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

SOP No.: PDP-Glossary	Page 1 of 14	
Title: Glossary		
Revision: 11	Replaces: 01/01/2015	Effective: 04/02/2020

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 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. If a compound is reclassified as a Marginal Performing Analyte following Method Validation, 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.

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: Method 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.

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:

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

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.

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 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.

Unvalidated Compounds: These are compounds that are not designated as validated or marginally performing following the completion of method validation. These can include, but are not limited to, compounds that exhibited unacceptable recoveries during method validation, compounds that the laboratory has previously validated but no longer includes in their screens, compounds validated in other commodities, or a compound the laboratory is in the process of validating. Compounds that are not designated as validated or marginal performing for a commodity are considered unvalidated and identified as such in the method validation letter of concurrence issued by MPD.

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.

SITRA ABUBEKER Digitally signed by SITRA ABUBEKER
Date: 2020.04.02 11:31:25 -04'00'

Revised By: Sitra Abubeker Date
Monitoring Programs Division
1400 Independence Ave, SW
Washington, DC 20250
(202) 572-8175

Original signed by H.Robinson *4/6/2020*

Reviewed By: Hugh Robinson Date
Member of PDP Technical Advisory Group
Texas Department of Agriculture
1500 Research Parkway Suite B100
College Station, Texas 77845
(979) 458-4213

BRENDA FOOS Digitally signed by BRENDA FOOS
Date: 2020.04.02 12:18:30 -04'00'

Approved By: Brenda Foos Date
Monitoring Programs Division Director
1400 Independence Ave, SW
Washington, DC 20250
(202) 572-8167

Revision 11 April 2020 Monitoring Programs Division

- Removed PDP LABOP revision number for homogenate definition
- Updated Marginal Performing Analyte reclassification requirement during continuing quality control
- Updated the primary identification technique definition
- Removed definitions for Program Administrative Director and Technical Director
- Updated the Relative Standard Deviation definition by changing %RPD to %RSD
- Added definition for unvalidated compounds

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