances of structurally significant ions by adding ± the pre-assigned confidence level. For example, an absolute confidence limit of 15%, for ion 149 with a relative abundance of 45%, the confidence interval would be 30% to 60%.
>
> "Relative" confidence limits: Confidence limits determined for relative abundances of structurally significant ions by multiplying ± the pre-assigned confidence level. For example, a relative confidence limit of 15% for ion 149 with a relative abundance of 45%, the confidence interval would be 38% [45×(100-15)/100] to 52% [45×(100+15)/100].

Confirmation: Verification of a previous analyte identification that is performed by another analytical system.

Deconvolution: Process to extract clean spectra from a complex mixture of overlapping peaks using mathematical algorithms.

Diagnostic ion(s): Ion(s) used to identify and quantitate the target compound. Diagnostic ions include the molecular ion, characteristic adduct ions, characteristic fragment ions (structurally significant ions), and isotope ions.

Electron ionization (EI): Ionization process initiated by the interaction of the gas-phase molecule with an energetic electron to create a charged ionic form of the molecule. Electron ionization is sometimes called electron impact.

Electrospray ionization (ESI): Ionization process where a sample solution is pumped into a capillary which is held at high potential causing a reaction between a reagent ion and a neutral molecule to create a charged ionic form of the molecule. The solution emerges from the capillary as a mist which is sprayed at atmospheric pressure into the mass spectrometer.

Fragment ion(s): Ion(s) formed when the precursor or product ion fractures after undergoing CID. All fragment ion(s) are product ion(s), but not all product ion(s) are fragment ion(s)

Full scan: The practice of monitoring and recording a wide range of ion mass-to-charge ratios (m/z) produced following sample ionization.

Ion trap: Type of mass analyzer consisting of two end caps and a ring electrode forming a three-dimensional quadrupole that stores ions at its center. An additional electrical signal is used to selectively eject ions to an external detector.

Ionspray™ ionization: Pneumatically assisted ESI. Ionspray ionization is also called turbospray ionization.

Internal standard: A substance not contained in the test sample with physical and chemical properties as similar as possible to those of the target analyte to be identified. An isotope-labeled form of the target analyte can also serve as an internal standard. The internal standard is added to each test sample as well as to each calibration standard at the beginning of the analytical process and used in the quantitative determination of the target analyte by taking into account the recovery of the internal standard.

Matrix-assisted laser desorption ionization (MALDI): Ionization process where sample molecules are mixed with an excess of energy-absorbing matrix. The subsequent mixture is co-

crystallized in a thin film on an inert support. Repetitive irradiation with a pulsed laser releases ions from the surface.

Molecular ion: An ion formed by the removal or addition of one or more electrons to a molecule without fragmentation; the peak representing the ionized molecule that contains only the isotopes of greatest natural abundance.

Mass spectrometry (MS): Analytical technique used to identify compounds based on their chemical structures' fragmentation patterns. MS instruments are called mass spectrometers.

Mass spectrometry/mass spectrometry (MS/MS): A form of mass spectrometry whereby ions are separated according to their mass-to-charge ratio in the first stage and are then fragmented by collisionally-induced dissociation, and the resultant fragment ions separated and measured in the second stage. MS/MS is also referred to as tandem mass spectrometry.

MS^n: MS/MS reactions recurring over multiple steps.

MS spectrum: Graphical representation of ion intensity vs. m/z data at a single point in time.

MS/MS spectrum: Graphical representation of ion intensity vs. m/z data at a single point in time produced by an isolated mass undergoing CID.

Multiple reaction monitoring: Selected reaction monitoring for more than one precursor-to-product ion transition.

m/z: A ratio of mass-to-charge.

Precursor ion: An abundant, structurally significant ion selected from the full scan spectrum to be isolated and subsequently subjected to CID. A precursor ion may be a molecular ion or a fragment ion. The precursor ion is sometimes called the parent ion.

Precursor ion scan: The practice of using the second stage mass analyzer in an MS/MS experiment to select a specific product ion and then using the first stage mass analyzer to scan for the precursor ion(s). The term parent ion scan is also used.

Product ion(s): Ion(s) formed from the reaction of the precursor ion. The reaction need not involve fragmentation through CID (e.g., the reaction involves a change in the number of charges carried by the precursor ion). If the reaction does involve CID, the product ion is also a fragment ion. Product ion(s) are sometimes called daughter ion(s).

Product ion scan: The practice of using the first stage mass analyzer in an MS/MS experiment to select a specific precursor ion and then using the second stage mass analyzer to scan for the resulting product ions. The term daughter ion scan is also used.

Quadrupole: Type of mass analyzer consisting of four parallel rods arranged in a square array. Radio frequency and direct current voltages are applied to the rods creating a hyperbolic field that filters ions based on their mass-to-charge ratio.

Qualifier ion(s): Structurally significant ion(s) chosen from the reference spectrum to show consistent relative abundances when compared to the target ion. Qualifier ion(s) are sometimes called secondary ion(s).

Quantitation ion: A structurally significant ion that demonstrates a linear response over a broad range of concentrations. It is typically the target ion.

Reconstructed ion chromatogram: A plot of the intensity of specific ions in a MS or MS/MS spectrum (based on m/z) versus time.

Reference spectrum: Graphical representation of ion intensity vs. m/z data at a single point in time.

Relative abundance: The abundance of an ion relative to that of the most abundant ion, or base peak, in the spectrum.

Selected ion monitoring (SIM): Data acquisition technique of monitoring and recording one or more ion mass-to-charge ratios (m/z) rather than monitoring and recording the full MS spectra (i.e., a wide range of m/z values). This technique can greatly improve instrument sensitivity, albeit at a cost of reduced specificity. The term single ion monitoring is sometimes used.

Selected reaction monitoring (SRM): The MS/MS techniques of monitoring and recording one or more precursor-to-product ion transitions rather than monitoring and recording the full MS/MS spectra (i.e., all precursor or product ions). This practice can serve to greatly increase signal-to-noise by reducing noise.

"Soft" ionization: Low energy ionization process that typically results in little or no molecule fragmentation. The ions are usually either protonated $(M+H)^+$ or deprotonated $(M-H)^-$. Soft ionization processes include (but are not limited to) CI, ESI, APCI, and APPI.

Structurally significant ion: Ion with a mass-to-charge ratio (m/z) which indicates a characteristic structural grouping formed by the fragmentation of a molecule.

Target ion: A structurally significant ion selected from the reference spectrum, typically the most abundant ion, to be used to generate relative abundance ratios with qualifier ions. The target ion is sometimes called the primary ion.

Time-of-flight (TOF) mass analyzer: Type of mass analyzer that uses the flight time of an ion over a fixed distance to measure its mass. Lower mass ions will move through fixed distance faster than higher mass ions.

Total ion current: A plot of the summed intensity of all acquired ions in a MS or MS/MS spectrum versus time. The term total ion chromatogram is also used.

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

SOP NO.: PDP-ADMIN		Page 1 of 35
Title: Administrative Procedures for the Pesticide Data Program		
Revision: 7	Replaces: 05/01/2017	Effective: 07/01/2019

1. Purpose:

To standardize administrative procedures for sampling and testing activities of the US Department of Agriculture (USDA) Pesticide Data Program (PDP).

2. Scope:

This standard operating procedure (SOP) shall be followed by the USDA Monitoring Programs Division (MPD) and all facilities involved in the collection of samples and performance of analytical determinations for PDP, including support laboratories conducting non-routine activities that may impact the program. This SOP does not supersede any requirements specified in the Cooperative Agreement between USDA and the participant.

3. Outline of Procedure:

4. References:

- U.S. Environmental Protection Agency (EPA), *Inspection of a Testing Facility*, 40 CFR part 160.15
- U.S. EPA, *Personnel*, 40 CFR part 160.29
- U.S. EPA, *Testing Facility Management*, 40 CFR part 160.31
- U.S. EPA, *Study Director*, 40 CFR part 160.33
- U.S. EPA, *Quality Assurance Unit*, 40 CFR part 160.35
- U.S. EPA, *Facilities for Handling Test, Control, and Reference Substances*, 40 CFR part 160.47
- U.S. EPA, *Laboratory Operation Areas*, U.S. EPA, 40 CFR part 160.49
- U.S. EPA, *Specimen and Data Storage Facilities*, 40 CFR part 160.51
- U.S. EPA, *Equipment Design*, 40 CFR, part 160.61
- U.S. EPA, *Standard Operating Procedures*, 40 CFR part 160.81
- USDA, *Uniform Administrative Requirements for Grants and Cooperative Agreements to State and Local Governments*, 7 CFR, part 3016
- USDA, *Equipment Management Requirements*, 7 CFR, part 3015.169
- U.S. EPA, *Determining Compliance of Audited Studies with GLP Standards Requirements*, SOP GLP-02
- U.S. EPA, *Preparation of Standard Operation Procedures*, SOP GLP-S-01
- Garfield, F.M., Klesta, E., Hirsch, J., *Quality Assurance Principles for Analytical Laboratories*, pg. 9, 1991
- Taylor, J.K., *Quality Assurance of Chemical Measurements*, pp. 85, 90, 113, 114, 173, 210, 223, 236, 261, and 262, 1989
- US Department of Health and Human Services, Center for Disease Control and Prevention (CDC), and National Institute of Health (NIH), *Biosafety in Microbiological and Biomedical Laboratories*, 5th ed., US GPO, 2007
- U.S. EPA, *Good Laboratory Practices for Commodity Laboratory Analyses, 7* CFR Subchapter E, Subpart C, Section 90.3.
- U.S. EPA, *Storage and retrieval of records and data,* 40 CFR 160.190
- U.S. National Archives and Records Administration (NARA, *Unscheduled Records FAQS,* http://www.archives.gov/frc/unscheduled-records-faqs.html

- U.S. National Archives and Records Administration (NARA, *Records Transmittal and Receipt, SF-135, instructions*, http://www.archives.gov/frc/forms/sf-135-intro.html
- AOAC International, *Guidelines for Laboratories performing Microbiological and Chemical Analyses of Food and Pharmaceuticals, An Aid to Interpretation of ISO/IEC 17025:2005 (Rev March 2010), Section: General requirements for the competence of testing and calibration laboratories.*

5. Facilities

5.1 Facilities for Handling Test, Control, and Reference Substances

Adequate space shall be provided for conducting sampling and analytical laboratory work performed for PDP. Space shall be as needed to prevent contamination or mix-ups of samples, reference materials, and other work in place in the facility.

5.2 Specimen and Data Storage Facilities

5.2.1 Adequate space shall be provided for the storage and retrieval of all samples, for raw data including archived data and for the analysis of samples to ensure integrity and prevent the possibility of contamination and cross-contamination. Access to this space shall be limited to authorized personnel.

5.2.2 Each participating laboratory shall maintain a site-specific record system to suit its particular circumstances, which assures orderly storage and expedient retrieval of data and other records.

5.2.3 Physical and environmental conditions of storage shall minimize deterioration of the documents in accordance with the requirements for the time period of their retention and the nature of the documents.

5.2.4 Where computers or automated equipment are used for the storage or retrieval of data, the laboratory shall ensure that:

- Computer software is documented, adequate for use and is run periodically to verify correct operation. Computer and automated equipment is maintained to ensure proper

functioning and provided with the environmental and operating conditions necessary to maintain data integrity; and

- Appropriate procedures are established and implemented for protecting the integrity of data (such procedures shall include but not be limited to integrity of data entry or capture and data storage) and for the maintenance of security of data including the prevention of unauthorized access or amendment of electronic records.

5.3 Inspection of Facilities

5.3.1 A sampling or laboratory facility shall permit an authorized employee or duly designated representative of USDA/Agricultural Marketing Services (AMS), at reasonable times and in a reasonable manner, to inspect the facility and to inspect (and in the areas of records to copy) all records and samples required to be maintained regarding PDP operations.

5.3.2 USDA/AMS shall communicate any serious deficiencies identified during the facility inspection in a memo format within 10 days. Additionally, USDA/AMS shall provide a draft, written report for the sampling or laboratory facility's comments. A final report incorporating any comments received shall be issued within 60 days of the last day of the review.

5.3.3 When the review results in adverse findings, the sampling or laboratory facility shall provide a written response to the USDA/AMS report, outlining plans to correct any adverse findings within 60 days of receipt of the report.

5.4 Data and Records Retention Periods

5.4.1 Monitoring Programs Division (10 years)

- General information relating to USDA/AMS PDP correspondence,
- SOPs,
- protocols,
- semi-annual program plans,
- annual and/or semi-annual Federal/State meeting minutes and/or presentations,
- sampling plans,

- sampling site information,
- semi-annual State Meeting minutes,
- interim and final reports,
- data interpretations, and
- other significant program-unique information.

5.4.2 States and Laboratories

5.4.2.1 25 years

- PDP sample data packages
- PDP method validation data packages
- PDP proficiency testing data packages

5.4.2.2 5 years

Supporting data generated by PDP Federal/State laboratories including, but not limited to:

- historical internal SOPs and work instruction documents,
- logbooks (e.g. standard preparation, instrument, freezer, temperature, etc.),
- chromatograms generated during standards checking,
- sample worksheets (e.g., homogenization, extraction, etc.),
- correspondences and other documents relating to interpretation and evaluation of data,
- corrective actions,
- deviation letters,
- method development studies other than official PDP method validation packages,
- control charts, etc.

5.4.2.3 2 years

Supporting data and records for PDP sampling including, but not limited to:

- historical internal SOPs,
- sampling plans,
- site information,

- commodity payment records,
- surplus commodity disposition records,
- raw Sample Information Form data sheets, etc.

5.4.3 Data Transfer

5.4.3.1 The minimum on-site retention for items in 5.4.2 above is 2 years after which they may be transferred to a designated Federal Records Center (FRC) per section 5.5 below or, if longer retention is not stipulated, destroyed according to applicable internal records destruction procedures.

5.4.3.2 USDA/AMS shall be contacted if a laboratory wishes to transfer records within a timeframe shorter than 2 years.

5.5 Records Archival Procedure

5.5.1 Data Archival at the Participating Laboratory

5.5.1.1 An individual(s) shall be identified as the archivist for the laboratory.

5.5.1.2 Access to archived records shall be monitored and controlled. Use of manual or electronic logs is recommended.

5.5.1.3 Physical and environmental conditions of storage shall minimize deterioration of the documents in accordance with the requirements for the time period of retention and the nature of the documents. Locked file cabinets, temperature-controlled and/or secured records storage facilities, etc. are acceptable.

5.5.1.4 Each data package retained shall be filed by calendar year and month.

5.5.2 Transferring Records to the Federal Records Centers

5.5.2.1 Dispose of all extra copies of records, non-record material (e.g., buckslips, post-it notes, etc.), and metal items (e.g., paperclips, binder clips, etc.) in accordance with individual laboratory security policies. The use of accordion folders is

suggested. Binders with non-metal parts (e.g., plastic combs/spirals, 3-ring "Tuffy" mechanisms, etc.) are also acceptable.

5.5.2.2 Sample data packages representing a single calendar year must be transferred separately from other calendar years (i.e., utilizing a different transfer number). Within each calendar year, the data packages shall be filed by month and commodity. Supporting documentation must be archived separately by time span and subject (e.g., 2007-2009 Temperature Logs, 2006-2009 Administrative Documents, etc.) at the discretion of the laboratory.

5.5.2.3 PDP method validation sets and proficiency testing sample sets may be transferred concurrently with sample data packets from the same calendar year or they may be transferred separately at a later date. If transferred separately, method validation and proficiency testing sets may be archived together as long as they are within a three year time span in a single box.

5.5.2.4 All transfers must be requested electronically using the SF-135 fillable form (*Attachment 2*, Standard Form SF-135, Fillable Template) with a copy of the box listing through the USDA/ AMS NARA liaison.

5.5.2.5 Refer to SF-135 Records Transmittal and Receipt (*Attachment 2)* for form template. Example of the required information on the SF-135 form for various records are provided in Attachment 3. Example for documents included in box listing are provided in Attachment 4.
Note: An Adobe Acrobat fillable form SF-135 is available on the internet at Federal Records Centers — Records Retrieval Services, Records Transmittal and Receipt, SF-135 (http://www.archives.gov/frc/forms/sf-135-intro.html).

5.5.2.6 Use only FRC boxes when transferring records. Boxes may be obtained by contacting USDA/AMS. Refer to *Attachment 5* for illustrated box assembly and packing instructions.

5.5.2.7 When packing records, do not force files into the boxes. Leave approximately one inch of space in each box to permit easy withdrawal of folders. Pack folders upright, with letter size folders facing the front of the container. Do not place folders one on top of another.

5.5.2.8 Number the cartons sequentially (e.g., 1/10, 2/10, 3/10, etc.) with permanent black marker in the upper right front corner. The box numbers shall correspond to the completed SF-135.

5.5.2.9 USDA/AMS will submit the SF-135 to the FRC for approval and assignment of the transfer number. Once the transfer number is received by USDA/ AMS, a hard copy of the SF-135 will be generated and sent back to the transferring laboratory. Upon receipt of the approved SF-135, the transfer number shall be placed in the upper left front of the carton. All transfers must be forwarded to the FRC within 90 days of the assignment of a transfer number. If the FRC does not receive the records during the allotted time period, the transfer number becomes null and void. Include the date of disposal on the approved SF-135 on the outside of each box.

5.5.2.10 Place the approved SF-135 and box listing inside the first box of the transfer.

5.5.2.11 Close all boxes and seal with filament tape. Ensure that the filament tape does not cover the transfer number or the carton number.

5.5.2.12 Ship all boxes to the appropriate designated FRC using the most economical and secure carrier (e.g., Certified US Mail 3rd Class or equivalent). All expenses incurred in transferring records must be charged to the laboratory's PDP allocated funds. The records will be retained by the FRC and will be available for retrieval during the specified storage time through the USDA/AMS NARA liaison.

6. Personnel and Organization

6.1 Personnel Requirements

Employee Qualifications

Each individual responsible for the supervision of or engaged in the conduct of the sample collection process or laboratory analyses for PDP shall have the education, training, and experience, or combination thereof, to enable that individual to perform the assigned functions.

Note: The term "each individual" includes temporary and part-time workers as well as aides and others who participate in PDP-related activities.

6.1.2 Employee Records shall be kept current and shall include:

- Information to support that the individual meets at least the minimum standards for the position which they hold.
- Information pertaining to training, competency, and authorization to perform activities. The records for laboratory personnel shall reflect whether an analyst's proficiency is individual or as part of a team.
- Publications and articles authored as well as participation in professional societies should be included in the records.

Note: Each participating State/facility stipulates the specific information required (e.g., resumes, CV, employment applications, job descriptions, etc.).

6.1.3 Technical Personnel Performance Evaluation

Each laboratory shall document the procedures for individual performance evaluation in an internal SOP. Suggestions for performance evaluation include:

- Proficiency Test (PT) results
- Control charting of process controls and fortification spikes. Acceptance criteria for recoveries and coefficient of variation are outlined in PDP-QC.
- Internal blind check samples prepared by the QAU and fortified with PDP pesticides varying between 1xLOQ and 10xLOQ. Acceptance criteria for recoveries and coefficient of variation are outlined in PDP-QC.

6.2 USDA/AMS Responsibilities

6.2.1 USDA/AMS has named the MPD Director as the PDP Program Administrative Manager and the PDP Technical Program Manager in charge of administrative and technical affairs. See the appropriate section of this SOP.

6.2.2 Technical program reports shall be made to the MPD Director at USDA/AMS, S&T, MPD, 1400 Independence Avenue, SW, Room 0601, Washington, DC 20250, (telephone (202) 572-8167 or FAX (202) 619-1724)

6.2.3 USDA/AMS management shall:

- Replace the MPD Director promptly if it becomes necessary to do so during the conduct of the PDP studies.
- Ensure that personnel, resources, facilities, equipment, materials, and methodologies are available as scheduled.
- Ensure that personnel clearly understand the functions they are to perform.
- Ensure any PDP-related records (e.g., sampling, laboratory, equipment, financial, etc.) are available for inspection by authorized employees or duly designated representatives of USDA/AMS.

6.3 MPD Director

USDA/AMS shall identify a scientist or other professional of appropriate education, training, and experience, or combination thereof, as the MPD Director for PDP. The MPD Director has the overall administrative responsibility for program expansion, budgeting, cooperative agreements, memoranda of understanding, and major disbursement of funds. The MPD Director also has overall responsibility for the sampling and technical conduct of the PDP studies. The MPD Director, through their own efforts or through the work assignments of PDP staff, shall ensure:

6.3.1 The Deputy Administrator for USDA/AMS, Science and Technology, is kept informed on PDP financial, administrative affairs.

6.3.2 Annual budgets for the administration of PDP at the national level are prepared and submitted.

6.3.3 Work contracts are negotiated in cooperation with the States and/or other Federal agencies.

6.3.4 The States' and/or Federal facilities' use of Federal funds is monitored through appropriate documentation.

6.3.5 MPD serves as liaison to CDC, EPA, U.S. Food and Drug Administration (FDA), and other USDA agencies participating in PDP.

6.3.6 The quality assurance of sampling, technical, and database operations are monitored to assure management that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with USDA/AMS program plans and SOPs.

6.3.7 PDP data are reported in an annual program summary. This includes the interpretation, analysis, documentation, and reporting of results.

6.3.8 The program plans and PDP SOPs, including any changes, are approved and followed.

6.3.9 All sampling information and experimental data are accurately recorded and verified.

6.3.10 Unforeseen circumstances that may affect the quality and integrity of PDP samples and/or studies are documented as they occur, and corrective actions are taken and documented, as necessary.

6.3.11 PDP sampling procedures and test systems are as specified in the program plans and SOPs. This shall be accomplished through conference calls, reviews, and frequent communications with participants.

6.3.12 Reviews of participant sampling and laboratory facilities are performed at intervals adequate to ensure the integrity of PDP samples and analytical results and written records of each review are maintained. The frequency of reviews for a particular participant shall be based on two factors:

- Time elapsed since the last review; and/or
- Designated need due to problems associated with the collection or analysis of samples performed by that participant. Participant Administrative Managers shall be notified and final arrangements shall be made at least two weeks in advance of the review, if at all possible.

For sampling reviews, the review report is distributed to:

- The participant's Administrative Manager, supervisor of the Sampling Manager, and Sampling Manager; and
- The USDA/AMS MPD Director

For laboratory reviews, the review report is distributed to:

- The participant's Administrative Manager, Technical Program Manager (TPM), and Quality Assurance Officer (QAO); and
- The USDA/AMS MPD Director

6.3.13 All raw data and supporting laboratory records are stored, retained, and transferred to the archives during or at the close of PDP.

6.4 Responsibilities of Participants

6.4.1 Each participant State/facility shall designate an Administrative Manager. Each sample collection participant shall designate a Sampling Manager. Each laboratory participant shall designate a TPM and a QAO. See the appropriate sections of this SOP.

6.4.2 The participant management shall:

6.4.2.1 Replace the Administrative Manager, Sampling Manager, QAO, or the TPM promptly if it becomes necessary to do so during the conduct of the PDP testing.

6.4.2.2 Ensure that there is a Quality Assurance Unit (QAU) as described in this SOP.

6. 4.2.3 Ensure that personnel, resources, facilities, equipment, materials, and methodologies are available as scheduled.

6.4.2.4 Ensure that personnel clearly understand the functions they are to perform.

6.4.2.5 Ensure that laboratory activities are conducted in compliance with applicable Federal, State, and local safety and waste disposal codes/requirements. Laboratories shall also comply with applicable Chemical Hygiene Plan (CHP), biosafety manual, Injury and Illness Prevention Programs, Employee Right-To-Know

Programs, etc., and have Material Safety Data Sheets (MSDS) and/or Safety Data Sheets (SDS) available to all applicable personnel.

6.4.2.6 Ensure that any unauthorized deviations from the PDP SOPs, program policies, and approved analytical methodologies as reported by the QAU are communicated to the USDA/AMS MPD Director and laboratory liaison and that corrective actions are taken and documented.

6.4.2.7 Ensure an accurate and timely inventory of supplies and equipment purchased or utilized for PDP is maintained. See section 7.2 and Attachment 7.

6.4.2.8 Ensure any PDP-related records (e.g., sampling, laboratory, equipment, financial, personnel, etc.) are available for inspection per section 5.3 by authorized employees or duly designated representatives of USDA/AMS.

6.4.2.9 Provide the name and position for all administrative, sampling, and technical personnel associated with PDP-related activities annually, at the beginning of the Federal fiscal year (October 1). An update shall be submitted to USDA/AMS within 30 days of any staff changes that may affect sample collection and/or data delivery.

6.4.2.10 Inform USDA/AMS of any critical personnel vacancies, staffing issues, expected increases in rent (due to laboratory or office renovation/relocation, etc.), sampling issues, and technical issues.

6.5 State/Facility Administrative Manager

Each participating agency shall identify a scientist or other professional of appropriate education, training, and experience, or combination thereof, as the Administrative Manager for PDP. The Administrative Manager has overall administrative responsibility for their organization's participation in PDP. This would include but not be limited to PDP activities such as: sampling operations, laboratory management, budgeting, contracting, purchasing, inventory maintenance, and receipt of QA reports and associated corrective actions. The State/facility Administrative Manager shall:

6.5.1 Prepare and maintain annual budgets for PDP contract administration. For States/Facilities where budget functions are managed by person(s) other than the assigned Administrative Manager, a description of how laboratory costs are calculated (number of FTEs including salary and benefits, supplies, rent, utilities, etc.) shall be provided to the MPD Director when requesting funding to cover program operations.

6. 5.2 In cooperation with USDA/AMS, prepare and negotiate work contracts for PDP.

6.5.3 Maintain appropriate accounting records to document the State/facility use of Federal contract funding.

6.5.4 Maintain appropriate performance records to document State/facility performance and productivity on PDP studies (e.g., records of samples analyzed).

6.6 State Sampling Manager

Each sample collection participant shall identify a professional of appropriate education, training, and experience, or combination thereof, as the Sampling Manager for PDP. The Sampling Manager is responsible for the conduct of the participant's sampling procedures. The Sampling Manager shall ensure that:

6.6.1 The PDP program plan and USDA/AMS Sampling SOPs, including any changes, are followed. Any problems regarding compliance with the program plan or Sampling SOPs shall be communicated immediately to the MPD Director or designee.

6.6.2 The participant sampling plan and internal sampling SOPs, including any changes, are followed. Participant internal sampling SOPs document specific procedures utilized by the State in collecting and shipping PDP samples. These SOPs are intended to augment the USDA/AMS SOPs, by providing State-specific instructions.

6.6.3 All required sampling information is accurately recorded and verified, including unforeseen circumstances that may affect the quality and integrity of PDP samples and when corrective actions were taken and documented, as necessary.

6.6.4 Internal reviews of the procedures utilized by the sample collectors are performed at intervals adequate to ensure the integrity of PDP samples. The timeframe for

performing internal reviews shall vary among participants based on the number of collectors to be reviewed. Each collector should be reviewed once before repeating the process. An exception would be if a number of problems are determined to be the result of a particular collector's negligence or failure to comply with the program SOPs.

6.6.5 Records of each review are maintained. Each review report shall show:

- The date of the review;
- The name(s) and title(s) of the person(s) performing the review; and
- Observations, findings and problems, recommendations and suggested corrective actions.

6.6.6 Group/individual training sessions are held periodically for the sample collectors. This is especially important if there are major program changes, or a number of sampling problems have been reported by either the MPD Director or the applicable analytical laboratory(ies).

6.6.7 Any other documents required in the PDP Sampling SOPs shall be kept on file and updated as necessary (e.g., master site lists, FTE information, volume weighting information for collection sites, donation receipts, etc.).

6.6.8 All PDP supporting records for sampling activities are stored, retained, and transferred to the archives as specified in Sections 5.4 and 5.5.

6.7 State/Facility Technical Program Manager (TPM)

Each participating laboratory shall identify a scientist or other professional of appropriate education, training, and experience, or combination thereof, as the TPM for PDP. The TPM has overall responsibility for the technical conduct of the PDP testing contracted to the laboratory, as well as for the interpretation, analysis, documentation, and reporting of results. The TPM shall ensure that:

6.7.1 The PDP program plan and all USDA/AMS SOPs, including any changes, are followed. Any problems regarding compliance with the program plan or SOPs shall be communicated immediately to the MPD Director or designee.

6.7.2 The laboratory plan, internal SOPs, and analytical methodologies, including any approved changes and/or deviations, are followed.

6.7.3 All experimental data are accurately recorded and verified, including unforeseen circumstances that may affect the quality and integrity of the PDP testing, and corrective actions, if any, are documented.

6.7.4 All PDP test systems are as specified in the plan, SOPs, or analytical methods, including any approved changes and/or deviations.

6.7.5 When requested, project status reports (e.g., progress on validation studies) are prepared.

6.7.6 All required data is accurately transmitted electronically to USDA/AMS via Remote Data Entry (RDE).

6.7.7 All PDP raw data and supporting laboratory records are stored, retained, and transferred to the archives as specified in Sections 5.4 and 5.5.

6.8 State/Facility QAU

Each PDP participating laboratory shall have a QAU consisting of one or more personnel of suitable qualifications. For those participants where there are two or more field facilities under a common administration there only needs to be a single QAU. Each PDP participating laboratory shall appoint an individual within the QAU to serve as the QAO.

6.8.1 QAU Independence

The QAU shall be entirely separate from and independent of the personnel engaged in the technical direction and/or conduct of sample analyses. The QAU shall report to non-technically involved laboratory management such as the laboratory director or the Administrative Manager. The TPM is considered to be involved in the technical direction and conduct of the residue studies and therefore may not direct the QAU.

6.8.2 Data Review and Transmission

The QAU shall review all data packages as one of the final steps prior to submission to USDA/AMS. The QAU review shall be documented. See PDP DATA SOPs for guidelines. After the QAU review of a data package, data may not be changed by any laboratory personnel unless as a response to comments/concerns/recommendations by the QAU.

6.8.3 Internal Audits

The QAU shall perform audits of the laboratory operations at intervals adequate to ensure the integrity of PDP sample analyses and to evaluate the compliance of laboratory facilities, equipment, personnel, methods, practices, records, and controls are in conformance with the plans and SOPs issued by USDA/AMS and by the laboratory. Each segment or phase of PDP laboratory operations shall be audited at least every two years. Audit records shall include the dates the audits were performed, the audit findings, and reference to any corrective actions initiated.

6.8.4 Proficiency Testing (PT)

The QAU shall notify the MPD Director and assigned laboratory liaison of any corrective actions initiated in response to a PT result, and the resolution of each corrective action.

6.8.5 Reports

The QAU shall prepare and submit to USDA/AMS semi-annual updates based on calendar year (i.e., January through June and July through December) summarizing QA issues. Updates shall be submitted within 30 days after the completion of the reporting period and should include the status of the following:

- Progress on Method Validations
- Corrective Action Summary
- Laboratory SOPs, New and Revised, titles and status specified
- Internal Audit Summary, including dates, areas audited, corrective actions, and unresolved issues
- Internal PT Sample Results, where applicable

- PT Sample Summary
- Changes to Methodology
- Miscellaneous QA Issues
- Status of two times the Limit of Quantitation (2x LOQ) quarterly spike results for all reported compounds (refer to PDP-QC).

7. Purchase, Inventory, and Salvage Procedures

7.1 Purchases

All purchases must be made within the confines of the current year's Cooperative Agreement terms. Details regarding purchase and reimbursement may be found in the agreement.

The requirements below are for routine planned purchases. In emergency cases that may impact production, the MPD Director should be contacted immediately.

7.1.1 Equipment Purchases

7.1.1.1 Equipment used in the generation, measurement, or assessment of data for PDP and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to PDP protocols and SOPs. Equipment shall be suitably located for operation, inspection, cleaning, and maintenance. Equipment is defined as nonexpendable, tangible personal property with a unit cost of $5,000 or more.

7.1.1.2 Equipment purchases costing $5,000 or more using USDA funds, including split-funded purchases, require USDA/AMS authorization. The laboratory shall contact the assigned laboratory liaison to discuss purchase plans. If the laboratory liaison is unavailable, the MPD Director may be contacted instead. Upon concurrence of the purchase, the laboratory will then obtain formal cost estimates and complete a GSA-49 Requisition/Procurement Request (*see Attachment 6*). The GSA-49, along with estimates, will be emailed to the assigned laboratory liaison.. The laboratory liaison will obtain the MPD Director's signature on the GSA-49 and it will be returned to the laboratory. Upon receipt of signed GSA-49, the laboratory may then proceed with the necessary steps to complete the purchase. ***Purchases are not authorized to occur until the signed GSA-49 is in hand.***

7.1.1.3 Equipment purchases costing less than $5,000 and required to conform to PDP SOPs do not require prior authorization if they meet the "prudent person" rule. For an expense/cost/need to be reasonable, the total cost may not be more than a "prudent person" would spend under the circumstances prevailing at the time the decision was made to incur the cost. If this rule cannot be met or the decision could be questionable, refer to the steps above for obtaining prior approval.

7.1.1.4 Upon receipt, installation, and training (if included in the purchase) of equipment requiring approval, the laboratory shall send an email notification to USDA/AMS stating that the equipment is installed and operational and enter the equipment into the PDP Equipment Inventory Database (see Section 7.2). After all of these steps have occurred, the equipment purchase may then be reimbursed via SF 270 but not before.

7.1.1.5 The equipment shall vest with the State Agency upon acquisition. The equipment shall be tagged as State inventory; however, documentation shall be maintained citing the equipment as purchased with Federal funds. USDA/AMS retains the right to transfer said equipment for use by another State Agency or Federal facility performing PDP analyses during the course of the residue studies; however, the equipment remains tagged as the property of the originating State Agency or facility. Upon termination of the program, equipment will become the property of the originating State Agency.

7.1.2 Supply Purchases

7.1.2.1 Supplies are generally defined as an item with an acquisition cost of $5,000 or less and a useful life expectancy of less than one year. Supplies are generally consumed during the project performance. Supply items must be direct costs to the project and meet the "prudent person" rule. For an expense/cost/need to be reasonable, the total cost may not be more than a "prudent person" would spend under the circumstances prevailing at the time the decision was made to incur the cost. If this rule cannot be met or the decision could be questionable, refer to the steps below for obtaining prior approval.

7.1.2.2 Supplies costing more than $5,000 per item or $10,000 for multiples of the

same item in one purchase require USDA/AMS authorization. The laboratory shall contact the assigned laboratory liaison to discuss purchase plans. If the laboratory liaison is unavailable, the MPD Director may be contacted instead. Upon concurrence of the purchase, the laboratory will then obtain formal cost estimates and complete a GSA-49 Requisition/Procurement Request (*see Attachment 6*). The GSA-49, along with estimates, will be emailed to the assigned laboratory liaison.. The laboratory liaison will obtain the MPD Director's signature on the GSA-49 and it will be returned to the laboratory. Upon receipt of signed GSA-49, the laboratory may then proceed with the necessary steps to complete the purchase. ***Purchases are not authorized to occur until the signed GSA-49 is in hand.***

7.1.2.3 Supply purchases costing less than $5,000 and required to conform to PDP SOPs do not require prior authorization if they meet the "prudent person" rule as defined in 7.1.2.1 above. If this rule cannot be met or the decision could be questionable, refer to the steps above for obtaining prior approval.

7.1.3 Non-Equipment or Supply Expenses

7.1.3.1 Examples include maintenance agreements for laboratory equipment, repairs, renovations, vehicles, employee development, all training, conferences, meetings, seminars, accreditation fees/charges, consultants, etc. This list is not all-inclusive. Contact your laboratory liaison and/or MPD Director if an expense could be questionable.

7.1.3.2 Non-equipment/supply expenditures require USDA/AMS authorization regardless of cost. The laboratory shall contact the assigned laboratory liaison to discuss purchase plans. If the laboratory liaison is unavailable, the MPD Director may be contacted instead. Upon concurrence of the purchase, the laboratory will then obtain formal cost estimates and complete a GSA-49 Requisition/Procurement Request (*see Attachment 6*). The GSA-49, along with estimates, will be emailed to the assigned laboratory liaison. The laboratory liaison will obtain the MPD Director's signature on the GSA-49 and it will be returned to the laboratory. Upon receipt of signed GSA-49, the laboratory may then proceed with the necessary steps to complete the purchase. ***Expenditures are not authorized to occur until the signed GSA-49 is in hand.***

7.1.3.3 Travel that is part of the employees day-to-day routine duties does not require USDA/AMS authorization (e.g., travel to and between collection sites by Samplers).

7.1.3.4 A synopsis of topics covered, benefits, received, etc. shall be provided to the assigned laboratory liaison and/or MPD Director when attending or presenting at a meeting, seminar, or training.

7.1.4 Memberships

Individual memberships may not be expensed to PDP, as these are considered personal in nature.

7.2 Equipment Inventory

7.2.1 The laboratory shall maintain up-to-date property records for any piece of equipment (defined in 7.1.1.2) purchased with PDP funds, including split-funded purchases.

7.2.2 A physical inventory of property shall be taken and the results reconciled with the PDP Equipment Inventory database at least once per year. After reconciling the individual State spreadsheet in the PDP database, include the date and name of the person that performed the reconciliation at the top of the spreadsheet. The PDP Equipment Inventory Database is located on the USDA/AMS Extranet (*see requirements in Attachment 7*).

7.3 Permission to Salvage, Dispose of Equipment, or Trade In

7.3.1 For equipment purchased by PDP or using PDP Cooperative Agreement funds and that is no longer in working condition or is technically outdated, the laboratory must complete form AD-112, Report of Unserviceable, Lost, Stolen, Damaged or Destroyed Property (*see Attachment 8*), by dating and completing Section 1, numbers 1-4, and email to the assigned laboratory liaison. The AD-112 will be submitted to the MPD Director requesting permission to salvage, or dispose of the equipment. If approved, USDA/AMS will return the signed AD-112 authorizing disposal. The laboratory shall use their internal equipment salvage/disposal procedures to dispose of the property.

7.3.2 For equipment purchased by PDP or using PDP Cooperative Agreement funds and that is in working condition, but no longer being used, the laboratory will notify USDA/AMS. If the MPD Director authorizes the donation of the property, the laboratory must complete Form AD-107, Report of Transfer or Other Disposition or Construction of Property (*see Attachment 9*), by dating and completing Sections 1, 3-5 (if applicable), 4.a (with the laboratory name as the organizational unit) and 6 and submit it to the MPD Director. Expenses related to the Transfer of said property will be incurred by the recipient.

7.3.3 For equipment purchased by PDP or using PDP Cooperative Agreement funds that is being traded in, the laboratory must complete Form AD-107, Report of Transfer or Other Disposition or Construction of Property, and submit it to the MPD Director (see *Attachment 9).*

7.3.4 The laboratory must inform the laboratory liaison and MPD Director, in writing, of any changes regarding the disposition of equipment and the inventory list must be updated within 30 days.

7.4 Forms Instructions

7.4.1 The PDP Cooperative Agreement Number should be used for the GSA-49 Box 12, Contract # field.

7.4.2 Required fields are highlighted on the fillable versions of GSA-49, AD-107, and AD-112 that are posted on the USDA/AMS Extranet.

8. PDP Quality Assurance Program

8.1 Overview

8.1.1 The MPD Director shall ensure that a quality assurance (QA) program is in place to monitor overall QA for sampling, technical, and database functions. The MPD Director shall have overall responsibility for assuring management that facilities, equipment, personnel, methods, practices, records, and controls of the program are in conformance with the plans and SOPs issued by USDA/AMS.

8.1.2 The MPD Director shall appoint an individual to serve as the PDP QA Coordinator. The QA coordinator shall be responsible for selected SOPs as detailed below and shall serve as the focal point for selected documents, reports, and correspondence pertaining to program quality control (QC) and/or QA issues.

8.1.3 Additional, specific QA functions shall be assigned by the MPD Director to appropriate sampling, technical, and database staff.

8.1.4 Appropriate PDP records shall be maintained by assigned staff. Documents shall be maintained in a secure manner with reasonable environmental protection from deterioration for the life of the program. Electronic and hardcopy records shall be centrally maintained (i.e., on the shared drive and/or in the QA Records Room) according to established PDP procedures. Maintenance shall be in an organized and systematic manner which allows accessibility by authorized staff.

8.2 Files and Records

8.2.1 The MPD Director shall ensure that copies of the following documents are maintained in the centralized files:

- PDP Semi-Annual Program Plans that specify the commodities and chemicals to be tested, as well as quarterly shipping charts that provide a schedule of samples to be collected and/or tested by each participant.
- A current PDP Master Schedule of administrative, sampling, and laboratory reviews and report submissions. The Master Schedule shall include the dates reviews were made and the dates findings were reported to appropriate individuals. The Master Schedule shall be posted to the Extranet.

8.2.2 The following documents shall be maintained in the centralized files by the assigned sampling and/or laboratory liaison(s):

- Administrative, sampling, and laboratory review reports.
- Authorizations for deviations from PDP SOPs.
- Semi-annual laboratory QA reports.

8.3 SOPs

8.3.1 The PDP Sampling Manager is responsible for maintaining all program sampling SOPs. This includes: scheduling issuance of SOPs, developing/revising SOPs in consultation with the MPD Director, distributing SOPs, updating the program Extranet/website with active SOPs, and maintaining all current and historical program SOPs (electronic and hardcopy files) related to sampling according to established PDP procedures.

8.3.2 The PDP QA Coordinator is responsible for maintaining all program administrative and laboratory SOPs. This includes: scheduling issuance of SOPs, developing/revising SOPs in consultation with the MPD Director, distributing SOPs, updating the program Extranet/website with active SOPs, and maintaining all current and historical program SOPs (electronic and hardcopy files) related to administrative and testing activities according to established PDP procedures.

8.3.3 The MPD Director is responsible for ensuring that internal PDP SOPs are prepared/revised. The QA coordinator is responsible for: distributing SOPs, updating the program Extranet/website with active SOPs, and maintaining all current and historical program SOPs (electronic and hardcopy files) related to internal PDP activities according to established PDP procedures.

8.4 Method Validation

All laboratories perform method validation studies and submit method validation reports and records to USDA/AMS in accordance with PDP-QC SOP.

8.4.1 The MPD Director shall appoint an individual to serve as the PDP Method Validation Coordinator. The Method Validation Coordinator shall:

- Perform a final review of all validation study reports prepared by laboratory liaisons to ensure that consistent policies are applied, makes recommendations based on findings.
- Track and file all method validation documentation (i.e., Letters of Intent, Method Validation Data Packages, associated PDP reviews, Letters of Concurrence, etc.) to ensure that all required studies are performed by all applicable laboratories and that Letters of Concurrence/requests for further data are issued by USDA/AMS within 90 days of receipt of the data package.

- Promptly communicate to the MPD Director delays in study reports submission, or issuance of USDA/AMS Letters of Concurrence/requests for further data.

8.4.2. Letters of Intent

- Letters of Intent submitted by laboratories shall be reviewed and verified against electronically submitted data (upon availability) by the assigned laboratory liaison..
- Letters of Intent shall be tracked and maintained in centralized files by the Method Validation Coordinator.

8.4.3. Method Validation Data Packages

8.4.3.1 The assigned laboratory liaison shall review the data package according to established internal PDP procedures and draft a Letter of Concurrence, including any recommendations or requirements for additional data. Refer to PDP internal procedure, PDP-INTN-QC-01.

8.4.3.2 The Method Validation Coordinator shall perform a final review of all validation study reports prepared by laboratory liaisons to ensure that consistent policies are applied, to make recommendations based on findings, and ensure all required studies are performed by applicable laboratories.

8.4.3.3 The MPD Director is responsible for final authorization of the Letter of Concurrence issued to the submitting laboratory.

8.5 Proficiency Testing (PT) Program

8.5.1 All PDP laboratories analyzing routine PDP samples are required to participate in PT programs as coordinated by USDA/AMS.

8.5.2 The MPD Director is responsible for management of the PT programs and shall assure that PT samples are delivered on schedule and reports are prepared. The PT schedule and the reports will be posted to the Extranet.

8.5.3 The assigned laboratory liaison shall be responsible for monitoring that laboratory's performance on PT rounds and shall communicate any concerns/corrective

actions to the MPD Director. The MPD Director shall be responsible for overall monitoring of the proficiency of laboratories.

8.6 Technical Advisory Group (TAG)

8.6.1 The PDP QA Coordinator , in consultation with the MPD Director, shall serve as liaison to the PDP Technical Advisory Group (TAG). The TAG shall be comprised of at least three selected members of participant QAOs and/or TPMs and shall address program QA issues/concerns.

8.6.2 Each TAG member shall serve a three-year term, with the final year served as the Presiding Member. The Presiding Member shall have sign-off responsibility for PDP program SOPs, with the exception of administrative SOPs, developed or revised during their term.

8.7 Records Archival

The MPD Director shall appoint an individual to serve as the National Archives and Records Administration (NARA) contact for records disposition. The NARA contact shall be responsible for coordinating and tracking data submissions to NARA.

9. Standard Operating Procedures (SOPs)

9.1 Description of an SOP

SOPs are written instructions on how to perform tasks and procedures. SOPs are intended to ensure consistency of data, quality, and procedures throughout the PDP studies and to be utilized for audit or review purposes. **Note**: The term "SOP" may be interpreted as any type of participant internal document (e.g., policy, work instructions, etc.).

9.2 Components of an SOP

9.2.1 This SOP serves as a guideline of the basic components to be included in the preparation of an SOP. They may contain a Purpose, Scope, Outline of Procedures, References (if any), and Specific Procedure(s).

9.2.2 Program and participants' SOPs shall be uniquely identified and shall include at least a title, revision number, and effective date.

9.2.3 The Specific Procedure(s) shall be written in precise and explicit terminology. The SOP shall be detailed enough to cover every aspect of the procedure and is intended to provide consistency in the conduct of routine operations and to serve as a guide for the conduct of audits. It is not intended to replace experience and basic training but may be used as a training tool.

9.3 USDA/AMS SOPs

9.3.1 USDA/AMS shall provide SOPs giving the requirements for common aspects of the program, and specific requirements as needed. These include SOPs in the areas of:

- Administrative Procedures
- Sampling Procedures
- Laboratory Procedures
- Internal MPD Procedures

9.3.2 All USDA/AMS SOPs shall be considered directive, unless the SOP explicitly states that the SOP or a section of the SOP is suggestive in nature.

9.3.3 USDA/AMS shall have immediately available manuals and SOPs relative to the laboratory or field procedures being performed. Published literature may be used as a supplement to SOPs.

9.3.4 Each USDA/AMS administrative SOP, as well as USDA/AMS internal MPD SOPs, shall be approved and signed by the USDA/AMS MPD Director. Each USDA/AMS sampling SOP shall be prepared and signed by the author/revisionist, approved and signed by the MPD Director, and reviewed and signed by the Presiding Member of the Sampling Advisory Group. Each USDA/AMS laboratory SOP, with the exception of the administrative series, shall be prepared and signed by the author/revisionist, approved and signed by the MPD Director and reviewed and signed by the Chairperson/Presiding Member of the PDP Technical Advisory Group.

9.3.5 All USDA/AMS SOPs shall be revised as needed.

9.3.6 An index of USDA/AMS SOPs shall be maintained and distributed along with any SOP revisions.

9.3.7 Distribution of the SOPs, original and subsequent revisions, shall include the USDA/AMS MPD Director and MPD Archives; participating facilities' Administrative Managers, Sampling Managers, TPMs, and QAOs; and all other applicable personnel.

9.3.8 Each sampling and laboratory participant shall maintain a copy of current USDA/AMS PDP SOPs and SOP index.

9.4 State/Facility Internal SOPs

9.4.1 Each participant shall prepare internal SOPs in writing, giving specific details of procedures and methods utilized to comply with the USDA/AMS SOPs. Following changes to the USDA/AMS SOPs, each participant shall update their internal SOPs (if necessary for compliance) no later than three months after the USDA/AMS SOPs' effective date. The internal SOPs shall ensure the quality and integrity of data.

9.4.2 Each participant shall have immediately available manuals and SOPs relative to the procedures being performed. Published literature may be used as a supplement to SOPs.

9.4.3 Authorized employees or duly designated representatives of USDA/AMS shall have access to internal SOPs during sampling and laboratory reviews.

9.4.4 Each internal SOP shall be approved by at least two of the following senior managers: the QAO, the laboratory Administrative Manager or TPM, the Sampling Manager, or Sampling Administrative Manager, and the approval shall be recorded. The approval may be recorded by use of signature blocks in the SOP itself, or separately. Alternatively, electronic document management systems may also be utilized to record approvals. Each participant shall maintain copies of current and historical internal SOPs as well as records of the dates they are (or were) in effect.

9.4.5 SOPs shall be revised as needed.

9.4.6 Distribution of the internal SOPs, original and subsequent revisions, shall be available to each affected participant employee.

9.5 SOP Deviations

9.5.1 An SOP Deviation is the mechanism to allow participants to make pre-approved changes to written PDP requirements (SOPs, program plans, etc). Changes that are not pre-approved are dealt with via the participant's corrective action process.

9.5.2 An SOP Deviation request is submitted from the participant to USDA/AMS. The request shall be in writing but may be informal (e.g. e-mail) and may originate from the TPM, QAO, Sampling Manager, and/or Administrative Manager. Requests from laboratory participants shall include the QAU in order to ensure that any deviations do not compromise data quality.

9.5.3 The SOP Deviation request shall cite the particular SOP (including revision and subsection numbers) or other requirement. A description of need and/or rationale shall be included. The narrative should make clear the scope of the request (e.g. a particular sample, project, timeframe, etc., or a permanent change that would be in effect until affected by an SOP revision).

9.5.4 Additional information may be requested from the participant by USDA/AMS in order to evaluate the request.

9.5.5 The MPD Director shall sign and approve all letters of deviation and shall ensure that any authorization for deviations from approved program plans or program SOPs does not compromise integrity of data. The MPD Director shall ensure that precise and technically accurate documentation of such deviations is maintained. In lieu of a formal deviation letter, approval via email is acceptable for one-time deviation requests (e.g., apple samples stored on the counter overnight instead of in a refrigerator) submitted via email by the laboratory (see 9.5.2 above).

9.5.6 USDA/AMS may issue program-wide deviations (e.g. addressed to all Sampling Managers, all TPMs, etc) if applicable. Program-wide SOP Deviations will be posted in the SOP section of the USDA/AMS Extranet.

9.5.7 The participant shall maintain records of USDA/AMS authorizations for deviation

from PDP SOPs/plans and ensure that they are communicated to appropriate personnel.

9.5.8 When a revised PDP SOP is issued, participants are not required to submit a new SOP Deviation request provided the revision to the SOP does not impact operations (e.g. revision number and subsection number changes).

Brenda Foos 6/28/19

Approved By: Brenda Foos Date
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ORIGINAL SIGNATURE PAGE MAINTAINED BY USDA, AMS, SCIENCE & TECHNOLOGY, MONITORING PROGRAMS DIVISION
ELECTRONICALLY REPRODUCED SIGNATURE

Revision 7 July 2019 Monitoring Programs Division

- Changed PDP Program Administrative Director and PDP Technical Director titles to MPD Director throughout the document
- Changed liaison chemist to laboratory liaison throughout the document
- Renumbered sections 6.3.1 through 6.9.5
- Updated sections 6.3.1 through 6.4.2.6 by combining PDP Program Administrative Director and PDP Technical Director duties
- Updated section 6.3.12 by removing sampling and laboratory review report distribution to AMS Compliance and Analysis Programs
- Changed laboratory to State/facility in section 6.4.1
- Added laboratory liaison to section 6.4.2.6
- Added clarification to sections 7.1.1.2, 7.1.2.2 and 7.1.3.2 regarding MPD contact for laboratory purchase plans
- Added liaison reference to section 7.1.3.4
- Changed PDP QAO to PDP QA Coordinator in sections 8.1.2, 8.3.2, 8.3.3 and 8.6.1
- Updated section 8.4.1
- Clarified section 8.4.3.2 by removing duplicate entries referenced in section 8.4.1

Revision 6 May 2017 Monitoring Programs Division

- Updated section 3 by changing Attachment 4, removing Attachments 5 and 6 and renumbering Attachments 7 through 11
- Updated section 5.5.2.4 to include box listing
- Updated section 5.5.2.5
- Renumbered Attachments 7,8,9,10 and 11 to 5,6,7,8 and 9 throughout the document

Revision 5 May 2016 Monitoring Programs Division

- Clarified archival procedures for method validation and proficiency testing packages in section 5.5.2.3
- Clarified requirements for completing AD112 in section 7.3.1
- Added option for email approval of deviation requests in section 9.5.5

Revision 4 April 2015 Monitoring Programs Division

- Reformatted sections 5.4.1, 5.4.2.2, and 5.4.2.3
- Updated MDP address in section 6.2.2
- Added reference to Safety Data Sheet to section 6.5.2.5
- Added requirement for trip synopsis to section 7.1.3.4
- Reformatted section 7.3
- Added section 7.4 Forms Instructions
- Updated internal SOP approval documentation in section 9.4.4

Revision 3 October 2013 Monitoring Programs Division

- Removed references to MDP throughout document
- Changed MP references to USDA/AMS or PDP (as appropriate) throughout the document
- Modified Section 2, Scope to indicate SOP does not supersede Cooperative Agreement
- Specified laboratory shall notify USDA/AMS of corrective action resolution in section 6.9.4
- Updated Section 7
- Added language to section 9.4.1 to allow States 90 days to modify internal SOPs after USDA/AMS SOPs issued

Revision 2 October 2011 Monitoring Programs Division

- Updated the entire SOP, including SOP number, with the new program's name Monitoring Programs Division or MP instead of MPO
- Section 5.4.1.1: added annual and/or semi-annual Federal/State meeting minutes and/or presentations to list of records to be maintained by MP for 10 years
- Removed section 5.4.1.2 regarding MP retention of electronic databases and data summaries
- Section 5.4.2.1: removed statement regarding 2 year retention at laboratory of records requiring a total of 25 years retention (2 year requirement is addressed in Section 5.4.3.1)
- Moved Section 5.5.2.1 ("Each data package retained shall be filed by calendar year and month.") to become new Section 5.5.1.4
- Renumbered sections in 5.5.2
- Section 6.4.7.5: removed QAO from sampling review report distribution list
- Section 6.7.6: removed requirement for signature of reviewer (first bullet) and removed word "printed" from second bullet ("The printed name(s) and title(s) of the person(s) performing the review")
- Moved Section 6.7.6 to become Section 6.7.5 and renumbered old Section 6.7.5 as 6.7.6
- Section 6.7.8 – reworded as: "All MDP/PDP supporting records for sampling activities are stored, retained, and transferred to the archives as specified in Sections 5.4 and 5.5."

- Section 6.8.7 – reworded as: "All MDP/PDP raw data and supporting laboratory records are stored, retained, and transferred to the archives as specified in Sections 5.4 and 5.5."
- Section 6.9: added requirement for laboratory to appoint and individual within the QAU as the QAO
- Moved Section 6.9.6 requirement for QAU to ensure that deviations are properly authorized and documented to new Section 9.5, "SOP Deviations"
- Section 7.3: revised inventory requirements for clarification (laboratory shall immediately notify MP via email of equipment installation and shall add the equipment to the MDP/PDP Equipment Inventory Database within 30 days of installation) and to provide the reason for immediate MP notification of equipment installation (allows MP to process payment and reconciled affected reimbursement request)
- Section 8.3: changed title from "SOPs and Deviations from SOPs" to "SOPs"
- Moved Section 8.3.4 requirement for Technical Director approval and documentation of deviations to new Section 9.5
- Moved Section 9.4.5 requirement for participant maintenance and communication of deviations as well as stipulation that MP may require supporting documentation to new Section 9.5; renumbered remaining Sections
- Added new Section 9.5, "SOP Deviations"
- Section 9.4.1 – added provision for updating internal SOPs: "Due to the time interval between issuance dates and effective dates for USDA/AMS SOPs, each participant may update their internal SOPs in order to comply at any time during the time interval."
- Updated Attachment 1 with the e-mail addresses
- Updated Attachment 9 by replacing "Room Location" with "Location" and added footnote that the specific location within the laboratory is required to be documented (examples provided are room number, GC section)
- Updated Attachment 9 by adding new, required field for funding source (percentage of MDP/PDP funds used for purchase)

Revision 1	October 2010	Monitoring Programs Office

- Updated References section
- Updated and reorganized section 5.4 (Data and Records Retention Periods)
- Updated and added new requirements for records' transfers to FRC in section 5.5.2
- Updated sections 6.5.2.9 regarding Responsibilities of Participants on updating MPO
- Updated requirements for laboratory review repots in section 6.7.6
- Updated sections 7.2 and 7.3 regarding Purchases and Equipment Inventory requirements
- Updated section 8.6.1 regarding MDP TAG
- Removed section 9.2.4 regarding the internal SOP formatting.

USDA/AMS Pesticide Data Program
Designated Federal Records Centers

Laboratory+	Region	Send to:		
		Name	Address	MP to E-Mail
California Department of Food and Agriculture	Pacific Region	FRC	1000 Commodore Drive San Bruno, CA 94066-2350	SanBruno.transfer@nara.gov
Florida Department of Agriculture and Consumer Services	Southeast Region	FRC	4712 Southpark Blvd. Ellenwood, GA 30294	atlanta.transfer@nara.gov
Maryland Department of Agriculture	Washington National Records Center	FRC	4205 Suitland Road Suitland, MD 20746-8001	suitland.transfer@nara.gov
Michigan Department of Agriculture	Great Lakes Region	FRC	7358 South Pulaski Road Chicago, IL 60629-5898	chicago.transfer@nara.gov
New York Department of Agriculture and Markets	Northeast Region	FRC	National Archives-Central Plains Region 200 Space Center Drive Lee's Summit, MO 64064-1182	KansasCityCave.transfer@nara.gov
Ohio Department of Agriculture	Great Lakes Region	FRC	Federal Records Center – Dayton 3150 Springboro Road Dayton, OH 45439-1883	kingsridge.transfer@nara.gov
Texas Department of Agriculture	Southwest Region	FRC	1400 John Burgess Drive Fort Worth, TX 76140	FtWorth.transfer@nara.gov
Washington State Department of Agriculture	Pacific Alaska Region	FRC	6125 Sand Point Way NE Seattle, WA 98115-7999	seattle.transfer@nara.gov
USDA, AMS, S&T, National Science Laboratory	Southeast Region	FRC	4712 Southpark Blvd. Ellenwood, GA 30294	atlanta.transfer@nara.gov
USDA, GIPSA, TSD, Pesticide Residue Laboratory	Central Plains Region	FRC	17501 West 98th Street, Room 47-48 Lenexa, KS 66219	lenexa.transfer@nara.gov

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

RECORDS TRANSMITTAL AND RECEIPT	Complete and send original and one copy of this form to the appropriate Federal Records Center for approval prior to shipment of records. See specific instructions on reverse.	PAGE 1	OF PAGES

1	TO	(Complete the address for the records center serving your area *as shown in 36 CFR 1228.150.)* **Federal Records Center**		5 FROM (*Enter the name and complete mailing address of the office retiring the records. The signed receipt of this form will be sent to this address.)*
2	AGENCY TRANSFER AUTHOR-IZATION	TRANSFERRING AGENCY OFFICIAL *(signature and title)*	DATE	
3	AGENCY CONTACT	TRANSFERRING AGENCY LIAISON OFFICIAL (*Name, office and telephone No)*		
4	RECORDS CENTER RECEIPT	RECORDS RECEIVED BY (*Signature and Title)*	DATE	

Fold Line

6 **RECORDS DATA**

ACCESSION NUMBER			VOLUME *(cu. ft.)*	AGENCY BOX NUMBERS	SERIES DESCRIPTION *(with inclusive dates of records)*	RESTRIC-TION	DISPOSAL AUTHORITY *(schedule and item number)*	DISPOSAL DATE	COMPLETED BY RECORDS CENTER			
RG	FY	NUMBER							LOCATION	SHELF PLAN	CONT.TY PE	AUTO. DISP.
(a)	*(b)*	*(c)*	*(d)*	*(e)*	*(f)*	*(g)*	*(h)*	*(i)*	*(j)*	*(k)*	*(l)*	*(m)*

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

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1 TO (Complete the address for the records center serving your area *as shown in 36 CFR 1228.150.)*

Federal Records Center

(TYPE YOUR RECORDS FACILITY ADDRESS, SEE ATTACHMENT 1 LIST)

5 F

USDA-AMS-S&T
Monitoring Programs Division
1400 Independence Ave, SW
Room 0611, Stop 0275
Washington, DC 20250

2	AGENCY TRANSFER AUTHOR-IZATION	TRANSFERRING AGENCY OFFICIAL *(signature and title)* Dawn Fay, Management Analyst	DATE
3	AGENCY CONTACT	TRANSFERRING AGENCY LIAISON OFFICIAL (*Name, office and telephone No)* (Laboratory point of contact)	
4	RECORDS CENTER RECEIPT	RECORDS RECEIVED BY (*Signature and Title)*	DATE

6 **RECORDS DATA**

ACCESSION NUMBER			VOLUME *(cu. ft.)*	AGENCY BOX NUMBERS	SERIES DESCRIPTION *(with inclusive dates of records)*	RESTRIC-TION	DISPOSAL AUTHORITY *(schedule and item number)*	DISPOSAL DATE	COMPLETED BY RECORDS CENTER			
RG	FY	NUMBER							LOCATION	SHELF PLAN	CONT. TYPE	AUTO. DISP.
(a)	*(b)*	*(c)*	*(d)*	*(e)*	*(f)*	*(g)*	*(h)*	*(i)*	*(j)*	*(k)*	*(l)*	*(m)*
			Number of boxes		United States Department of Agriculture, Agricultural Marketing Service S&T, Monitoring Programs Division, Pesticide Data Program (PDP) ROUTINE DATA PACKAGE RECORDS (INCLUDE YEAR) METHOD VALIDATION DATA PACKAGE RECORDS (INCLUDE YEAR) SUPPORTING DOCUMENTATION RECORDS (INCLUDE YEAR) SAMPLING DOCUMENTATION RECORDS (INCLUDE YEAR)	R						

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

United States Department of Agriculture
Pesticide Data Program
Box Listing

PDP RAW-DATA PACKAGE RECORDS (CY 2014)

Box #1/6	PDP Data Set SS 1401 PDP Data Set SS 1402 PDP Data Set SS 1403
Box #2/6	PDP Data Set SS 1404 PDP Data Set SS 1405 PDP Data Set SS 1406

PDP SUPPORTING DOCUMENTS RECORDS (CY 2014)

Box #3/6	PDP Working/Calibration Standard Logbook 2014 PDP Mixed/Intermediate Standard Logbook 2014 PDP Standard Disposal Logbook 2014 PDP Standard Use Logbook 2014 PDP Reference Freezer Temperature Logbook 2014 PDP Reagent Logbook 2008-2014 PDP Stock Standard Logbook 2014 PDP Pipette Performance Logbook 2009-2015 PDP Standard Comparison Logbook 2014 (1 of 2) PDP Standard Comparison Logbook 2014 (2 of 2)

PDP METHOD VALIDATION DATA PACKAGE RECORDS (CY 2014)

Box #4/6	SS Method Validation/LOD Verification – 11/2012 SS Method Validation Precision & Accuracy -11/2012 SS Method Validation Method Range/Method Range Ext. 10/2014

PROFICIENCY TESTING RECORDS (CY 2014)

Box #5/6	PDP FAPAS PT 19165 Pears (Feb.-April 2014) PDP PT Set # 229 CDFA Grapes (Oct-Nov. 2014) PDP PT Set # 228 GB (May-June 2014)

PDP SAMPLING DOCUMENTS RECORDS (CY 2014)

Box #6/6	Food Donation Receipts (Feb.-April 2014) Vendor Payment Receipts (Feb.-April 2014) Hand-Written data for Sample Information Forms (Feb.-April 2014)

United States Department of Agriculture
Microbiological and Pesticide Data Programs
Instructions for Assembly and Packaging of Record Boxes

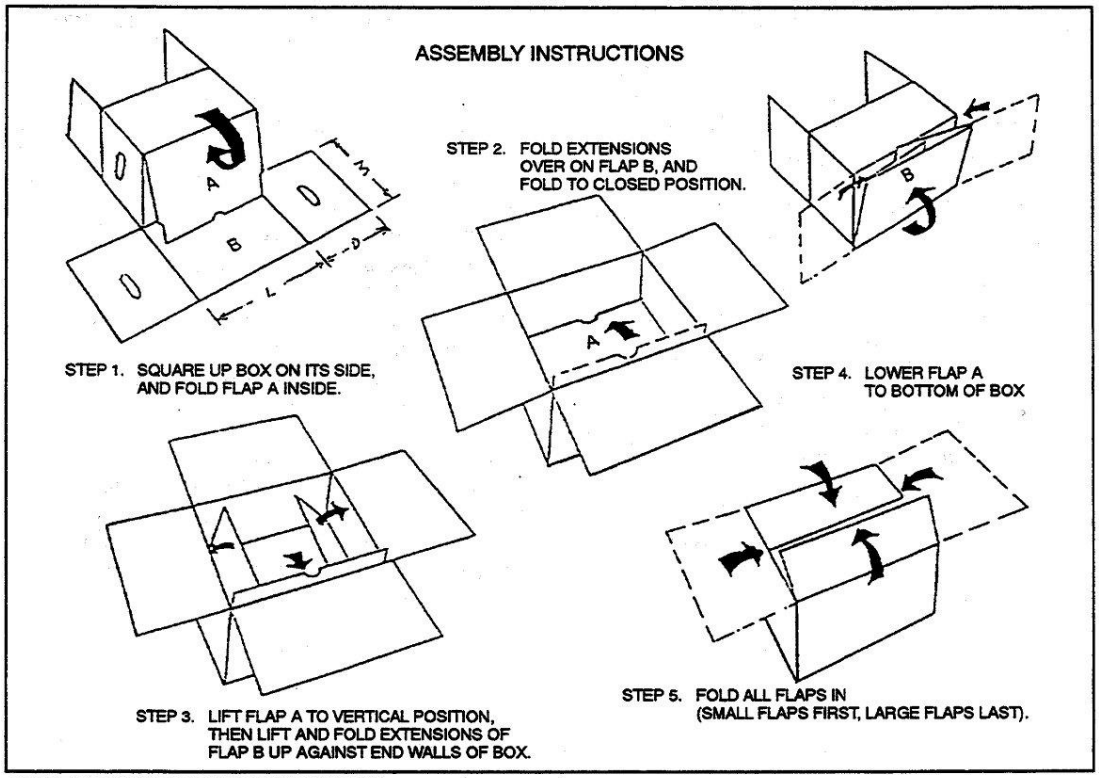

FIGURE 1 FRC BOX ASSEMBLY INSTRUCTIONS

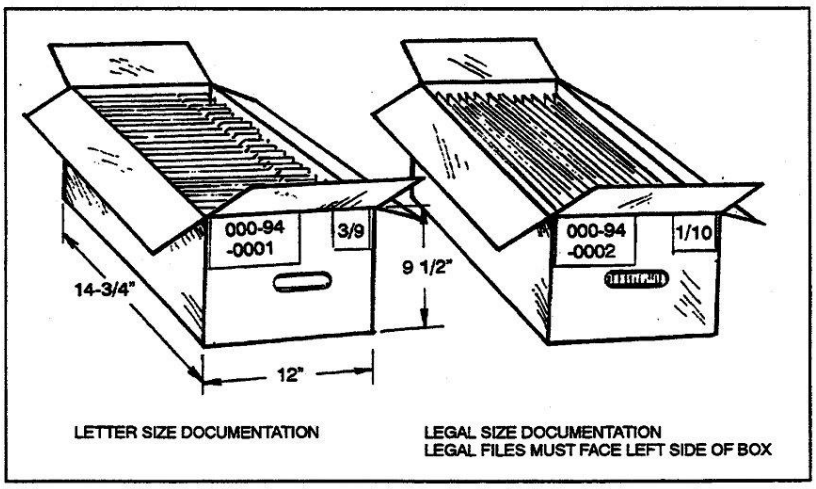

FIGURE 2 FILE PLACEMENT IN BOX AND LOCATION OF BOX IDENTIFICATION

REQUISITION/PROCUREMENT REQUEST FOR EQUIPMENT SUPPLIES OR SERVICES *(Instructions on reverse)*

PAGE 1 OF PAGES

2. REQUISITION/PROCURMENT REQUEST NO.	3. ACT NUMBER	4. DATE PREPARED	5. JOB/PROJECT NUMBER
1	N/A		

6. TO *(Stockroom/Contracting office, Name and Location)*	7. FROM *(Requisitioning office, Name, Symbol, Location and Telephone Number)*
MPD Director	
8. FOR INFORMATION CALL *(Name and Telephone Number)*	9. RECEIVING OFFICE *(Name, Symbol and Telephone Number)*
202-572-8167	

10. ACCOUNTING CLASSIFICATION

FUND	ORG. CODE	B/A CODE	O/C CODE
FUNC CODE	C/E CODE	PROJ/PROS. NO.	CC-A
W/ITEM	CC-B	PRT/CRFT	

11. SHIP TO *(Address, ZIP Code and Telephone Number)*

12. CONTRACT NUMBER

Enter Cooperative Agreement Number

ITEM NO. FORM OR STOCK NUMBER (13)	DESCRIPTION OF ARTICLES OR SERVICES (14)	QUAN-TITY (15)	UNIT OF ISSUE (16)	UNIT PRICE (17)	AMOUNT (18)
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
					$0.00
IF ADDITIONAL SPACE IS REQUIRED, USE GSA FORM 49A, REQUISITION/PROCUREMENT REQUEST		19. TOTAL AMOUNT INCLUDING CONTINUATION ▶			$0.00

20a. TYPED NAME AND TITLE OF FUND CERTIFYING OFFICIAL		21a. TYPED NAME OF REQUISITONER	
20b. SIGNATURE	DATE	21b. SIGNATURE	DATE
22. LIST ATTACHMENTS		23a. TYPED NAME OF APPROVING OFFICIAL	
		23b. SIGNATURE	DATE
		24. SHIPPED BY ☐ FREIGHT ☐ PARCEL POST ☐ EXPRESS ☐ MAIL	
		25. FILLED BY	26. PACKED BY / 27. CHECKED BY
		28. BILL OF LADING NUMBER	29. DATE SHIPPED

GENERAL SERVICES ADMINISTRATION — **GSA** FORM **49** (REV. 7-91)

Equipment Inventory

USDA, AMS, S&T, Monitoring Programs Division

Lab or State	Item Description	Manufacturer	Model	Serial Number	Location*	Unique Internal Lab ID	Acquisition Cost	Approval Date	Purchase Date	Purchase Ref	Surplus**	% of MDP/PDP Funds Used for Purchase	Remarks***
WA1	LC/MS/MS	Micromass	Quattro Premier	VAA 045	Rm 225	Waters #1	$300,000.00	1/1/2004	6/1/2004	041846	FALSE	65% USDA	

*Specific location within the laboratory (e.g., room number, GC section, etc.)
**Enter “True” if the equipment is designated as surplus, and “False” if the equipment is still active.
***Enter any additional comments concerning the item (e.g., more detailed description, asset number, etc.)

Appendix B – Laboratory Quality Standards for Pesticides: Cannabis Science Task Force

U.S. DEPARTMENT OF AGRICULTURE

REPORT OF UNSERVICEABLE, LOST, STOLEN DAMAGED OR DESTROYED PROPERTY

PROPERTY REPORT NO.

DATE

SECTION I - ACCOUNTABLE PROPERTY OFFICER'S REPORT

1. STATUS OF PROPERTY *(Check only one-report each one type separately)*

- ☐ Unserviceable
- ☐ Obsolete
- ☐ Damaged
- ☐ Lost or Stolen
- ☐ Cannibalized for parts
- ☐ Destroyed
- ☐ Others

2. REPORTING ACTIVITY *(Show agency, unit and address)*

3. PROPERTY ITEMS *(See attachment for additional entries)*

QUANTITY (Or property no.) A	ITEM DESCRIPTION AND OTHER DETAILS, INCLUDING SERIAL NUMBERS AND ACQUISITION DATE *(Give present condition and estimated cost of repair)* B	ACQUISITION COST C	*EXPLANATION/DISPOSAL INSTRUCTIONS (If lost, stolen, or destroyed, give detail. Was this reported to proper authorities?)* D

4. NAME IN PRINT AND SIGNATURE OF CUSTODIAN	DATE	5. NAME IN PRINT AND SIGNATURE OF ACCOUNTABLE PROPERTY OFFICER	DATE

SECTION II - PROPERTY MANAGEMENT OFFICER'S REVIEW AND RECOMMENDATION

DETERMINATION FOR LOST, STOLEN, DAMAGED, OR DESTROYED PROPERTY

1. After due consideration of all known facts and circumstances in this case, it is determined that:

- ☐ a. The loss, theft, damage or destruction did not result from employee negligence and any involved employees are hereby relieved of liability.
- ☐ b. There appears to be gross negligence involved; therefore, the case returned to agency officials for appropriate action under the Debt Collection Act.
- ☐ c. There appears to be negligence involved; therefore, the case is returned to agency personnel officials for consideration of disciplinary action.

2. NAME IN PRINT AND SIGNATURE OF PROPERTY MANAGEMENT OFFICER	3. DATE

SECTION III - AUTHORIZATION FOR CANNIBALIZATION, ABANDONMENT, OR DESTRUCTION OF UNSERVICEABLE PROPERTY

1. Unserviceable property listed above is hereby authorized for cannibalization, abandonment, or destruction in accordance with FPMR 101-45.9 based on any of the following determinations as further explained in section I-3(D):

- ___ a. Property has no commercial value.
- ___ b. Health, safety, or security considerations require immediate abandonment or destruction.
- ___ c. Costs of care and handling exceed expected small lot sales proceeds.
- ___ d. Regulation or directive requires abandonment or destruction.
- ___ e. Property is uneconomical to repair/not needed by another user and may be cannibalized for parts. *(Cannibalization is a form of use and property management regulations shall apply. Remainder of property must be disposed of through usual procedures.)*

2. SIGNATURE OF PROPERTY MANAGEMENT OFFICER	3. DATE

SECTION IV - CERTIFICATION FOR COMPLETION OF CANNIBALIZATION, ABANDONMENT, OR DESTRUCTION: *I certify that cannibalization, abandonment, or destruction action for the items authorized under Section III was completed on this date in accordance with I-3(D).*

1. SIGNATURE OF ACCOUNTABLE PROPERTY OFFICER	2. DATE
3. SIGNATURE OF WITNESS	4. DATE

SECTION V - CERTIFICATIONS OF PROPERTY AND FISCAL OFFICERS

1. SIGNATURE OF PROPERTY MANAGEMENT OFFICER *(The necessary entries have been made to adjust property records.)*	2. DATE
3. SIGNATURE OF FISCAL OFFICER *[The necessary action has been taken to adjust the accounting records and, where required by a determination made under Section II above, to effect collection from involved employee(s).]*	4. DATE

AD FORM 112 (Rev. 3/94)

Clear Form

This form was electronically produced by National Production Services Staff

United States Department of Agriculture

Report of Transfer or Other Disposition or Construction of Property

Report No.

Date

3. Proceeds Received

$

1. Type of Transaction (Report each type separately)

☐ Transfer ☐ Sale ☐ Trade In ☐ Donation

☐ Construction ☐ Rehab ☐ As-Is

2. Authorization Reference

4. Reporting Agency

USDA/AMS/S&T/MPD

A. Organizational Unit

B. Location

C. Signature

D. Title

5. Receiving Agency (Or Name of Purchaser or Donee):

A. Organizational Unit (Or Address of Purchaser)

B. Location

C. Signature

D. Title

E. Date

6. Property Items

Quantity (Or Prop. No.)	Item Description (Give Full Details Including Serial Numbers, If Any, and Condition Code)	Inventory Value

Certifications of Property and Fiscal Officers

7. Property Officer: This transaction is completed and the necessary entries have been made to adjust the property records proceeds, if any, are to be deposited to:

Signature

Date

8. Fiscal Officer

A. ☐ The sum indicated below has been received in payment for the property disposed of.

B. ☐ The necessary entries have been made to adjust the accounting records.

Amount ($)

Schedule No.

Signature

Date

This form was electronically produced by National Production Services Staff

Form AD-107 (11/89)

Clear Form

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

SOP No.: PDP-Glossary		Page 1 of 14
Title: Glossary		
Revision: 10	Replaces: 10/01/2011	Effective: 01/01/2015

1. Purpose:

To ensure correct and consistent usage of the specific technical terms associated with USDA/AMS-Pesticide Data Program (PDP) Standard Operating Procedures (SOPs).

2. Scope:

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

3. Outline of Procedure:

5.1 Glossary of Terms

4. References:

- U.S. EPA, *Good Laboratory Practices Standard Regulations*, TSCA 40 CFR part 792
- U.S. EPA, *Good Laboratory Practices Standard Regulations*, FIFRA 40 CFR part 160
- Taylor, J.K., *Quality Assurance of Chemical Measurements*, Lewis Publishers, 1989

5. Specific Procedures:

5.1 Glossary of Terms

Administrative Manager: A scientist or other professional of appropriate education, training, and experience, who is designated by participant to administer PDP activities. These activities may include sampling management, laboratory management, budgeting, contracting, purchasing, inventory maintenance, and receipt of QA reports and associated corrective actions.

Accuracy: The concept of “exactness” or “correctness”. It answers the question, “how close is the result to the true value?”

Analyte Protectant: Substance added to sample extracts and analytical standard solutions to reduce analyte interactions with active sites in a GC system and thus increase analyte response and improve peak shape similarly to matrix-induced peak enhancement.

Analytical Method: A procedure consisting of several laboratory procedures, which when completed, produces a quantitative and/or qualitative result for the tested substance.

Annual Plan: A general series of projected proposals, actions, and/or activities to be undertaken by an organization during a twelve month period to accomplish its goals and mission.

Batch: A specific manufactured or formulated quantity or lot of test, control, or reference substance used in analytical determinations or a study that has been characterized by physical attributes such as a source identity, purity, composition, and stability. Batch can also include a discreet quantity of chemical or product prepared in a single procedure which exhibits uniform characteristics.

Below Quantifiable Level (BQL): The amount of residue in a sample matrix that is above the limit of detection and below the limit of quantitation. Confirmed data between LOD and LOQ shall be reported as BQL.

Bias: A systematic error inherent in a method or caused by some artifact or idiosyncrasy of the measurement system. Temperature effects and extraction inefficiencies are examples of the first kind. Blanks, contamination, mechanical losses, and calibration errors are examples of the latter kinds. Bias may be both positive and negative, and several kinds can exist concurrently so that net bias is all that can be evaluated, except under special conditions.

Blank Matrix: A matrix that does not produce an analytical response by the analytical method under investigation for the analyte(s) of interest.

Calibration: Comparison of a measurement standard or instrument with another standard or instrument to report, or eliminate by adjustment, any variation/deviation in the accuracy of the item being compared.

Characteristic: A physical or chemical property that serves to differentiate between compounds. The differentiation may be either quantitative (by variables) or qualitative (by attributes).

Check sample: Any matrix sample prepared for the purpose of determining biases, accuracy, and/or precision among analysts and/or laboratories or of a single analyst or laboratory.

Chromatographic Time Segment (CTS): The segment along the baseline of a chromatogram used in the determination of method noise (e.g., a broad CTS - the length of the entire chromatogram, a narrow CTS - an elution window of one or more analytes).

% Coefficient of Variation (CV): The ratio of the standard deviation, *s*, of a set of numbers, *n*, to their average, $\bar{x}$, expressed as a percentage.

$$\%CV = \frac{s}{\bar{x}} * 100$$

Commodity Grouping: PDP commodity groups established to facilitate method evaluation. Grouping is based on EPA commodity grouping under 40 CFR 180, with modifications to further combine those commodities having similar matrix characteristics for analytical purposes.

Confirmation: Verification of an analytical finding.

Control Limits: Control chart limits established at the 99% confidence interval for a monitored system. Acceptance limits are set at three times the standard deviation, *s*, of a system around the best estimate of the data, generally the mean, $\bar{x}$. Thus, control limits established at $\bar{x} \pm 3s$ are expected to contain 99.7% of data produced by a system in statistical control.

Data Package: Package containing raw data for an analytical set. Each data package is uniquely labeled by year, month, and commodity and contains, at minimum, the following: instrument methods, reports/summaries of sample results, standardization/calibration reports or summaries, Sample Information Forms (SIFs), Laboratory Information Forms (LIFs), QA Information Forms (QIFs), and documentation of technical and QA review.

Data Set: Analytical results for samples in the same group.

Distinct Chromatographic Peak: A peak that displays an essentially Gaussian shape and is a least 3 times the peak height of the matrix plus high frequency noise.

Drift Noise: Drift appears as a continuous increase or decrease of signal in the chromatogram. This source of noise is typically due to fluctuation in variables such as temperature, pressure, and flow as well as electronic and electrical variations. Excess drift makes it impossible to do quantitative analysis.

Fortification Recovery: The ratio of the measured quantity of a given analyte to the known quantity spiked into the matrix spike. It is usually expressed as a percentage.

High Frequency Noise: The random or periodic signal fluctuation of the order of ten or more cycles per minute. This type of noise appears as a fuzzy baseline. It is typically caused by the electronics of the chromatographic system.

Homogenate: A sample that has been prepared according to sample preparation instructions and stored under appropriate conditions as stated in USDA/AMS-PDP SOP LABOP-3 Section 5.

Horwitz Expected %CVs: The interlaboratory (between laboratories) and intralaboratory (within laboratory) %CV values predicted by Horwitz based on concentration and defined as:

Interlaboratory $\%CV = 2^{(1 - 0.5\log C)}$, where C = concentration.

The intralaboratory %CV is defined as ⅔ times the interlaboratory value. A table of selected concentrations is presented below:

Concentration (ppm)	**C**	**Expected Interlaboratory %CV**	**Expected Intralaboratory %CV**
1	$1x10^{-6}$	16	11
0.5	$5x10^{-7}$	18	12
0.25	$2.5x10^{-7}$	20	13
0.1	$1.0x10^{-7}$	23	15
0.05	$5.0x10^{-8}$	25	17
0.01	$1.0x10^{-8}$	32	21
0.001	$1.0x10^{-9}$	45	30

The appropriate values may be used as a guideline when evaluating data and/or determining whether analytes should be considered a Marginal Performing Analyte.

Intermediate Dilutions: Dilutions from stock solutions used to prepare working solutions.

Limit of Detection (LOD): The lowest observable peak response for an analyte above the background noise, at least 3 times the system noise in matrix. This is normally calculated from a blank matrix in the retention window, or chromatographic time segment (CTS), of the peak of interest.

Limit of Quantitation (LOQ): The lowest concentration for which quantitative analytical data shall be reported in a particular laboratory. This is at least 10:1 signal:noise as described in LOD above.

Low Frequency Noise: This type of noise appears as very broad peaks in the chromatogram. It is most often caused by carryover of late eluting peaks from previous injections or low frequency electrical or electronic variations.

Marginal Performing Analytes: Analytes which do not meet linearity, calibration integrity, recovery (individual or mean), reproducibility (%CV values within the expected Horwitz intralaboratory values) or precision and accuracy criteria during method validation or continuing quality control. Marginal performing analytes established after following Method Validation must be documented in a deviation letter.

Marker Pesticides: Analytes specified as required to be spiked for each sample set analyzed due to their characteristics that represent some of the properties of the other analytes screened by that method.

Material Safety Data Sheets (MSDS): OSHA required documentation provided by manufacturers for each chemical produced. Information includes adverse effects, toxicity and relevant chemical data and necessary safety precautions.

Matrix Blank: Ideally, a previously characterized sample which shows no detectable or defined response for the analyte of interest within that analyte's chromatographic time segment (CTS). If a suitable sample is not available, a portion of one of the samples or purchased (e.g., organic) sample may be used.

Matrix Noise: Increase in baseline noise caused by co-extractives. Matrix noise may appear as a series of ill-defined and overlapping peaks on expansion of the baseline.

Matrix Spike: A blank matrix spiked with a known quantity of analytes. The spike is subjected to the entire analytical method along with samples within that set and provides a measure of the behavior of the analyte(s) for the sample set.

Mean: The arithmetic mean of a set of *n* values is the sum of all values divided by *n*.

Method Evaluation: That study conducted prior to the utilization, distribution, or publication of analytical methodology. The study determines if a specific analysis is feasible and sets acceptable statistical requirements for analytical results for future use of the method.

Neat Standard: Solid or liquid form of a pesticide, metabolite, or degradate obtained directly from the manufacturer or distributor with certified purity, expiration date, and lot number.

Peak-to-Peak Noise: Measured difference from the most positive noise to the most negative noise in the retention window of interest.

Post-extraction/pre-instrumentation: Stage following primary extraction (e.g., solvent, microwave) and prior to injection on the analytical instrument to be used for determination of residues. Examples would include solid phase extraction (SPE) cartridge used for extract clean-up or addition of internal standards for quantitation.

Precision: The degree of mutual agreement among individual measurements under similar experimental conditions.

Presumptive Tolerance Violation: A result is considered to be a presumptive tolerance violation if, one, the residue exceeds the tolerance level for a given commodity or, two, the confirmed residue found has no established tolerance on the given commodity and is above the limit of detection.

Primary Identification Technique: System used for initial determination/quantitation of residue to be reported.

Process Control: A compound spiked into each sample in an analytical set to give a measure of the integrity of a particular sample passing through an analytical process. The compound(s) should be chosen as representative of the compounds screened by that method, but should not be a compound of interest.

Proficiency Testing Sample: A check sample prepared as part of an interlaboratory proficiency testing program to determine accuracy, biases, and/or precision among participating laboratories.

Program Administrative Director: A scientist or other professional of appropriate education, training, and experience who is designated by USDA/AMS to be responsible for overall program administrative functions. These functions include program expansion, budgeting, cooperative agreements, memoranda of understanding, and major disbursement of funds.

Protocol: Approved written document clearly stating the plan of a study. The protocol shall address, at minimum, the following: objective of the study; sampling, testing, and reporting requirements and procedures; and QA requirements and criteria.

Quality Assurance: A system of activities whose purpose is to provide to the producer or user of a product or a service the assurance that it meets defined standards.

Quality Control: The overall system of activities whose purpose is to control the quality of a product or service so that it meets the needs of users. The aim is to provide quality that is satisfactory, adequate, dependable, and economical.

Quality Control Program: The collection of activities and events that serve to implement a system that assures that the quality of a product, process, or service satisfies the needs of the users.

Quality Assurance Unit (QAU): An individual or organizational unit designated by USDA/AMS or the management of an individual testing facility to be responsible for assuring the appropriate management that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with USDA/AMS program plans and SOPs. An individual participating facility QAU shall also be responsible for assuring that plans and SOPs issued by the laboratory conform to USDA/AMS requirements and are followed. No QAU duties may be performed by any technical personnel directly involved with the conduct of the analytical findings or a study.

Quarterly Plan: A general series of projected proposals, actions, and/or activities to be undertaken by an organization during a three month period to accomplish its goals and mission.

Range: The difference between the largest and the smallest value in a set.

Raw Data: Laboratory worksheets, logbooks, records, notes, chromatograms, calculations, instrument printouts, and any other data which are the result of original observations and activities of the testing program and are necessary for reconstruction and evaluation of the residue set. Computer printouts, data from automated instruments, chromatograms, maintenance and calibration logs, reference substances and samples etc., could be construed as raw data.

Re-aliquot: Removal of an additional portion of the original extract for clean-up and re-analysis.

Reference Substance: Any chemical substance, mixture, analytical standard, material other than a test substance, or water, that is administered to or used in analyzing the test system in the course of the testing program for the purpose of establishing a basis for comparison with the test substance for known chemical or biological measurements. Most commonly, reference substance refers to an analytical reference standard.

Re-injection: Re-injection of initial sample extract with appropriate analytical standards in order to obtain a reportable result(s). Fortification recovery failure, process control failure, instrument malfunction, etc may necessitate re-injection.

Relative Percent Difference (RPD): Expression of relative difference between two values. This number is defined as the absolute value between the first result, X_1, and the second result, X_2, divided by the mean of the two results. This is expressed as a percent and calculated as follows:

$$RPD = \frac{|X_1 - X_2|}{\frac{X_1 + X_2}{2}} * 100$$

Relative Standard Deviation (RSD): Expression of relative standard deviation of multiple values (e.g., points defining a calibration curve). This number is defined as the standard deviation of the values divided by the mean of the individual response factors. This is expressed as a percent and calculated as follows:

$$\%RPD = \frac{SD}{avg.\ RF} * 100,$$

where SD is standard deviation,

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

and RF is response factor, or the area or height of each standard divided by the concentration of that standard.

Remote Data Entry (RDE): System by which data may be transmitted electronically to USDA/AMS.

Rerun: Re-extraction of frozen homogenate for analysis. Process control failure, fortification recovery failure, tolerance violation issues, instrument malfunction, etc. may necessitate reruns.

Reserve Sample: An aliquot of a homogenate, which is stored under appropriate conditions (see definition of "homogenate" above) for the purpose of replicating tests or when immediate testing cannot be done.

Response Factor: Response of an analytical standard expressed as peak area or peak height divided by the concentration of that standard.

Review: A formal methodical examination by authorized USDA/AMS personnel of an organization's accounts, financial situation, raw data, records, reports, SOPs, and/or GLP/QA compliance of the laboratory facility, as well as all documents pertaining to the general operation of the facility.

Sample: Representative portion of material taken from a larger quantity of homogenate for the purpose of examination or analysis which can be used for judging the quality of a larger quantity.

Sample Set: A sample set is a group of samples, which are spiked individually with the designated process control(s), extracted with the required QC samples, and analyzed with the applicable required QC samples. Each set shall not exceed 35 samples. Required QC samples per set consist of a reagent blank, matrix blank, and matrix spike(s).

Sampling Manager: A professional of appropriate education, training, and experience who is designated by a participant to be responsible for the conduct of the participant's sampling procedures.

Semi-Annual Plan: A general series of projected proposals, actions, and/or activities to be undertaken by an organization during a six month period to accomplish its goals and mission.

Standard Deviation: Whenever a large number of measurements are made on a particular sample, the results of these measurements are distributed across a curve called a Gaussian Distribution Curve. The standard deviation, *s*, is a measure of width of distribution, which simplifies the results of a large number of measurements; *s* represents about 68% of the area, 2*s* about 95%, and 3*s* more than 99% of the area on both sides of the curve.

Stock Solution: Original solution made from the neat standard in a designated solvent. This solution will be used to prepare further dilutions.

Surrogate Spike: See Process Control.

Technical Director: A scientist of appropriate education, training, and experience who is designated by USDA/AMS to be responsible for overall sampling and technical conduct of the PDP residue study and monitoring of QA. The conduct includes interpretation, analysis, documentation, and reporting of results in an annual program summary, as well as providing technical guidelines for participating test facilities.

Technical Program Manager: A scientist or other professional of appropriate, education, training, and experience, who is designated by a participating laboratory to administer the technical conduct of PDP activities in that facility. These activities may include interpretation, analysis, documentation, and reporting of results.

Test Sample: Any item to which the test, control, or reference substance is administered or added to obtain an analytical profile to quantitate test substances or an unknown(s). The test system also includes appropriate groups or components of the system not directly treated with the test, control, or reference substance.

Testing Facility: A laboratory involved in the performance of analytical determinations for USDA/AMS-PDP, including those laboratories which are conducting residue studies for PDP and support laboratories conducting stability or other types of studies which may impact the program.

Testing Program: The Pesticide Data Program as conducted by designated sampling and laboratory participants; the program is also referred to as the "study".

Test Substance: A chemical substance or mixture of substances administered to or added to a test system as the subject of study.

Validation: The process of determining the suitability of methodology for providing useful analytical data. For PDP, this term is used interchangeably with method evaluation.

Verification: To verify or confirm that a residue is present by an alternate identification system (*note: due to the nature of mass spectrometry, this method is considered self-confirming*).

Warning Limits: Control chart limits established at the 95% confidence interval for a monitored system. Warning limits are set at two times the standard deviation, s, of a system around the best estimate of the data, generally the mean, $\bar{x}$. Thus, warning limits established at $\bar{x} \pm 2s$ are expected to contain 95.5% of data produced by a system in statistical control.

Working Dilutions: Solutions prepared from neat standards, stock solutions, or intermediate dilutions of stock solutions for spiking or injection.

Worst Case Matrix: The matrix that produces the highest average noise for a specified commodity group.

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

SOP No.: PDP-Glossary		Page 12 of 14
Title: Glossary		
Revision: 10	Replaces: 10/01/2011	Effective: 01/01/2015

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ELECTRONICALLY REPRODUCED SIGNATURE

Revision 10 December 2014 Monitoring Programs Division

- Updated MPD address

Revision 9 July 2011 Monitoring Programs Division

- Updated the Sample Set definition

Revision 8 July 2010 Monitoring Programs Office

- General update

Revision 7 July 2009 Monitoring Programs Office

- Added definition for marginal performing analyte
- Updated the SOP references throughout the document
- Updated references
- Updated Horwitz Expected %CVs definition

Revision 6 February 2008 Monitoring Programs Office

- Added Section 3, Outline of Procedure and renumbered remaining sections
- Removed acronym listing

Revision 5 October 2007 Monitoring Programs Office

- Modified format to conform with other SOPs
- Added acronym for MS/MS to section 4.1.b
- Added reference to SOP PDP-QC-10 to LOQ definition
- Corrected SOP PDP-QC-01 reference to matrix blank definition
- Added reference to SOP PDP-QC-13 to commodity group definition
- Added “at least” to LOD definition

Revision 4 July 2003 Monitoring Programs Office

- Modified scope for consistency with other SOPs
- Updated references
- Added to acronyms: micro-ECD, PFPD, and XSD
- Removed UAR from acronyms
- Added definitions for: analyte protectants, blank matrix, chromatography time segment, distinct chromatographic peak, drift noise, high frequency noise, low frequency noise, matrix noise, peak to peak noise, post-extraction/pre-instrumentation, precision, primary identification technique and the confirmation technique, re-aliquot, remote data entry, validation, verification, and worst case matrix

- Removed definitions for linearity and UARs
- Modified definition for data set, Material Safety Data Sheets

Appendix C. Analytical Workgroup Meeting Summaries

The following summarizes the Analytical Workgroup meeting discussions and highlights. Note that recommendations made by the Analytical Workgroup were not always adopted by the Task Force Steering Committee (Task Force). The final Task Force motions (Appendix F) display the final Steering Committee recommendations that were formulated from the Analytical Workgroups original recommendations.

August 21, 2019

Initial meetings were held immediately following the initial Task Force meeting on August 21, 2019. At that first meeting, the workgroup members shared a bit about their expertise and background in laboratory sciences. The members were briefed on the scope of the required initial legislative task, and a timeline and general work plan were presented to the workgroup.

September 3, 2019

Performance-based methods

The workgroup discussed various laboratory pesticide testing methods, practices, and protocols employed in environmental, agricultural, public health and current pesticide-certified cannabis testing laboratories. Currently there is a wide variety of pesticide methods and practices available for testing pesticides. For this reason, the concept of performance-based methods was introduced to the discussion. Under this approach, no specific preparation method, instrument, or detection method would be required. Rather a selected method or procedure would necessitate validation against an established standard set method validation protocols and performance criteria.

This approach allows each laboratory to select, or continue to use, any pesticide method and any analysis instrumentation designed with the analytical capability to detect pesticides, as long as it can be demonstrated to meet validation and performance criteria. The approach would not force a laboratory into purchasing any specific type of instrument, or in some cases, to purchase two instruments or any specialized piece of equipment. Additionally, the thought prevailed that the selection of one specific method or process now, one that currently performs well with current regulations, might not be flexible enough to accommodate future regulatory changes or advances in technology.

The performance-based methods design is purposely set up for flexibility. For example, if the regulatory thresholds of the pesticide are lowered, or if additional high-risk pesticides are identified, each laboratory could initially attempt to adapt and validate its current method. For some regulatory updates, method adaptation and method validation of current methods could quickly be accomplished and result in less delay in testing cannabis products to the new requirements. Use of additional or alternative methods or instruments would only be necessitated based on the performance capability of an individual lab. Meaning, some labs might be capable of testing all pesticides by employing one method, where another lab might need to use two or more methods or instruments. Laboratories would, however, be permitted to bring on additional methods or instruments whenever they desired, as long as they can successfully validate their new methods. Laboratories may choose to do this to employ the newest available technology, expand their workflow capacity, or possibly just to operate more economically, and doing all so by choice.

Several national and international regulatory programs use the performance-based methods (and standards) approach. The United States Department of Agriculture (USDA) Pesticide Data Program (PDP), a national pesticide residue-monitoring program, uses this approach when testing U.S. agricultural food supply. The European Commission for Health and Food Safety also does not mandate any particular testing method. Rather it requires the application of overarching analytical quality control and method validation procedures for testing pesticide residues.[9] Additionally, the performance-based method approach is used by environmental laboratories performing work with Environmental Protection Agency (EPA) SW- 846[10] test methods for evaluating sediments, soils, and wastes for regulatory purposes. Further, Ecology's Laboratory Accreditation Unit is well experienced in performing accreditation with this type of approach within Washington's current Environmental Laboratory Accreditation Program.

The recommendation that performance-based methods approach be used for testing pesticides in cannabis flower became the first workgroup recommendation to the Steering Committee.

Performance Criteria: United States Department of Agriculture (USDA) Pesticide Data Program

The WSDA workgroup lead, Mike Firman, presented the USDA PDP published standard operating procedures (SOPs) to the workgroup to use as a model for pesticide testing in cannabis. The USDA PDP employs the performance-based method concept and was developed specifically for agricultural testing.

The USDA PDP uses the established USDA PDP SOPs to serve as the guidance in testing commodities in the U.S. food supply, including fresh and processed fruit and vegetables, meats (beef and pork), poultry, eggs, fish, milk and dairy products, nuts, honey, infant formula, grains and grain products, and drinking water (surface, ground, and bottled). The USDA PDP SOPs supports mission and objectives specific to the USDA and the USDA PDP. The PDP SOP framework relies on trained USDA chemists and subject matter experts to preside over testing laboratories and the practices described within the SOPs.

Within Washington State, the USDA PDP SOPs are used in conjunction with testing practices that are performed by participating State-run laboratories, including the WSDA Chemical and Hop Laboratory in Yakima, Washington, where Mike Firman serves as the laboratory's director.

The Analytical Workgroup agreed to focus their work towards adapting the USDA PDP model design by reviewing the following USDA PDP SOPs:

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[9] "Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed" (SANTE; SANTE/11813/2017) https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides_mrl_guidelines_wrkdoc_2017-11813.pdf

[10] https://www.epa.gov/hw-sw846/sw-846-compendium

- PDP-QC - Chemical Compounds, PDP Commodity Groupings, Method Validation and Quality Control (Rev. 9, 09/01/19)
- PDP-LABOP - Sample Processing and Analysis (Rev. 10, 07/01/18)
- PDP-DATA - Data and Instrumentation (Rev. 6, 04/01/18)
- PDP-ADMIN - Administrative Procedures for the Pesticide Data Program (Rev. 7, 07/01/2019)
- PDP Glossary - Abbreviations and Terms used in SOPs (Rev. 10, 01/01/1)

The USDA PDP SOPs[11] selected for review are included in Appendix B.

Designation of a Client

The workgroup identified that an entity in Washington State would need to fill the oversight role that USDA holds within the USDA PDP SOP model. With regard to the PDP model, this entity would need to serve as the subject-matter expert for the content in, and application of, the PDP SOPs. To effectively function as the USDA does for its PDP, the state entity would need to function as a technical resource to the laboratories for guidance and use of PDP SOPs. This would require subject matter expertise in laboratory sciences, specifically in analytical methods and techniques used in the analysis pesticides, preferably with testing agricultural and food commodities using the PDP SOPs.

This entity should also function to ensure that work performed by the testing laboratories under this model is suitable and appropriate to support how the data will be used (e.g., enforcement or public health advisories). The term "Client" was suggested to convey that the final data user or client should preside over all aspects of the processes used in generation of data, the implementation of those processes, and the final use and acceptance of the data. This entity would serve to fill many roles and responsibilities explicitly described in the USDA PDP SOP. This concept became the second workgroup recommendation to the Steering Committee.

Overarching Concepts of the USDA PDP SOPs

The workgroup discussed that the use of the model requires that all users understand that it is designed around the "fit to purpose"12 concept. With this understanding, lab-analytical results need to be sufficiently reliable for decisions that will be made using them. For instance, the degree of confidence necessary for data used for enforcement of human health regulations likely would be greater than that for screening or data needed for qualitative purposes. By that merit, methods and processes generating data for decisions that carry more risk should receive more scrutiny that they are designed to perform appropriately. Purposeful method validation practices, ongoing

[11] Other USDA PDP SOPs exist; however, it was decided that their inclusion was deemed not necessary to apply for this purpose. All USDA PDP SOPs can be found at: https://www.ams.usda.gov/datasets/pdp/pdp-standard-operating-procedures

[12] Also known as "fit for purpose" and "fit for use".

performance criteria, quality assurance/quality control (QA/QC) requirements will further provide regular verification that the methods are implemented as intended and continue to fit the designed purpose.

The workgroup would provide those recommendations through the workgroup amendment of the PDP SOPs, however, the long term-term practice of updating and administering the cannabis-adapted PDP SOPs would need to be assumed by the established client. A final summery of adaptions document[13] would contain all workgroup proposed changes (Appendix A).

USDA PDP-QC - Chemical Compounds, PDP Commodity Groupings, Method Validation, and Quality Control

The Analytical Workgroup focused its first detailed SOP review on the USDA PDP-QC SOP, *Chemical Compounds, PDP Commodity Groupings, Method Validation and Quality Control*. This work would be guided towards providing recommendations to meet the objectives of the RCW 43.21A.735 relating to testing pesticides[14] in cannabis flower.

The USDA PDP-QC SOP affirms that the USDA holds authority to identify priority level for each commodity, consistent with the needs of all the data users/stakeholders (e.g., U.S. Environmental Protection Agency, U.S. Food and Drug Administration, grower groups, and others). Additionally, current tolerances, action levels, and national/international maximum residue levels would be considered and applied as determined appropriate by the USDA. Updates are provided in a memorandum to PDP data users, stakeholders, and States' testing programs.

Where there is a clear authority, process, and expertise in the USDA model, members felt that the WSLCB currently lacks the appropriate expertise to assume all the roles of the USDA in this and the other PDP SOPs. This further endorsed the idea of the need for a well-defined "Client". The group discussed the current authority and potential future authority and expertise desired specifically when it comes to oversite of analytical practices. Ultimately, the group felt the client authority should be clarified, or possibly redefined. An effective "Client" would need to be knowledgeable in agricultural and health sciences, pesticide policy and practices, and hold a high level of expertise in pesticide laboratory testing practices. For the purposes of future proofing the client, the client should also hold expertise in other analytical testing fields including microbiology and metals.

Further, the workgroup felt current WSLCB and DOH rules for pesticide testing were not clear as to what was required to be tested. The list of pesticides maintained on a DOH website created additional confusion. In order to follow the PDP SOP model for cannabis testing, a concise list of pesticide compounds must be clear to those that are required to test. Overall, to ensure that adaptation of the model would be successful, the workgroup felt that the Steering Committee needed to recognize:

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[13] The final summary of adaptations document was not conceptualized until a later meeting.
[14] The generic term "pesticides" is used to cover a suite of compounds that includes herbicides, fungicides, insecticides, insect growth regulators (IGRs), and synergists.

- Appropriate expertise and authority should be in place to cover the roles necessary to make decisions around pesticides as they pertain to testing and practices described the PDP SOPs.
- That WAC 314-55-108 does not contain language that states those listed in the table are the "required to test" compounds.
- The current two "lists" of compounds (WSLCB WAC 314-55-108) and (DOH website) are not harmonized, nor connected clearly.
- Analytically appropriate significant digits need to be expressed in the action levels presented in WAC 314-55-108.
- All pesticide compounds, including those on the list maintained on the DOH website, should include Chemical Abstract Service (CAS) numbers.
- Appropriate metabolites and isomers of the currently required pesticides should be reviewed and clarified. This would help ensure that all pesticide testing performed will generate the same type of information for each pesticide test.

Commodity Groups and Commodities

The USDA PDP-QC SOP introduces practices supporting testing samples from within pre-determined commodity groups. The PDP groupings are based on EPA commodity grouping under 40 CFR 180, with modifications to further combine those commodities having similar matrix characteristics for analytical purposes. As an example, the EPA sets up commodity groups, one such is "Citrus Fruit", where the commodities in the group would be satsuma mandarin, orange, lime, and kumquat. Testing is performed on the specific commodities, or representative commodity, within each group of analytically similar likeness. Examples of other EPA designated food and feed commodities include wheat, corn, dry beans, rice, tomatoes, apples, grapes, beef, poultry, eggs, timber, tobacco, and as of 2018[15], this list also includes hemp.

This design allows for different types of cannabis or cannabis products, with different analytically driven needs, to be treated separately. It is another recommendation that allows for future flexibility while recognizing the complexity of the matrix. For the purposes of adapting the PDP SOPs for use on cannabis, the workgroup suggested initially establishing the one commodity group as "cannabis flower" consisting of three commodities:

- High tetrahydrocannabinol (THC) cannabis flower
- High cannabidiol (CBD) cannabis flower
- High THC/high CBD hybrid cannabis flower

The three initial commodities determinations under "cannabis flower" were thought be sufficient to address the matrix complexities that exist in the cannabis flower samples received and tested.

[15] 2018 Farm Bill: https://www.farmers.gov/manage/hemp

Future commodity groups, such as one for "high purity cannabis concentrates", could be added to address the individual commodities such as isolates and distillates.

The recommendation of commodity group "cannabis flower" and three commodities were moved to the Steering Committee as a discussion item contained in the Analytical Workgroup First Report[16].

Side-issues

Several of the workgroup members brought forth issues that they hoped that the Task Force could help resolve. Many of those items brought forth, ultimately, were determined to reside outside of the Task Force legislative objectives. However, the nature of some outside issues do currently, and would continue to, affect the quality of data generated by cannabis testing laboratories. These items were noted and forwarded to the Steering Committee as side issues (Appendix E).

September 17, 2019

The second workgroup meeting continued with the workgroup reviewing line for the line the contents of the USDA PDP-QC SOP. Most of the sections the members worked through quickly and advised that no change or adaptation was necessary. Other sections were discussed at length due to the perceived practicality or practicability when applied to cannabis.

Storage Requirements and Freezer Temperatures

A considerable amount of discussion occurred around the USDA PDP requirements of storing all standards in a separate freezer from the freezer containing the samples. The workgroup chemists from the cannabis testing labs felt that the requirement of having two lab-grade freezers capable of reaching -20 ˚C or below was unreasonable, and further, too costly for the labs to implement.

For the question about the appropriate temperature, the WSDA workgroup lead, Mike Firman, discussed the rationale for the low temperature storage requirements. Stability studies, many that are cited within the USDA PDP-QC SOP, were referenced as evidence for the low temperature storage requirement. Adequate low temperature storage practices are necessary to prevent the pesticide compounds from volatilizing, degrading, or transforming. Additional dialogue continued around the fact that studies have shown that select pesticides are not even stable until they are held at -30 ˚C, -40 ˚C or even -80 ˚C. Maintaining the longevity and stability of the pesticide standards is necessary to accurately perform any analytical pesticide technique.

[16] https://www.ezview.wa.gov/Portals/_1962/Documents/CannabisSTF/AnalyticalWorkgroupFirstReport.pdf

For the storage separation practices, some cannabis laboratory chemists believed utilizing separate shelfs or placing standards in a container to isolate them would be an effective measure to prevent cross-contamination between samples and standards. The final storage requirement, that standards needed to be signed in and out of storage, was also discussed. The cannabis laboratory chemists all said that this was unnecessary and burdensome.

Each of the chemists from the agencies acknowledge the expense of low temperature lab-grade freezers, however each felt the reliability of the standards should not be put at risk in favor of a cost-saving route. Minus 30 ˚C was the highest possible temperature that was considered as suitable by the agency chemists. For standards and samples separation practices, and the signing in/out of standards, the agency chemists also noted that these storage practices are part of all regulatory pesticide testing programs. Preprinted sign-out sheets and established-traceable nomenclature to abbreviate long standard names would help ease the perceived time sink in implementing this practice.

The workgroup was not able to come consensus on the storage, freezer, and sign in/out requirements so this topic would be forwarded to the Steering Committee as a minor item. See final motions for October 18, 2019 (Motion #3) in Appendix F.

Calibrations, Process Controls and Matrix Spike Criteria

All members agreed that instrument calibration is a fundamental requirement for all analytical testing practice. Calibration consists of a series of standards of known concentrations used to calibrate the instrument for each method. Over time, every method/instrument eventually will fall out of calibration. Running check standards at an appropriate frequency helps to determine when the calibration falls outside of the method tolerance limits.

The USDA PDP SOP model requires that each batch of samples would require a full calibration at the start and a check standard at the end of each batch. For PDP SOP model, a batch is defined as up to 35 samples that are tested together. The laboratory members felt that the USDA calibration requirements were too frequent and that restricted batch sizes would interrupt their current workflow processes too significantly. With the exception of the DOH, each agency member felt that a robust practice of frequent calibrations and check standards was necessary for regulatory testing. The DOH proposed daily calibrations with only check standards run between batches and the WSLCB suggested the European Union's Health and Food Safety (SANTE) requirement of bracketing calibrations before and after samples. The laboratory members' preferences aligned with the DOH proposal. Due to the divide within group, the topic was moved to the Steering Committee for the final decision. See final motions for October 18, 2019 in Appendix F.

The workgroup did reach a consensus to go more restrictive than the USDA PDP criterion for the tolerance limits for matrix spikes and process controls. The USDA PDP limits of 50-150% were established for risk assessment specific to the USDA, where a greater level of uncertainty is acceptable. All members felt that that level of uncertainty was not applicable to regulatory threshold testing. The workgroup considered the SANTE quality control guidance specification of 70-120%, however the workgroup compromised with +/- 30%, or 70-130% to allow for the matrix complexities of cannabis flower.

Other minor recommendations, side issues, and general changes proposed for the cannabis-adapted PDP-QC SOP are summarized in the Analytical Workgroup First Report document[17].

October 2, 2019 and October 16, 2019

PDP-DATA- Data and instrumentation

The Analytical workgroup focused their next review on the USDA PDP-DATA SOP. Discussions continued around calibration, and moved on to criteria for data reporting and data review. The structure of the PDP-Data often drove the conversation to touch back on aspects discussed in earlier meetings, such as using SANTE limits, defining the action limit at or below the limit of quantitation (LOQ), and the roles and responsibilities of the client. The workgroup summarized the major and extensively detailed minor recommendations in the Analytical Workgroup Second Report[18]. This document was present to the Steering Committee on October 18, 2019. [The document's minor recommendations were later incorporated into the final Task Force *Summary of Adaptations to the PDP Model SOPs* document (Appendix A) or presented as motions (Appendix F)]

Clean Matrix Material

The PDP SOP model assumes that there is as abundance of matrix blank material. Pesticide-free "clean" matrix material is necessary to validate each pesticide method and to use for continual performance checks and testing batch quality control. While the USDA laboratories have access to pesticide-free peas, apples, and limes, the cannabis testing laboratories do not have a readily available clean source of matrix material (i.e., cannabis flower). For example, WSDA Chemical and Hop Laboratory staff are able to store excess sample from tested pesticide-free samples for use as matrix blanks. If they run out of their stock of clean material, staff can simply go to a grocery store and purchase organic products to use as matrix blanks. Once the products are verified as free of pesticides, staff are able to use and store material for later testing. Environmental and food laboratories also do not suffer from burdensome restrictions that limit them from acquiring most matrix materials. Most can be sourced from reference material (RM) producers or even within the lab, such as when using reagent water (blank) from a lab water purification system.

Often cannabis testing laboratories are provided with only the minimal amount of sample, so storing excess material is not an option. While the labs can purchase products already in the market, they would be able to purchase only one ounce of cannabis flower at a time. Clean matrix materials and RMs for cannabis flower (Δ-9-THC > 0.3%) are simply not available for sale to Washington State cannabis testing laboratories. This is due to the location of the RM producers being located outside of the state and the federal restrictions on transporting it across state lines. Intra-state transport of cannabis is also subject to WSLCB licensing and transport restrictions.

[17] https://www.ezview.wa.gov/Portals/_1962/Documents/CannabisSTF/AnalyticalWorkgroupFirstReport.pdf
[18] https://www.ezview.wa.gov/Portals/_1962/Documents/CannabisSTF/AnalyticalWorkgroupSecondReport.pdf

October 30, 2019

Quality Control Rules and Criteria

The workgroup wrapped up the PDP-DATA SOP by deliberating quality control (QC) rules and criteria. This topic garnered much discussion around batch QC and QC failures. Particularly with respect to how the cannabis laboratories currently treat or adjust for failures. Some of the current laboratory practices implemented do not follow a regimented quality design, rather the analysts' judgement prevailed. Most cases for brought forth for making QC adjustments, such as not reanalyzing samples when QC fails because no observable peak was viewed. Most described deviations would not be supported under the PDP SOPs. The PDP SOPs are designed to maintain the quality of the results generated be requiring appropriate QC and QC criteria, run at the appropriate frequency. The workgroup members all agreed that there might be edge cases, and that client expertise must be consulted. Additionally, it was noted that the client would hold the ultimate responsibility for making updates to QC rules and criteria in the PDP model.

PDP-LABOP SOP – Sample Processing and Analysis, and PDP-Glossary

The PDP-LABOP SOP covers the receipt, storage, archiving, and disposal of samples and sample portions, as well as the preparation of samples by commodity type. Most of the workgroup changes or adaptations to this SOP were minor, or touched back to earlier topics that elicited motions or discussions moved to the Steering Committee.

The workgroup determined that the PDP-Glossary should be included with the PDP SOP model to provide definitions and terminology used within the USDA PDP SOPs. This would be valuable for both the laboratories and the client.

The PDP-Admin SOP and the Role of the Quality Assurance Officer

The most significant discussion occurring during the review of the PDP-Admin was role of the Quality Assurance Officer (QAO). Within a laboratory, the QAO is ultimately responsible for ensuring that the laboratory implements and maintains quality assurance (QA) practices to achieve objectives of a laboratory quality system. Primary roles include reviewing and approving SOPs and data, maintaining data records and document control, conducting internal audits, maintaining the laboratory's QA Manual, and coordinating external PT and accreditation activities.

The USDA PDP SOP model is based on a system where the QAO in a testing laboratory must be separate individual from both laboratory technicians and laboratories scientific director, or Technical Program Manager (TPM), as defined by the USDA PDP. The role must remain independent to ensure the highest level of objectivity, as to not bias QA practices not consistent with the objective to generate sound scientific data, such as workload issues or monetary influences. It was recognized that it would be difficult for new and small labs to establish a completely independent QAO.

All the testing laboratory workgroup members felt that it was not necessary for this practice to be initiated for cannabis testing laboratories. However, all members did agree that if the QAO is not a separate and independent role, the QAO could not review their own work.

Ecology suggested that this position could be part-time or filled by a contracted individual that serves as QAO for more than one laboratory. This practice is currently implemented in several small laboratories that Ecology accredits for environmental testing throughout Washington State.

The workgroup was not able to come to consensus, with all testing labs and the DOH representatives feeling that the QAO could have additional duties, such as testing or serving as the scientific director. All members from WSLCB, WSDA, and Ecology felt the QAO should be a separate, independent position. Due to the discordance, determination of the QAO position requirements was forwarded to the Steering Committee as a motion. See final motions for November 15, 2019 (Motion #4), and December 16, 2020 (Motion # 3) in Appendix F.

November 13, 2019

Workgroup Deliverables

The conversation began with a discussion about the required workgroup deliverables and how to conclude the pesticide in flower workgroup objective. The workgroup wanted to ensure that all their hard work put into developing the new pesticides standards would be adopted and established in a usable form. They recognized that the summary of changes document would not be the most user-friendly document for laboratories to use as is. As the PDP model centers around the client establishing and maintaining the functioning of the PDP, the final format, as well as the authoring, editing, and publishing user-friendly SOP(s) or manual would be a task forwarded to the newly defined client. The workgroup would continue to focus on ensuring the science is valid and that the changes made are concise and as necessary for cannabis flower.

WAC Updates

The workgroup review Chapter 314-55 WAC for inconsistencies and areas that need to be updated to enable the effective use of this nearly complete legislative-mandated task. The workgroup had trepidation about suggesting rule updates and the timing of those update, as none of the workgroup members were policy experts. They were however able to point out some areas that should be considered for revision to support their significant scientific contributions and the overall Task Force efforts:

- In WAC-314-55-0995, the American Herbal Pharmacopeia (AHP) should be deleted, as it does not support concise, appropriate, or best practice science. Also, Chapter 314-55 WAC should include e requirement that samples should be tested on an “as is” “as received” basis to support the PDP model [Appendix F, 11/15/2019 Motion #3 shows the recommendation for the removal of the AHP, and addition of “as is”/”as received” testing, as adopted by the Task Force]

- WAC 314-55-101 3(a) should be updated to include a minimum of <u>two</u> 4 gram marijuana flower test samples must be collected. Each sample shall be collected following the existing collection requirement (e.g., four separate samples of not less than 1 gram each per five-pound lot. Appendix F, 11/15/2019 Motion #2 shows the recommendation for the removal of the AHP as adopted by the Task Force.

- The recommended Summary of Adaptions to the PDP Model SOPs and USDA PDP SOPs contains the criteria for, and to verify, acceptance. With WSLCB adoption of the PDP model

recommendation, this criterion should supersede that of the WAC 314-55-103 checklist for pesticide-testing practices used by the current accreditation provider.

  - Specifically, in WAC 314-55-103, the good laboratory practice checklist used by the current accreditation body holds some inconsistences when applied to the recommended PDP model (e.g., 0.990 vs 0.995 r^2 criteria for calibrations, in PDP-DATA 6.2.2 and WAC 314-55-103 30c., respectively).
  - The workgroup recognized that once Ecology takes over as the authority for accreditation, using Ecology's accreditation model, that WAC-314-55-103 must be rescinded. Ecology's accreditation style supports the use of the PDP (and method) established criteria.

- WAC 314-55-108 (3) language provides action limits for "all other pesticides" at 0.1 ppm, with 59 pesticides pulled out and placed in a table form (with higher thresholds). Nowhere does it state what pesticides must be tested. The WSLCB should clarify in rule or maintain a formal list or the priority "to test list" of pesticides. This is important to inform the laboratories, accreditation, and the client when making future decisions about methods and testing practices. It is highly suggested that WSLCB cross-reference the DOH priority list and produce one joint list.

- WAC 314-55-108 action levels should be updated to contain appropriate significant figures and define the required pesticide isomers to be tested and listed; e.g., update all action limits to include two significant figures, change table footnote c to only include pyrethrins 1 and 2, remove footnote b, and clarify spinosad to be spinosyn a and spinosyn d.

- There are some conflicts between WSLCB's Chapter 314-55 WAC for testing practices and DOH's WAC 246-73-50. As a general recommendation, the WSLCB and DOH should cross-reference and harmonize the information and requirements in their WACs.

Finally, the workgroup formalized their motions to move to the steering committee covering both previous meeting topics and the topics discussed during this meeting (Appendix F).

December 11, 2019

The final discussions of the workgroup focused on the drafted *Summary of Adaptations to the PDP Model SOPs* to enable delivery to the Task Force. The members revisited several topic and conversations and clarified content to be incorporated into the summary document (Appendix A).

Commodity Groups and Commodities

Most notably the workgroup revisited the topic of the cannabis specific commodity groups and commodities, as the initial introduction of the topic of designating commodity groups and commodities to the Steering Committee did not result in a final recommendation. The workgroup again discussed the interferences and analytical challenges exhibited by the various types of cannabis flower submitted to the labs. The members firmly agreed at a minimum, with cannabis flower as the commodity group, the three commodities should be 1) high THC cannabis flower, 2) high CBD cannabis flower, and 3) high THC/high CBD cannabis flower. The members established "high" as levels ≥ 10%. The 10% designation was thought to adequately demark a point that interferences are prevalent, and also be a level at which a source of matrix blank material could be easily acquired. The workgroup also recognized that that commodity groups and commodities periodically should be reviewed and updated moving forward. To address this need, the current

EPA responsibility for this would have to be taken over from by the new Washington State designated client. The Analytical Workgroup recommendations for the commodity group and commodities, and the additional defined client role would be forwarded to the Steering Committee as a motion. See final motions for December 16, 2019 (Motion #1) in Appendix F.

Defining Other Laboratory Roles

Other roles and responsibilities outlined in the PDP SOPs would require certified laboratories to establish internal positions. The workgroup had previously discussed the role of the QA officer (QAO), and Steering Committee had adopted a motion recommending that the QAO should be a separate potion that could not conduct testing on regulatory or PT samples. However, it was felt that the responsibilities of the QAO, and other roles cited in the PDP still needed further defining. The roles and responsibilities of the Technical Program Manager (TPM) and Administrative Manager were forwarded as a discussion point to the Steering Committee. See final motions for December 16, 2019 (Motion #4) in Appendix F.

January 17, 2019 (Task Force Public Meeting)

The Task Force assembled and adopted additional summary language stating that separate standard preparation area is not required if there are appropriate cleaning procedures and controls to ensure against cross contamination. The Task Force then adopted the final pesticide in plants task that would culminate the Task Forces mandated legislative objective. The final recommendation, as approved, was to 'adopt all changes to the model documents in the (Appendix B) document, "Summary of Adaptations to Model Documents". The final motions are exhibited in Appendix F (January 17, 2019).

February 12, 2019

The Analytical Workgroup convened for one final meeting to discuss providing adaptations the Task Force Task Force *Summary of Adaptations to the PDP Model SOPs* document and the five USDA PDP SOPs (Appendices A and B) to incorporate the compliant intermediate cannabis products.

Due to the flexibility of the PDP SOP model, only minimal edits were necessary to incorporate all of the products that are tested for pesticides into this Task Force approved PDP SOP model. The workgroup recommended that five new commodity groups be recognized to incorporate compliant intermediate cannabis products (Appendix F, February 20, 2020 Motion #1):

- Commodity Group 1: Includes One Commodity; Commodity: Hydrocarbon and CO2 cannabis products: examples include butane, propane, pentane, and heptane extracts; and CO2 wax.
- Commodity Group 2: Includes One Commodity; Commodity: Non-solvent cannabis products: examples include kief, hash, and rosin.
- Commodity Group 3: Includes One Commodity; Commodity: Food Grade and Ethanol cannabis products: examples include cannabis extracted with glycerin, propylene glycol, and ethanol.
- Commodity Group 4: Includes One Commodity; Commodity: Infused Oil cannabis products: examples include cannabis extracts infused into Medium Chain Triglycerides oil, butter, coconut oil, medium chain (C6-C12) oils, polyethylene glycol, glycerin, and propylene glycol.
- Commodity Group 5: High Purity cannabis products, Includes Two Commodities; Commodity

1: Distillates Commodity 2: Isolates

No other changes to the Task Force *Summary of Adaptations to the PDP Model SOPs* were deemed necessary to facilitate the use of this for testing compliant-intermediate cannabis products.

The workgroup decided that three other recommendations should be considered as Task Force recommendations:

- Ensure that the laboratories perform the appropriate validated testing method required for each of the defined commodity groups by requiring documentation detailing the ingredients, process to produce the same and final compositions to accompany every intermediate sample submitted for testing (Appendix F, February 20, 2020 Motion #2).
- The commodity lists should be reviewed and updated at least every two years (Appendix F, February 20, 2020 Motion 3).
- Updates to the sample requirements for intermediate products should be made. Samples submitted for testing should be a minimum of 2 grams and be provided in a single shoulder-less jar, or single centrifuge tube. The laboratories shall homogenize the sample prior to subsampling for testing. (Appendix F, February 20, 2020 Motion #4)

Appendix D. Analytical Workgroup '*Client*' Recommendation: Roles, Duties, and Responsibilities of the Client

Content abstracted from the Pesticide Workgroup presentation from the October 18, 2018 Steering Committee Meeting[19]. The full discussion can be heard by listening to the Steering Committee public meeting recording[20].

- The client is not who pays the money.
- The client is a technical expert.
- The client is State Government.
- The client has authority to approve and deny lab work.
- The client can reject results including retroactive rejection.
- The client has effective power to suspend laboratory testing.
- The client is liaison with various State Government bodies and private laboratories.
- The client understands the state requirements.
- The client ensures tests results are fit to purpose.
- The client communicates with data uses and laboratories. *The workgroup recommends a single point of contact.*
- The client interprets technical requirements.
- The client must be available to the make decisions in a timely manner on an ongoing basis.
- The client identifies issues/trends across all labs.
- The client is responsible for Washington States interests. The client provides flexibility.

The documents that the workgroup looked at all had a client identified with client roles. The workgroup recommends the use of USDA/AMS/PDP documents that have USDA/AMS/PDP as the client. This role would have to be reassigned to use the documents or they would have to be substantially changed.

So far the workgroup has identified several tasks that the client performs in the USDA documents. This list if from the first document reviews. PDP-QC. More will be found in future documents.

- Client sets list of compound to test.
- Client approves Special Methods.
- Client approves Standards.
- Client reviews and approves methods. Explains why methods do not meet requirements.
- Client Marginal Performing.
- Client desired differences with Commodity Groups. *This will be very useful with a challenging commodity such a cannabis.*
- Measurement Uncertainty.

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[19] *Supplemental Report on Quality Control and Defining the Client*: *Recommendation Two*
https://www.ezview.wa.gov/Portals/_1962/Documents/CannabisSTF/SupplementalReportQCCalibrationAndClient.pdf
Note: Only edits to format were made to enhance readability of this content.

[20] https://www.youtube.com/watch?v=VamTvhRJgHk

- Client determines Reporting requirements
- Client approves Method Validation.
- QA is reported to client and client reviews.
- Client determines result Coding.
- Client approves QA Ranges.
- Client is advised when Ok in Validation, poor in practice.
- Client approves Exceptions – Example 150% recovery but all “ND”

Appendix E. Side Issues

Side issues were raised in meeting discussions by workgroup members. The workgroup members asserted that these issues were substantial; however, these items did not fall under the scope of HB 2052 and were not addressed by the Task Force.

Sample requirements and sampling issues

The current WSLCB sample requirement of just one 4-gram sample for testing may be insufficient or unsuited for all the required testing that is to be performed on that sample. Specifically, for samples arriving for analysis of pesticides, the entire sample should be homogenized and split to ensure a representative subsample will be tested. However, many mixing and sample splitting techniques that would be most appropriate for pesticide sample preparation may consequently contaminate the sample for microbiological testing. While the labs can request additional samples, some growers and processors are reluctant to part with more of their marketable product. The workgroup felt that due to the analytically driven need for two samples, the requirement should come from rule rather than leaving it up to the individual labs to handle.

Additionally, many workgroup members felt that fraudulent activities, inadvertent sampling error, and non-randomized sampling will continue to produce altered or biased samples if additional regulatory sampling controls are not put in place. Further, this problem will undermine any good scientific practices the Task Force recommends for testing pesticides in cannabis flower. Using appropriate methods and practices, the laboratories would be producing results showing cannabis products that appear to meet the regulatory requirements, but in actuality, the sample tested may not be representative of the actual product going to market. A change in the WSLCB rule around who can sample and how sampling is performed were offered as options. Another option would be to establish a mechanism to investigate fraudulent activities both at sampling and in the labs. The EPA uses their Office of Inspector General[21] to perform investigations of instances of intention misrepresentation, intent to deceive (usually for monetary gain), lying, cheating, and stealing.

Moisture content and dry weight correction

The treatment of the moisture content or "loss on drying" performed on samples is, in most cases, inappropriate. It becomes problematic where sample drying occurs before testing and when correcting final testing results for moisture content, leading to samples appearing to meet the regulatory requirements, when they may in fact be falsely corrected in a manner to pass the regulatory requirements. [This topic was later resolved for pesticides, as the procedures adopted mandates "as is" or "as received" testing. Treatment of moisture may still be problematic if not resolved for other lab testing, e.g., potency]

Cannabis flower matrix blank

A reoccurring challenge for the laboratories is acquiring enough material to use as a matrix blank. The PDP SOP model assumes that there is as abundance of available matrix blank material.

[21] https://www.epa.gov/office-inspector-general

Pesticide-free matrix "clean" material is necessary for validating each pesticide method, and for use in continual performance check standards. For cannabis flower, the labs have two sources 1) left over sample material that was determined to be free of pesticides, and 2) purchasing cannabis flower from WSLCB-licensed cannabis retail stores at the personal-use quantity of one ounce. The laboratories need a stable source and quantity of matrix blank to perform required regulatory testing.

Required pesticides: "to test list"

Lists maintained in the WSLCB WAC Chapter 314-55 for action levels are inconsistent with those provided on the DOH website. Further, adding the chemical unique identifying Chemical Abstract Service (CAS) numbers to the DOH website list would clarify the appropriate pesticide compound, or compound mixture, or compound plus metabolites mixtures, as pesticides common names may not adequately differentiate (e.g., pyrethrins).

Reporting to WSLCB database

The current data reporting system does not accept common non-numeric codes that are widely used in USDA, EPA, and other data systems containing regulatory chemistry data. This forces laboratories to potentially report inaccurate values. For example, when pesticide method detection limits are determined analytically, and are less than the LOQ or LOD, results could be reported inaccurately.

Appendix F. Steering Committee Final Motions[22]

October 18, 2019

MOTION #1:
Adopt the Analytical Workgroups recommendation for a performance set of standards that any method, instrument, or detection method must meet rather than a specific method.

MOTION #2:
Make a request from the Steering Committee to the Washington State Department of Agriculture for a representative on the Proficiency Workgroup.

MOTION #3:
Maintain the current United States Department of Agriculture standards for freezers and traceability and have the Analytical Workgroup put details into their document for final approval.

MOTION #4:
Adopt as a minimum for the Analytical Workgroup, the United States Department of Agriculture Pesticide Data Program standards for calibrations.

November 15, 2019

MOTION #1:
Adopt the current versions of USDA SOPs[23] as a model with summary of adaptations to be provided later.

MOTION #2:
2a. Establish a two-sample requirement for sampling unless otherwise specified by the client. Labs will use a random process to match samples with tests.
2b. Each sample must individually meet the WSLCB sampling requirements (WAC 315-55-101).
2c. Certified labs must reject samples that do not meet sample requirements.

MOTION #3:
This motion would adopt a requirement that samples must be tested on an as/is, as/received basis and would remove American Herbal Pharmacopeia from references in WAC 314-55.

MOTION #4:
Certified labs are required to use a QA officer who should be a separate position that would not conduct testing on client or PT samples.

[22] https://www.ezview.wa.gov/site/alias__1962/37551/cannabis_science_task_force.aspx
[23] The USDA PDP SOPs are frequently updated by USDA. Future updates may not be consistent with the adaptions adopted by the Task Force, for this reason the current versions, as of November 15, 2019 are to be used as a static reference the cannabis testing laboratories. These documents are provided in Appendix B.

December 16, 2019

MOTION #1:
1A: Motion to initially designate Cannabis Flower as the Commodity Group and the cannabis flower as an individual Commodity.
1B: Motion to establish that the client will replace the EPA as the entity responsible for defining commodity groups and commodities.

MOTION #2:
2A. Motion to require certified laboratories to establish a role functioning as a Technical Program Manager (TPM) to support the adopted USDA PDP SOP model (CSTF Motion #1 11/16/2019). The TPM has the overall responsibility for the technical conduct of the PDP testing contracted to the laboratory, as well as for the interpretation, analysis, documentation, reporting of results, and others required by the adopted PDP model SOPs or client. These duties may be added to an existing qualifying position already established within a certified laboratory upon approval from the client.
2B: Motion to require certified laboratories to establish a role functioning as an Administration Manager to support the adopted USDA PDP SOP model (CSTF Motion #1, 11/16/2019). The duties of this role include: laboratory management, budgeting, contracting, purchasing, inventory maintenance, and or client. These duties may be added to an existing qualifying position already established within a certified laboratory upon approval from the client.

MOTION 3:
Motion to further define the role and functions of the Quality Assurance (QA) Officer (CSTF Motion #4, 11/16/2019), or QA unit (QAU). The QA officer or QAU shall perform and provide, at a minimum: data review, transmission, internal audits, proficiency testing oversight, reports preparation (e.g. method validation, corrective action summaries, SOP review), and others as required by the adopted PDP model SOPs or client.

January 17, 2020

MOTION #1:
Adopt a proposal to add language to the USDA PDP-QC to add language to 5.2.4 and 5.2.5 that says, “A separate standard preparation area is not required if there are appropriate cleaning procedures and controls to ensure against cross contamination.”

MOTION #2: ADOPTED
Adopt all changes to the model documents in the attached document, “Summary of Adaptations to Model Documents”.

February 20, 2020

MOTION #1:
Motion to add 5 new commodity groups as stated below:

- Commodity Group 1: Includes One Commodity Commodity: Hydrocarbon and CO2 cannabis products: examples include butane, propane, pentane, and heptanes extracts; and CO2 wax.
- Commodity Group 2: Includes One Commodity Commodity: Non-solvent cannabis products: examples include kief, hash, and rosin.

- Commodity Group 3: Includes One Commodity Commodity: Food Grade and Ethanol cannabis products: examples include cannabis extracted with glycerin, propylene glycol, and ethanol.
- Commodity Group 4: Includes One Commodity Commodity: Infused Oil cannabis products: examples include cannabis extracts infused into Medium Chain Triglycerides oil, butter, coconut oil, medium chain (C6-C12) oils, polyethylene glycol, glycerin, and propylene glycol.
- Commodity Group 5: High Purity cannabis products, Includes Two Commodities Commodity 1: Distillates Commodity 2: Isolates Thursday, February 20, 2020

MOTION #2:
Each sample will only be accepted by the lab if it includes proper documentation. Documentation must include, at a minimum, the following information: 1. A list of ingredients used to produce the sample (for example butane, MCT oil, and so on) 2. The process or processes used to produce the sample (for example distillation, cold press, …) 3. The expected composition of the sample (for example MCT oil) Only scientifically defined common names may be used. “Butane” is allowed, but trade names “x-23 oil”, ambiguous names “Rick Simpson Oil” are not.

MOTION #3:
The client shall update the commodity lists at least every two years.