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1. Purpose:

To provide standard procedures for:

- the receipt, storage, archival, and disposal of MPD Pesticide Data Program (PDP) samples and sample portions
- the preparation of MPD PDP samples
- the handling of 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
- 5.4 Preparation and Homogenization of Processed Commodities
- 5.5 Weighing of Analytical Portion

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- 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 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 https://ntp.niehs.nih.gov/iccvam/suppdocs/feddocs/epa/epa_glp40_160.pdf
- U.S. EPA, Maintenance and calibration of equipment, 40 CFR 160.63 https://ntp.niehs.nih.gov/iccvam/suppdocs/feddocs/epa/epa_glp40_160.pdf
- U.S. EPA, Standard operating procedures, 40 CFR 160.81 https://ntp.niehs.nih.gov/iccvam/suppdocs/feddocs/epa/epa_glp40_160.pdf
- U.S. FDA, Final Preparation Procedures, Standard Operating Procedure for the Total Diet Study KCX-1, Appendix E, January 19, 1993
- 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

Note: References consulted in the preparation of this SOP document.

5. <u>Sample Processing, Storage, and Disposal:</u>

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 Monitoring Programs Division (MPD) SOPs and the laboratories' internal SOPs and work instructions will be used as the measure

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of compliance in the event of an MPD 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	<u>5.1.5</u>	Required if there are problems
Sample unable to be analyzed by lab	Specify reason sample not analyzed	Reason NOT Analyzed	<u>5.1.4</u> <u>5.1.7</u>	Required if not analyzed
Received sample	Record date sample received	Date/Time Received	<u>5.1.1</u>	Required
Received sample	Record person that received the sample	Received By	<u>5.1.1</u>	Required
Sample weight received	Record weight of sample received	Sample Size	<u>5.1.5</u>	Required

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

Note: FGIS grain sample collection is completed by FGIS and sample information is imported to the laboratory RDE tool. Additional information on FGIS sampling procedures is documented in each statement of work. Contact the PDP Sampling Team (pdp.sampling@usda.gov) for additional information on these procedures.

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 PDP 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 label match each other. This may be entered manually 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 sample identification label information is retained (e.g., attaching to printed electronic SIFs (eSIFs), in a sample receipt logbook, etc.).

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5.1.2 Prepared Fresh Commodity Acceptability

Prepared fresh product (e.g., snipped green beans, chopped packaged lettuce) is acceptable if the commodity requirements on the Commodity 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 PDP Sampling Team (pdp.sampling@usda.gov) 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 (e.g., 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 PDP Sampling Team (pdp.sampling@usda.gov) to determine if analysis should be performed or if the sample should be re-collected.

5.1.4 Damaged Animal Tissue

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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 five pounds. The acceptable weight range is \pm 20% of the target weight (e.g., for 5 lb. samples: 4-6 lbs). The sample weight specific to each commodity is found in the Commodity Fact Sheet.

Note: Determine the sample weight at the time of sample receipt and enter the weight 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 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 from the large sample to reach the targeted weight (\pm 20%) 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 the sample weight being homogenized in the "Analytical Size" field of RDE as well as 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

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portions (e.g., wilted, mushy, moldy, etc.) are discarded (e.g., anything less than 3.5 lbs for a 5 lb sample is not viable for analysis).

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. The time stamp portion can be disregarded when determining matching lot numbers. 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. All unanalyzed samples should have the "Reason NOT Analyzed" field completed in RDE.

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 and the PDP Sampling Team (pdp.sampling@usda.gov) to arrange for recollection. 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@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@usda.gov or the PDP Sampling Team at pdp.sampling@usda.gov.
- **5.1.8.4** If an RDE SIF arrives for a non-collected sample, the SIF shall be attached

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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 samples were not collected.

5.1.9 Unresolved Sampling Issues

The receiving laboratory shall notify the PDP Sampling Team (<u>pdp.sampling@usda.gov</u>) of any continuing unresolved sampling issues monthly.

5.1.10 Paper SIFs

If a paper SIF is received, the laboratory shall scan and email to MPD (amsmpo.data@usda.gov) within 24 hours. Grain samples collected by FGIS are exempt from this SIF requirement.

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, a determination of whether 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 of homogenized units 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

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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 is perishable, from the time of arrival until the sample is homogenized. Section <u>5.3</u> of this SOP lists the maximum holding time prior to homogenization for each PDP commodity.
- **5.2.4** Processed 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 for a period not to exceed 120 hours from the time of arrival until the sample is homogenized. Processed commodities that require refrigeration (e.g. dairy, refrigerated juices, eggs, etc.), may not exceed 72 hours refrigeration from the arrival time 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 and the PDP Sampling Team shall be contacted at PDP.sampling@udsa.gov to determine sample viability. If the sample is determined to be viable, 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) may be

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acceptable.

5.2.7 Grain commodities shall be refrigerated for a period not to exceed 120 hours from the arrival time until the sample is homogenized.

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 ensure 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 $\underline{5.1.5.2}$ and $\underline{5.1.6}$ of this SOP.

Commodity		PDP Code	SOP Preparation and Homogenization Section
Almonds		AL	<u>5.3.1</u>
	Fruit	AP	<u>5.3.3</u>
Apples	Juice	AJ	(Juices/Concentrates)
Apples	Sauce	AC	5.4.5 (Other Processed, Semi- processed, Packaged)
Asparagus		AS	5.3.4
Avocados		AV	<u>5.3.5</u>
	Applesauce	IA	
Baby foods	Carrots	IC	<u>5.4.5</u> (Other
	Green Beans	IG	Processed, Semi- processed, Packaged)
	Peaches	IH	processed, rackaged)
	Pears	IP	

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Commo	dity		PDP Code	SOP Preparation and Homogenization Section
		Peas	IE	
		Sweet potatoes	IS	
Bananas			BN	<u>5.3.6</u>
Basil			BS	<u>5.3.16</u>
Barley			BY	<u>5.3.20</u> (Grains)
	Canned	Black Kidney Pinto	ВС	5.4.1 (Canned Commodities)
Beans	Callifed	Garbanzo (Chickpea)	ZB	Commodities)
	Dried	Garbanzo, dried (Chickpea)	ZD	5.4.5.6
	Green		GB	<u>5.3.7</u>
Beets, ca	nned		ВТ	5.4.1 (Canned Commodities)
		Fresh	BK	<u>5.3.8</u>
Blackber	rry	Frozen	KZ*	5.4.2 (Frozen Commodities)
		Fresh	BB	<u>5.3.8</u>
Blueberry		Frozen	BZ*	5.4.2 (Frozen Commodities)
Broccoli		BR	<u>5.3.9</u>	
Butter			BU	5.4.5 (Other Processed, Semi- processed, Packaged)
Cabbage			CG	<u>5.3.10</u>
Cantaloupe		CN	<u>5.3.11</u>	

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Commodity		PDP Code	SOP Preparation and Homogenization Section
Carrots		CR	<u>5.3.12</u>
Cauliflower		CF	<u>5.3.13</u>
Celery		CE	<u>5.3.14</u>
	Fresh	СН	<u>5.3.15</u>
Cherries, Sweet	Frozen	CZ*	5.4.2 (Frozen Commodities)
Cilantro		CL	<u>5.3.16</u>
	Grain	CO	<u>5.3.20</u> (Grains)
	Sweet, Fresh	СВ	<u>5.3.17</u>
Corn	Sweet, Frozen	CS	5.4.2 (Frozen Commodities)
Com	Sweet, Canned	CD	5.4.1 (Canned Commodities)
	Syrup	CY	5.4.5 (Other Processed, Semi- processed, Packaged)
	Fresh	CA	<u>5.3.8</u>
Cranberry	Frozen	AZ*	5.4.2 (Frozen Commodities)
Canned		RC	5.4.1 (Canned Commodities)
Cream, heavy		CM	5.4.5 (Other Processed, Semi- processed, Packaged)
Cucumbers		CU	5.3.18
Egg		EG	<u>5.4.5.5</u>
Eggplant		EP	<u>5.3.19</u>
Fish	Catfish	FC	<u>5.3.2</u> (Animal

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Commodity		PDP Code	SOP Preparation and Homogenization Section
	Salmon	FS	Tissue-Fish)
Grapefruit		GF	<u>5.3.31</u>
	Fruit	GR	<u>5.3.21</u>
Grapes	Juice	GJ	(Juices/Concentrates)
	Collard	GL	
Greens	Kale	GK	<u>5.3.22</u>
	Mustard	MG	
Honey		НҮ	5.4.5 (Other Processed, Semi- processed, Packaged)
Honey Dew Melon		HD	<u>5.3.23</u>
Y 0 . 0 . 1	Dairy-based	DF	5 4 5 4
Infant formula	Soy-based	YF	5.4.5.4
Kiwi		KW	<u>5.3.24</u>
	Head	LH/LT	5.3.25
	Leaf	LL/LT	<u>5.3.26</u>
Lettuce	Bagged	LB	5.4.5 (Other Processed, Semi- processed, Packaged)
Mangoes		MA	<u>5.3.27</u>
Milk, whole		MK	5.4.5 (Other Processed, Semi- processed, Packaged)
Mushrooms		MU	<u>5.3.28</u>
Nectarines		NE	<u>5.3.33</u>

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Commodity		PDP Code	SOP Preparation and Homogenization Section
Oats		OA	<u>5.3.20</u> (Grains)
Olives	Canned	OL	5.4.1 (Canned Commodities)
Oniona	Bulb	ON	5.3.29
Onions	Green	GO	<u>5.3.30</u>
	Fruit	OG	<u>5.3.31</u>
Orange	Juice	OJ	5.4.3 (Juices/Concentrates)
Papaya		YA	5.3.32
	Fruit	PC	<u>5.3.33</u>
Peaches	Canned	CC	5.4.1 (Canned Commodities)
	Frozen	PZ*	5.4.2 (Frozen Commodities)
Peanut Butter	'	РВ	5.4.5 (Other Processed, Semi- processed, Packaged)
	Fruit	PE	<u>5.3.34</u>
Pears	Juice	РЈ	(Juices/Concentrates)
	Canned	СР	5.4.1 (Canned Commodities)
	Green, Fresh	SE	<u>5.3.35</u>
Peas	Green, Frozen	PS	5.4.2 (Frozen Commodities)

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Commodity		PDP Code	SOP Preparation and Homogenization Section
	Green, Canned	SD	5.4.1 (Canned Commodities)
	Snap	SN	5.3.36
Damaga	Bell, Sweet	PP	<u>5.3.37</u>
Peppers	Hot	HP	5.3.38
	Fresh	PN	5.3.39
Pineapples	Canned	NC	5.4.1 (Canned Commodities)
Plums	Fresh	PU	5.3.40
Plums	Dried	PD	5.4.4 (Dried Fruits)
Potatoes	Fresh	PO	<u>5.3.41</u>
	Frozen	PZ*	5.4.2 (Frozen Commodities)
	Sweet	SW	<u>5.3.42</u>
Pumpkin	Canned	PK	5.4.1 (Canned Commodities)
Radish		RD	<u>5.3.43</u>
Raisins		RA	<u>5.4.4.1</u>
	Fresh	RS	<u>5.3.8</u>
Raspberries	Frozen	RZ*	5.4.2 (Frozen Commodities)
Rice		RI	<u>5.3.20</u> (Grains)
Soybeans, Grain		SY	<u>5.3.20</u> (Grains)
	Leafy	SP	5.3.44
Spinach	Canned	SC	5.4.1 (Canned Commodities)

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Commodity		PDP Code	SOP Preparation and Homogenization Section
	Frozen	SF	5.4.2 (Frozen Commodities)
	Summer	SS	<u>5.3.45</u>
Squash	Winter	WS	<u>5.3.46</u>
Squasii	Winter, frozen	WZ	5.4.2 (Frozen Commodities)
	Fresh	ST	5.3.47
Strawberries	Frozen	SZ*	5.4.2 (Frozen Commodities)
Tangerines		TA	<u>5.3.31</u>
Tomatillo		TT	_5.3.48_
	Cherry	CT	<u>5.3.49</u>
	Fresh	ТО	<u>5.3.50</u>
Tomatoes	Canned	TC	5.4.1 (Canned Commodities)
	Paste	TP	5.4.5 (Other Processed, Semi- processed, Packaged)
Watermelon		WM	_5.3.51_
	Grain	WH	_ <u>5.3.20</u> (Grains)
Wheat	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.

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Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

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 ensure 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. Alternatively, use a clean, dry knife to 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 stems, if inedible. Wash asparagus spears under cold running tap water for approximately 15-20 seconds to ensure 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.

5.3.5 Avocados

If necessary, avocado samples may be stored in a secure location at room temperature for 72 hours for ripening purposes. Wash avocados under cold running tap water for approximately 15-20 seconds to ensure 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. If avocado samples are too unripe for proper preparation, additional

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hours at room temperature to ripen are permissible. However, the samples should be checked daily and processed as soon as they are ripe. Samples can be stored in paper bags to assist with ripening.

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. To accelerate ripening, the bananas can be stored in a paper bag. 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 ensure 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/Blackberries

Wash blueberries/cranberries/raspberries/blackberries by the handful or by using a colander under cold running tap water for approximately 15-20 seconds to ensure 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. If the stem is less than 3 inches, do not trim. If the stem is longer than 3 inches, thinly slice away the tough outer layer on the stalk. Wash the sample under cold running tap water for approximately 15-20 seconds to ensure 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

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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 ensure 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

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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 ensure 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 ensure 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\Basil

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 ensure 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 ensure 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

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sample is homogenized.

5.3.18 Cucumbers

Wash each cucumber under cold running tap water for approximately 15-20 seconds to ensure 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 ensure 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

The grain sample should be inspected prior to processing and large debris (i.e., stones, sticks, large pieces of dirt, etc.) should be removed. The entire grain sample received from the Federal Grain Inspection Service (i.e., corn, wheat, soybean, barley) is to be homogenized, while for other grains (i.e., oats, rice) the entire sample is poured into a container and mixed thoroughly to obtain a 500 gram sub-sample for homogenization. Grind the sample using an appropriate device (e.g., Falling 3300 laboratory mill, Jacobsen grinder, UDY grinder). Tumble the resulting powder homogenate to obtain a homogeneous mixture. Unit counting is not required. Refrigeration may not exceed 120 hours from the arrival time until the sample is homogenized.

5.3.21 Grapes

Wash each sample under cold running tap water for approximately 15-20 seconds to ensure 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

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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 ensure 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 ensure 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.

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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 ensure 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. People with sensitive skin should handle mangoes with gloves. Wash each mango under cold running tap water for approximately 15-20 seconds to ensure 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 ensure 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

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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 ensure 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 hours for ripening purposes. Wash each papaya under cold running tap water for approximately 15-20 seconds to ensure 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. 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 ensure that all surfaces of the peach/nectarine 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

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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 ensure 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 ensure 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 ensure 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 ensure

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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 sample under cold running tap water for approximately 15-20 seconds to ensure that all surfaces are rinsed. Allow to drain for at least 2 minutes. Using a clean, dry knife, remove stem. 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 ensure 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 ensure 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 water for approximately 15-20 seconds to ensure that all surfaces of the

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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.

Due to the widespread use of chlorpropham in potatoes combined with the high concentrations present and the chemical nature of the compound, care must be taken to prevent laboratory cross-contamination. If multiple commodities are being processed, the potatoes should be held for last. All equipment, including cutting boards, knives, bowls, etc., should be solvent washed after potato contact. The use of separate grinding bowls is recommended.

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 water for approximately 15-20 seconds to ensure 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 Radish

Hold each radish under cold running tap water and remove any loose soil and grit (discard the leaf portion). Rinse each radish under cold running tap water for approximately 15-20 seconds to ensure that all surfaces are rinsed. Allow to drain for at least 2 minutes.

Note: Bagged pre-washed radishes do not require washing by the laboratory.

If there are tops or roots present, remove with a clean, dry knife. 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.

5.3.44 Spinach

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 ensure 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.

5.3.45 Squash, Summer

Wash each squash under cold running tap water for approximately 15-20 seconds to ensure 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.

5.3.46 Squash, Winter

Wash each squash under cold running tap water for approximately 15-20 seconds to ensure that all surfaces are rinsed. Allow to drain for at least 2 minutes. 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.

5.3.47 Strawberries

Wash strawberries by the handful or by using a colander under cold running tap water for approximately 15-20 seconds to ensure 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.

5.3.48 Tomatillos

Remove the husk, if present. After the husks are removed, wash tomatillos under cold running tap water for approximately 15-20 seconds to ensure that all surfaces of the

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tomatillos are rinsed, and the sticky residue is removed. Allow to drain for at least 2 minutes. Do not peel. Using a clean, dry knife, cut the tomatillo around the stem area. Remove any stem, being careful to remove as little of the meat as possible. The tomatillos 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 Tomatoes, Cherry

Wash tomatoes under cold running tap water for approximately 15-20 seconds to ensure 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.

5.3.50 Tomatoes, Fresh

Wash each tomato under cold running tap water for approximately 15-20 seconds to ensure 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.

5.3.51 Watermelon

Wash each melon under cold running tap water for approximately 15-20 seconds to ensure 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. If only pre-cut is available, do not rinse watermelon; remove the rind 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

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The sample is defined as the portion that the collector sends to the laboratory, usually between one and five 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 ensure an evenly mixed sample.

5.4.1 Canned Commodities

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. Refer to section 5.2 for sample hold time prior to homogenization.

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. A room temperature water bath may be used only if the sample is enclosed in watertight packaging, so that the water does not contact the sample. Open the containers and pour the entire contents into the chopper/homogenizer, except for frozen corn on the cob which requires removing the kernels from the cob using a clean dry knife or other appropriate utensil. Mechanically chop just until a visually homogeneous mixture is attained. Refer to section 5.2 for sample hold time prior to homogenization.

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

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undiluted concentrated product. Refer to section <u>5.2</u> for sample hold time prior to homogenization.

5.4.4 Dried Fruits

Open all 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. Refer to section <u>5.2</u> for sample hold time prior to homogenization.

5.4.4.1 For raisins:

Raisins can be prepared following section <u>5.4.4</u>, dried fruits. As an alternative, prepare raisins by opening all of the raisin package(s) into a container and mixing or shaking to obtain a representative analytical portion. Grind the representative portion using a cryogrinder, cover it with water, mix thoroughly and freeze it. Prepare the analytical portion for extraction and analysis. Unit counting is not required. Refer to section <u>5.2</u> for sample hold time prior to homogenization.

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. Refer to section <u>5.2</u> for sample hold time prior to homogenization.

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

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to weighing of analytical portion. Refer to section $\underline{5.2}$ for sample hold time prior to homogenization.

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. Refer to section 5.2 for sample hold time prior to homogenization.

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 eggshells. 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.

5.4.5.6 For garbanzo beans, dried:

Open all the garbanzo bean package(s) into a container and mix or shake to obtain a representative analytical portion. Grind the representative portion using a cryogrinder or an appropriate device (e.g., Falling 3300 laboratory mill, Jacobsen grinder, UDY grinder). If using a cryogrinder, grind the representative portion, cover it with water, mix thoroughly and freeze it. Prepare the analytical portion for extraction and analysis. Unit counting is not required. Refer to section <u>5.2</u> for sample hold time prior to homogenization.

5.5 Weighing of Analytical Portion

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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 paperwork that 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 quantity 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 <u>both</u> 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

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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 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, though the 90-day deadline for reporting requirement for final data is still in effect (see PDP DATA SOP Section 11.4.3).
- **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) for a period(s) as specified in the laboratory's internal SOP to protect them from degradation and solvent evaporation.

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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 <u>5.7</u> 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. 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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Original signed by Chantal Rollerson

11/09/2023

Date

Revised By: Chantal Rollerson PDP Chemist Monitoring Programs Division 1400 Independence Ave, SW Washington, DC 20250 (202) 572-8172

Original signed by Sarva Balachandra

11/09/2023

Date

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11/09/2023

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Revision 15

December 2023

Monitoring Programs Division

- Three memoranda removed from the references section because they are no longer available
- Added statement about tightly packed samples to section 5.1.12
- Added blackberries, tomatillos, and canned pumpkin to the table in section 5.3
- Clarified codes for head and leaf lettuce in the table in section 5.3
- Added blackberries to section 5.3.8
- Added new section 5.3.48 for tomatillos
- Renumbered the section headers in section 5.3 to accommodate the addition of tomatillos
- Added additional cleaning procedures for potatoes in section 5.3.41
- Clarified the use of water baths for frozen samples in section 5.4.2
- Updated the TAG approver information on the signature page

Revision 14

December 2022

Monitoring Programs Division

- Changed USDA/AMS/MPD to MPD throughout document for clarification
- Changed SOP title from "Sample Processing and Analysis" to "Sample Processing" for clarification
- Removed PCNG from the references as it has been retired.
- Changed "Record number of units received" to "Record number of units homogenized" in the table in section 5.1
- Changed "sample identification" to "sample identification label" in sections 5.1.1.2 and 5.1.1.3.
- Removed "thawed commodity" language from section 5.1.3.5.
- Clarified the wording for determining sample weight in the note in Section 5.1.5
- Added an example to section 5.1.6.1
- Changed the wording in the note in section 5.1.6.2 to remove the three-hour time window.
- Removed "if data is not available in RDE" from section 5.1.10 for clarification.
- Added the MPD email address to section 5.1.10
- Removed fact sheet references from section 5.2.4.
- Added the removal of debris from grains to section 5.3.20.
- Removed the 500g subsample requirement from section 5.3.20.
- Clarified the holding time for homogenized samples in section 5.7.1.
- Changed "MPD Sampling Team" to "PDP Sampling Team" throughout document for consistency.
- Updated "revised by PDP Chemist" signature line
- Updated the wording in the note under the Sample Receipt table.

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Revision 13

December 2021 Updated formatting throughout the document

Monitoring Programs Division

- Updated section 4 to organize references alphabetically
- Updated "revised by PDP Chemist" signature line
- Changed "assure" to "ensure" throughout the document
- Changed "MPD Sampling Manager" to "MPD Sampling Team" throughout document
- Added MPD sampling team email address throughout document
- Added note regarding grain sample receipt information in RDE in section 5.1
- Removed "Federal Grain Inspection Service (FGIS) designee(s)" in section 5.1.8
- Removed references to faxing and to the obsolete FGIS form in section 5.1.10
- Added "a determination of" for clarification in section 5.1.12
- Added reference to Fact Sheet packing and shipping conditions for commodity storage in Section
- Added maximum time between sample arrival and homogenization in section 5.2.4
- Added reference to section 5.3 for maximum holding hours prior to homogenization in section 5.2.4
- Added section 5.2.7 for explicit storage conditions and maximum storage time for grain commodities
- Added frozen peaches (PZ) row to Table 5.3
- Added maximum refrigeration time to section 5.3.1
- Updated wording for preparation of grains for homogenization in section 5.3.20
- Added maximum time between sample arrival and homogenization in section 5.3.20
- Added preparation and homogenization instructions for pre-cut watermelon in section 5.3.50
- Added reference to laboratory internal SOPs for extract storage time in Section 5.8

Revision 12 December 2020 Monitoring Programs Division

- Changed USDA/AMS to USDA/AMS/MPD throughout the document for clarification
- Added cross-referencing throughout the document
- Added reference links and a note indicating references were consulted in the preparation of this SOP in section 4
- Removed duplicate entry for reference to U.S EPA maintenance and calibration of equipment in section 4
- Added the full name and acronym for MPD in section 5
- Updated RDE entry language and changed the MPD name to acronym in section 5.1.1.2
- Added the full name for eSIF acronym in section 5.1.1.3
- Added clarification on selecting target weight for overweight samples received in section 5.1.5.2
- Updated GIPSA organization to FGIS in sections 5.1.8.1 and 5.1.10

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- Updated section 5.2.4 to include sample hold time for commodities that require refrigeration prior to homogenization
- Updated SOP preparation and homogenization reference for corn syrup, eggs and infant formula in section 5.3
- Added frozen commodities for peas and dried fruits for plums in section 5.3 in the SOP preparation and homogenization section
- Updated commodity rinse language in sections 5.3.4 and 5.3.9
- Updated broccoli preparation in section 5.3.9
- Specified UDY as a grinder in section 5.3.20
- Removed duplicate papaya ripening language in section 5.3.32
- Added nectarine reference for consistency in section 5.3.33
- Added sample hold time reference prior to homogenization to sections 5.3.20, 5.41, 5.42, 5.4.3, 5.4.4, 5.4.4.1, 5.4.5.1, 5.4.5.2, 5.4.5.4 and 5.4.5.6
- Added cryogrinding instructions and specified UDY as a grinder in section 5.4.5.6
- Updated sections 5.4.4, 5.4.5.6 and 5.6.2 for concise language

Revision 11 December 2019 Monitoring Programs Division

- Updated MPD's email address (section 5.1.8.2 and 5.1.8.3)
- Updated website for access to GIPSA's Grain Sample Ticket Form, FGIS-920 in section 5.1.10
- Updated table in section 5.3 by adding basil, garbanzo beans (dried), mustard greens, radish, canned cranberry, canned and frozen sweet corn, canned and fresh green peas
- Updated table in section 5.3 for green peas variety and code, and SOP preparation and homogenization for raisins
- Updated table in section 5.3 by changing codes for frozen spinach, sweet, fresh corn and collard greens
- Added clarification to banana sample preparation in section 5.3.6
- Added basil to cilantro sample preparation in section 5.3.16
- Updated preparation instructions for grains (section 5.3.20)
- Updated hot pepper sample preparation in section 5.3.38
- Renumbered sections 5.3.43 through 5.3.49
- Added preparation instructions for radishes (section 5.3.43)
- Updated canned commodity sample preparation in section 5.4.1
- Added raisin sample preparation in section 5.4.4.1
- Added dried garbanzo bean sample preparation in section 5.4.5.6
- Updated extract disposal documentation requirement in section 5.10

Revision 10 July 2018 Monitoring Programs Division

• Added preparation instructions for kiwi (section 5.3.24)

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- 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

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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

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- 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).