

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

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Title: Sample Processing and Analysis		
Revision: 8	Replaces: 07/01/2015	Effective: 07/01/2016

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:

- 5. Sample Processing, Storage, and Disposal
 - 5.1 Sample Receipt
 - 5.1.1 Sample Inspection at Receipt
 - 5.1.2 Prepared Fresh Commodity Acceptability
 - 5.1.3 Sample Containers
 - 5.1.4 Damaged Animal Tissue
 - 5.1.5 Sample Weight Acceptability Criteria
 - 5.1.6 Sample Viability
 - 5.1.7 Documentation for Samples Not Analyzed
 - 5.1.8 Missing/Late/Unacceptable Samples or eSIFs
 - 5.1.9 Unresolved Sampling Issues
 - 5.1.10 Paper SIFs
 - 5.1.11 Unique Laboratory Sample ID
 - 5.1.12 Unit Counting
 - 5.1.13 Sample Receipt Log
 - 5.2 Sample Storage Prior to Homogenization
 - 5.3 Preparation and Homogenization of Fresh Produce, Animal Tissue, Nuts, and Grains
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- 5.4 Preparation and Homogenization of Processed Commodities
- 5.5 Weighing of Analytical Portion
- 5.6 Transshipment of Homogenate Subsamples
- 5.7 Storage of Homogenate Subsamples
- 5.8 Storage of Extracts
- 5.9 Disposal of Reserve Samples
- 5.10 Disposal of Extracts

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, Washington 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

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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

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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 duplicate 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.

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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.

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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 $\pm 20\%$ 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.*

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 In order for a sample to be considered viable, a minimum of 50% 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.



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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

5.1.9 Unresolved Sampling Issues

The receiving laboratory shall notify MPD of any continuing unresolved sampling issues on a monthly basis

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

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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 in order 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

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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

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Commodity		PDP Code	SOP Preparation and Homogenization Section
Baby foods	Applesauce	IA	5.4.5 (Other Processed, Semi-processed, Packaged)
	Carrots	IC	
	Green Beans	IG	
Baby foods	Peaches	IH	5.4.5 (Other Processed, Semi-processed, Packaged)
	Pears	IP	
	Peas	IE	
	Sweet potatoes	IS	
Bananas		BN	5.3.6
Barley		BY	5.3.20 (Grains)
Beans	Canned	Black	5.4.1 (Canned Commodities)
		Garbanzo (Chick pea)	
		Kidney	
		Pinto	
	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)

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Commodity		PDP Code	SOP Preparation and Homogenization Section
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	CA	5.4.2 (Frozen Commodities)
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	5.3.30
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	

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Commodity		PDP Code	SOP Preparation and Homogenization Section
Lettuce	Head	LT	5.3.24
	Leaf	LT	5.3.25
	Bagged	LB	5.4.5 (Other Processed, Semi-processed, Packaged)
Mangoes		MA	5.3.26
Milk, whole		MK	5.4.5 (Other Processed, Semi-processed, Packaged)
Mushrooms		MU	5.3.27
Nectarines		NE	5.3.32
Oats		OA	5.3.20 (Grains)
Olives	Canned	OL	5.4.1 (Canned Commodities)
Onions	Bulb	ON	5.3.28
	Green	GO	5.3.29
Orange	Fruit	OG	5.3.30
	Juice	OJ	5.4.3 (Juices/Concentrates)
Papaya		YA	5.3.31
Peaches		PC	5.3.32
Peanut Butter		PB	5.4.5 (Other Processed, Semi-processed, Packaged)
Pears	Fruit	PE	5.3.33
	Juice	PJ	5.4.3 (Juices/Concentrates)
	Canned	CP	5.4.1 (Canned Commodities)
Peas	Green	PS	5.3.34
	Snap	SN	5.3.35
Peppers	Bell, Sweet	PP	5.3.36
	Hot	HP	5.3.37



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Commodity		PDP Code	SOP Preparation and Homogenization Section
Pineapples	Fresh	PN	5.3.38
	Canned	NC	5.4.1 (Canned Commodities)
Plums		PU	5.3.39
Potatoes	Fresh	PO	5.3.40
	Frozen	PZ*	5.4.2 (Frozen Commodities)
	Sweet	SW	5.3.41
Raisins		RA	5.4.4.1
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	5.3.42
	Canned	SC	5.4.1 (Canned Commodities)
	Frozen	SP	5.4.2 (Frozen Commodities)
Squash	Summer	SS	5.3.43
	Winter	WS	5.3.44
	Winter, frozen	WZ	5.4.2 (Frozen Commodities)
Strawberries	Fresh	ST	5.3.45
	Frozen	SZ*	5.4.2 (Frozen Commodities)
Tangerines		TA	5.3.30
Tomatoes	Cherry	CT	5.3.46
	Fresh	TO	5.3.47
	Canned	TC	5.4.1 (Canned Commodities)
	Paste	TP	5.4.5 (Other Processed, Semi-processed, Packaged)
Watermelon		WM	5.3.48

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Commodity		PDP Code	SOP Preparation and Homogenization Section
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.

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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

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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

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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

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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

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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 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 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.

5.3.25 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.

5.3.26 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

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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.

5.3.27 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.

5.3.28 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.

5.3.29 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.

5.3.30 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.

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5.3.31 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.

5.3.32 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.

5.3.33 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.

5.3.34 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.

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5.3.35 Peas, Snap

For each sample, remove the end caps, 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. 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 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.

5.3.37 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.

5.3.38 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.

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5.3.39 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.

5.3.40 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.

5.3.41 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.

5.3.42 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.

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5.3.43 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.

5.3.44 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.

5.3.45 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.

5.3.46 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.

5.3.47 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

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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.

5.3.48 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.

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5.4.3 Juices/Concentrates

For fresh and reconstituted juices, ensure that sample is evenly mixed in order 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

5.4.4.1 Raisins

Open all of the raisin 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.

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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 sample is evenly mixed in order 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.

5.5 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.

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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 PDP headquarters. 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.

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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-volatile 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. Disposal shall be documented (e.g., in the freezer log or sample log) and shall contain at minimum date of disposal, sample number, and initials of the individual who discarded the sample.

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 a minimum of 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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Sitra Abubeker

06/30/16

Revised By: Sitra Abubeker

Date

PDP Chemist

Monitoring Programs Division

1400 Independence Ave, SW

Washington, DC 20250

(202) 572-8175

Sarvamangala Gunjur

07/01/16

Reviewed By: Sarvamangala Gunjur

Date

Presiding Member of PDP Technical Advisory Group

California Department of Food and Agriculture

Division of Inspection Services

Chemistry Laboratory

3292 Meadowview Road

Sacramento, CA 95832

(916) 228-6857

Diana Haynes

06/30/16

Approved By: Diana Haynes

Date

PDP Acting Technical Director

Monitoring Programs Division

1400 Independence Ave, SW

Washington, DC 20250

(202) 572-8167

Diana Haynes

06/30/16

Approved By: Diana Haynes

Date

PDP Program Administrative Director

Monitoring Programs Division

1400 Independence Ave, SW

Washington, DC 20250

(202) 572-8167

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- 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
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