3- PESTICIDE SAMPLES

(includes 702(b) portion)

DO NOT FUMIGATE PESTICIDE SAMPLES

INTRODUCTION

The objectives of FDA's pesticide monitoring program are to gather information on levels and incidences of pesticide residues in the nation's food supply and to initiate enforcement actions against shipments of foods and feeds found to contain illegal pesticide residues. To meet both objectives, it is necessary to collect samples of foods and feeds for pesticide residue analysis. This section describes procedures for the collection of raw agricultural and processed commodity samples. These procedures apply to both domestic and import arenas. Additionally, a separate set of procedures for collecting samples in conjunction with special investigations, such as samples collected to determine levels of pesticide residues in soil, water, and growing crops, is included.

For pesticide samples, the laboratory will maintain a portion of the composited sample as the 702(b) [21 U.S.C.372(b)] portion.

Pesticide sample sizes no longer differentiate between Surveillance and Compliance Samples. All pesticide samples will be collected as directed below. Remember to include the state and county or country of origin in the Flag. See IOM 4.4.10.1.8.

For appraisal purposes, you must Flag each Domestic as to the basis for sampling in accordance with the definitions below.

Pesticide Compliance Sample. Collected on a selective basis as a result of inspectional or other evidence of suspected misuse of a pesticide on a food or feed commodity or as a follow-up to a "Pesticide Surveillance Sample" that was found to contain actionable levels of pesticide residues. Flag "Pesticide Compliance".

Pesticide Surveillance Sample. Collected on an objective basis where there is no evidence or suspicion of pesticide misuse on a food or feed commodity. Flag "Pesticide Surveillance".

Districts have the option to collect 1 intact shipping case of fresh produce from packing sheds or large produce warehouses. The one case must meet the minimum sample size specified below. This "one case" option may be used on any import sample or on domestic Pesticide Surveillance Samples, if the collector can be assured that the "one case" collected is representative of the lot or field. If the collector is not assured of this, collect the samples according to the instructions below. This "one case" sampling does not apply to large items such as melons.

NOTE: If "one case" option is used for surveillance samples of domestic produce, describe in the Remarks Section of the CR, the basis for determining that the sample is representative of the lot or field.

Plant products: description of primary samples and minimum size of laboratory samples (total weight of all subs or units collected).

Commodity classification	Examples	Nature of primary samples to be taken	Minimum sample size and number of units of each laboratory sample
1. PRIMARY FOOD COMMODITIES OF PLANT ORIGIN			
All fresh fruits, All fresh vegetables, Frozen bulk produce (not retail) except dry pulses			
Small sized products units generally < 25 g	Berries, peas, olives	whole units, or packages, or units taken with sampling device	1 kg (2.2 lbs)
Medium sized products units generally 25 - 250 g	Apples, oranges, corn on the cob, potatoes	whole units, or units taken with sampling device	1 kg (2.2 lbs) (at least 10 units)
Large sized products units generally > 250 g	Cabbages, lettuce, cucumbers, grapes (bunches, except for sulfites), sweet potatoes	whole units, units taken with sampling device	2 kg(4.4 lbs) (at least 5 units)
Pulses, Cereal grains	soy beans, peas, lentils, rice, wheat (except from rail carloads)		1 kg (2.2 lbs) 1 kg (2.2 lbs)
Tree nuts	(except coconuts)		1 kg (2.2 lbs)
	coconuts		5 units
Oilseeds	peanuts		0.5 kg (1.1 lb)
Seeds for beverages and sweets	coffee beans		0.5 kg (1.1 lb)
Herbs (*for dried herbs see section 3 of this Table*)	fresh parsley others, fresh	whole units or units taken with sampling device	0.5 kg (1.1 lb) 0.2 kg (0.5 lb)
Spices	dried	whole units or units taken with sampling device	0.1 kg (0.25 lb)
2. PRIMARY ANIMAL FEED COMMODITIES			
Primary feed commodities of pant origin			
Legume animal feeds, and other forages and fodders		whole units, or units taken with sampling device	1 kg (2.2 lbs) (from least 10 units)
Straw, hay and other dried products		whole units, or units taken with sampling device	1 kg (2.2 lbs) (from least 10 units)
Note. See IOM Sample Schedule Chart 4, Wheat Carload Sampling for guidance in the collection of samples by trier from railcars and trucks.			
3. PROCESSED FOODS OF PLANT ORIGIN			
Secondary food commodities of plant origin, dried fruits, vegetables, herbs, milled cereal products **Derived products of plant origin**, teas, vegetable oils, juices, by-products for animal feed and miscellaneous products **Manufactured foods (single ingredient) of plant origin**, **Manufactured foods (multi-ingredient) of plant origin**, including products with ingredients of animal origin where the ingredient(s) of plant origin predominate(s), and breads			
Products of high unit value		packages or units taken with a sampling device	0.1 kg* (0.25 lb)
Solid products of low bulk density	Hops, Tea	packaged units, or units taken with a sampling device	0.2 kg (0.5 lbs)
Other solid products	bread, flour, apple pomace, dried fruit	packages or other whole units, or units taken with a sampling device	0.5 kg (1.1 lbs)
Liquid products	vegetable oils, juices	packaged units, or units taken with a sampling device	0.5 L or 0.5 kg
** A smaller laboratory sample may be taken from a product of exceptionally high value but the reason for doing so should be noted in the collection report.*			
4. EGGS AND DAIRY PRODUCTS			
Poultry eggs			
Eggs, except quail and similar		whole eggs	12 whole chicken eggs, 6 whole goose or duck eggs
Eggs, quail and similar		whole eggs	24 whole eggs
Milks		whole unit(s), or unit(s) taken with a sampling device	0.5 L
5. PROCESSED FOODS OF ANIMAL ORIGIN			
Secondary food commodities of animal origin, skimmed milks, evaporated milks and milk powders **Derived edible products of animal origin**, milk fats, butters, butter oils, creams, cream powders, caseins, etc.			

Commodity classification	Examples	Nature of primary samples to be taken	Minimum sample size and number of units of each laboratory sample
Manufactured food (single ingredient) of animal origin, **Manufactured food (multi-ingredient) of animal origin**, (including products with ingredients of plant origin where the ingredient(s) of animal origin predominates(s))			
Liquid milk, milk powders, evaporated milk and cream, cream, dairy ice cream, yogurt		packaged unit(s), or unit(s) taken with a sampling device	0.5 L (liquid) or 0.5 kg(solid)
Notes. (i) Evaporated milks and evaporated cream in bulk must be mixed thoroughly before sampling aseptically. (ii) Milk powder in bulk should be sampled aseptically, passing a dry borer tube through the powder at an even rate. (iii) Creams in bulk should be mixed thoroughly with a plunger before sampling but foaming, whipping and churning must be avoided.			
Butter and butter oils (butter, whey butter, low fat spreads containing butter fat, anhydrous butter oil, anhydrous milk fat)		whole or parts of packaged unit(s), **or** unit(s) taken with a sampling device	0.2 kg or 0.2 L
Cheeses, including processed cheeses	units 0.3 kg or greater	whole unit(s) or units taken aseptically with a sampling device	0.5 kg
	units < 0.3 kg	whole unit(s)	0.3 kg
Note. Cheeses with a circular base should be sampled by making two cuts radiating from the center. Cheeses with a rectangular base should be sampled by making two cuts parallel to the sides.			
Liquid, frozen or dried egg products		unit(s) taken aseptically with a sampling device	0.5 kg

9. GRAPES FOR SULFITES

Collect approximately 900 - 1800 g (2 - 4 lbs) of grapes [10/100 - 200 g (1/4 to 1/2 lb) subs]. Each subsample will consist of individual grapes, not bunches, and will be collected from different lugs (cases) on as many different pallets in the lot as possible. No grapes that are damaged during the sampling procedure should be included in the sample. However, grapes with damage prior to sampling may be included in the sample.

If sulfiting pads are present, grapes sampled should be selected from areas closest to and directly under the pad.

Monitoring activities should be focused upon lots of grapes with the highest potential for violative sulfite residues.

Direct efforts to lots of grapes sulfited through fumigation or to lots with multiple fumigations especially towards the end of the harvesting season and also to lots with significant numbers of damaged grapes (split, crushed, or unusually wet, if such damage is apparent).

Sample lots of grapes sulfited through the use of sulfiting pads, with or without additional fumigation. If at all possible, sample lots subjected to the following conditions, which could cause high sulfite residues:

- Lots subjected to un-refrigerated storage of 2 or more hours during warm weather.
- Unusual shipping conditions (ships at sea during heavy storms).
- Lots with significant numbers of damaged grapes.
- Lots containing evidence of sulfite pad damage sufficient to cause spilling of sulfiting agent onto grapes.

Special Sample Handling

Place sample in tightly closed airtight glass mason jar(s) or sealed plastic bag(s). Although no effort should be made to commingle subsamples, more than one subsample may be placed in the same container for shipping convenience.

Appropriate cooling procedures are:
Place samples in shipping container or cooler with sufficient ice or other refrigerant to keep sample refrigerated until arrival at the laboratory. Sample should be placed immediately in a refrigerator at or below 7 degrees C. If sample is not to be analyzed within a few hours, the sample should be placed in a freezer, which is maintained at or below -20 degrees C.

Or, if the sample is frozen, place the sample in a container with sufficient dry ice to keep the sample frozen until arrival at the lab. The sample should then be placed in freezer upon arrival at the laboratory.

1. **FISH AND SHELLFISH PRODUCTS**

NOTE: THIS SAMPLE SIZE FURNISHES SUFFICIENT FISH FOR HEAVY METAL ANALYSIS.

Packaged Fish, fresh, frozen, smoked, cured, or shellfish (except oysters)
Collect 12 subs - minimum sub size is 453 g (1 lb)

Bulk Fish - .453 - 1.35 kg (1 - 3 lb)/fish
Collect 12 subs, each sub to consist of 453 g (1 lb) of edible fish

Bulk Shellfish (except oysters)
Collect 12 - 453 g (1 lb) subs

Canned Fish and Shellfish Products (except oysters)
Collect 12 subs - 5 cans per sub

Other Fish and Shellfish Products
Oysters - Collect 12 1 pint subs

Fish Flour and Meal
Follow the guidance in section 5 above.

SWORDFISH FOR HEAVY METALS

These sample sizes must be used whenever sampling swordfish, either for audit, surveillance, or compliance purposes.

Whole Fish (dressed, head removed)
Characterize lot in terms of fish sizes, i.e., small, medium, and large. The following dressed weight ranges are used for classification:

Small Fish - Weighs less than 36.4 kg (80 lbs)

Medium Fish - Weighs 36.4 - 54.5 kg (80 - 120 lbs)

Large Fish - Weighs more than 54.5 kg (120 lbs)

For lots consisting of 12 or more fish, the representative sample to be collected will be determined by the following formula:

$ns = (n)(Ns)/N$
ns = the number of fish in a given weight range from which subsamples must be taken
n = total number of subsamples to be collected from the lot. (In using this formula n will always equal 12)
Ns = the number of fish in a given weight range in the lot
N = the total number of fish in the lot

Example: If a lot consists of 25 fish and is characterized as: 5 small fish [less than 36.4 kg (80 lbs)], 15 medium fish [36.4 - 54.5 kg (80 - 120 lbs)], and 5 large fish [greater than 54.5 kg (129 lbs)], the sample should be collected as follows:

small fish $\frac{(12)(5)}{25} = 2.4 = 2$

medium fish $\frac{(12)(15)}{25} = 7.2 = 7$

large fish $\frac{(12)(5)}{25} = 2.4 = 2$

TOTAL SAMPLE: 11 sub samples

Usually, the total sample will consist of 12 subsamples. However, due to rounding numbers of subsamples determined by the formula may be 11 or 13 in some instances. The total sample should consist of the specific number of sub samples determined by the formula in all cases.

Each sub sample should consist of approximately a 0.5 kg (1 lb) steak cut from just below the nape of the fish. Care should be taken to avoid mutilation of fish. The sub must consist of edible flesh. If a private laboratory is conducting the analysis, individual fish from which the sub sample is taken should be identified with a tag or other suitable method. This will permit FDA to take audit samples from the same fish sampled by the private laboratories.

For lots consisting of 12 or less fish, collect 1 sub from each fish.

Swordfish Loins (slabs or sides cut from dressed whole fish which has been boned or trimmed).

Use the same formula stipulated for whole fish, with the exception that the following weight ranges should be used to characterize the lot:

Small fish loins = weighs 9.1 - 18.2 kg (20 - 40 lbs)
Medium fish loins) = weighs 18.2 - 36.4 kg (40 - 80 lbs)
Large fish loins = weighs over 36.4 kg (80 lbs)

Swordfish Steaks

Collect 12 sub samples, i.e., 12 steaks, at random from different containers in the lot (as many as possible)

Canned Swordfish

Collect 12/453 g (1 lb) sub samples at random

11. RETAIL CONTAINERS CANNED, FROZEN AND DRIED FOODS

Collect retail containers equal to the number of primary units specified above.

12. SPECIAL INVESTIGATIONS

Growing Crops

Superimpose an imaginary grid on the field dividing it into approximately 100 areas. Randomly select 10 areas to form a representative sample of the field. Collect one pound subs from each area. Combine to form a composite. If a sample is being collected to document drift, etc. DO NOT composite subs. In addition, diagram the field in the Remarks Section of the C/R and indicate sub number where each sub was collected.

For leafy vegetables, such as lettuce, cabbage, etc.: INV Samples collected in the growing field should be representative of local commercial harvesting practices If the local practice is to strip outer leaves at the time of harvest, this practice should be followed when collecting field samples. In head lettuce, for example, the lettuce may be packed directly into shipping cartons in the field, in which case 6 or 8 outer leaves are left on the head to be removed at the retail outlet. In other instances, each head is stripped of 2 or 3 outer leaves and individually wrapped in plastic, placed in shipping cartons, and the consumer receives the produce in this condition. Describe sampling method on C/R and describe how packing shed handles produce prior to shipping (e.g., washing, waxing, stripping, etc.).

Soil Samples

Collect soil samples from fields according to the following 3x3 grid diagram:

	a	**b**	**c**
1	o	o	o
2	o	o	o
3	o	o	o

Sample at the 9 locations indicated by the "o". If the field being sampled is very large, you may have to sample it using a 4x4, 5x5, or even larger grid pattern.

Subs are to be placed in clean quart glass jars, which have been washed in water, rinsed in methanol, and air dried. If methanol is not available, use washed, air dried jars and submit an empty jar as a control. Note on CR that jars were or were not rinsed with methanol.

Obtain two "6 in" deep plugs (1-2 in. in diameter from each sampling location. Place two plugs from each location in cleaned glass jars, place clean aluminum foil over top of jar and seal with screw cap.

Soil samples should be submitted to the lab at 4° C (39° F) or below.

Water Samples - Collect 3 quarts of water from the same sampling source (e.g., faucet, stream, lake, etc.) and place in cleaned, washed and methanol rinsed jars as described under "Soil Samples".

Submit water samples to lab at 4° C (39 ° F) or below.

GENERAL

Official Samples shall be collected whenever feasible unless they are not required to accomplish the objective of the assignment. Investigational Samples shall be collected only when Official Samples are not readily available.

Consult with your supervisor in cases of doubt as to sample cost, size, or collection technique.

When collecting samples in glass jars, line the lids with aluminum foil which has been certified by the laboratory as contaminant free or use Teflon lined lids.

If shipment of shell eggs is required and breakage may result during transit, subs may be broken, shells discarded, and liquid magma collected in clean glass jars. Each sub jar should be properly identified.

Samples collected at Packing Sheds should be representative of the produce as shipped in commerce. DO NOT strip outer leaves from subs collected at packing sheds from bulk lots, shipping cartons ready for shipment, in-transit lots or at final destination. If the packing shed practice is to strip outer leaves prior to shipment, follow this practice when collecting the samples. Describe the sampling method on the C/R.

DO NOT USE magic markers, etc. to identify sub bags, because the ink may affect assay results. Use stick on labels to identify sub bags.

Collect samples in the container in which the dealer is packaging the product. If the dealer is packaging the product in plastic bags, collect sample in these bags. If the firm is not packing the product, collect the samples in paper bags, cardboard cartons, etc. Do not use plastic bags as this may interfere with the analysis, unless the bags are certified as contaminant free by your district laboratory.

Samples must be delivered as promptly as possible to the laboratory if regulatory action is to be taken against actionable lots.

Hold samples in cold storage until ready to be shipped or delivered to the laboratory. If the sample is of a hard fruit or vegetable (such as apples, pears, butternut squash), and is shipped overnight delivery, it can be shipped to the laboratory unrefrigerated, but the FDA 525 should direct refrigeration upon receipt.

Use aseptic technique, where applicable, when collecting samples of finished products from bulk containers.