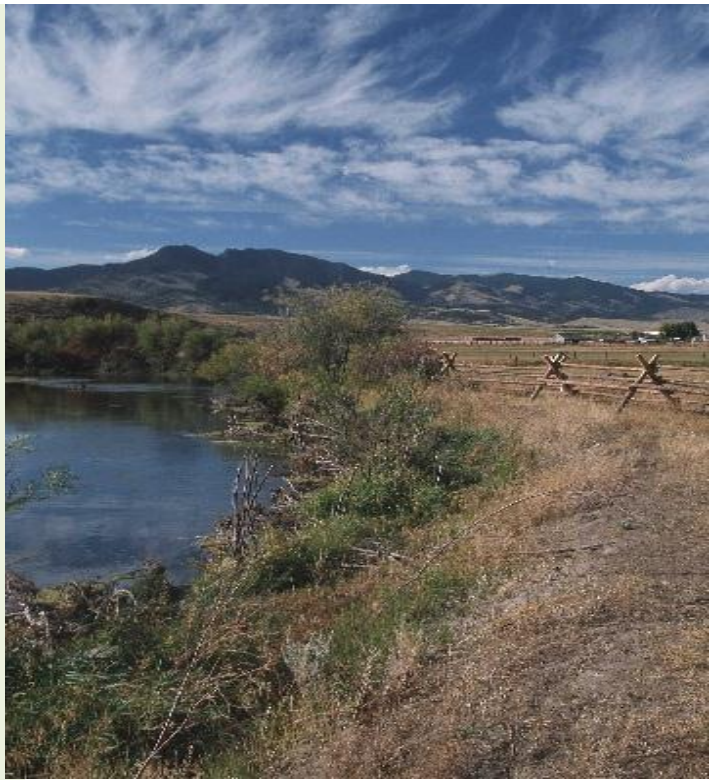
National Organic Program Accredited Certifying Agent Training 2011

RESIDUE SAMPLING

Section § 205.670 of the NOP regulations specifies the conditions under which responsible parties should conduct testing of agricultural products that will be sold, labeled, or represented as “100 percent organic”, “organic”, or “made with organic (specified ingredients or food group(s))”.

RESIDUE SAMPLING

Under § 205.504(b)(6), certifiers must have procedures for sampling and residue testing to ensure that proper sampling is routinely followed.



RESIDUE SAMPLING

To meet this requirement, these parties are responsible for the collection of samples of organic agricultural products that will be tested to detect the presence of residues in violation of the NOP regulations as specified under § 205.105 or other applicable laws as provided for at § 205.670(e).

Objectives

RESIDUE SAMPLING OBJECTIVES

Ensure consistency in the sampling of organic agricultural products, for residue testing requirements under §205.670 of the NOP regulations.

- Review the instructions provided by the NOP, for the collection, proper documentation, maintenance of the chain of custody and sample integrity, for samples collected for residue testing.
- Ensure that certifier's procedures for residue sampling are sufficient to meet the criteria addressed in today's training.

When to Collect Samples

Samples should be collected under the following conditions:

- When it is suspected that a prohibited substance has been applied.
- When it is suspected that contamination from genetically modified organisms, antibiotics, or prohibited substances may have occurred.
- When pesticide drift may have occurred.
- To gather evidence as part of an investigation.
- As part of a surveillance sampling program.

SAMPLE SELECTION

Sample collectors should collect a sample of a given organic agricultural product, selected from a single location in a field, bin, or pallet.

A single sample analyzed for residues using sensitive test procedures should provide enough information to determine if residues are present

Raw or processed sample

SAMPLE SELECTION

A sample of a crop could consist of the raw agricultural commodity (RAC) or processed commodity from the RAC (EPA Residue Chemistry Guidelines, Table 1).

Additional Resource - The Codex Alimentarius Commission (Codex) for recommended methods of sampling for the determination of pesticide residues.

PRIOR TO COLLECTING

Prior to Collecting Samples

Determine if collector has the scope of expertise needed to ensure that the sample collected is done according to stated procedures.

Risk Areas

SAMPLE SELECTION AREAS

Sample collectors may choose to select samples which attempt to detect contamination where it is most likely to occur due to risk factors present at a given operation or a location within an operation.

- Example: an organic field with fallow pasture on three sides and a conventional crop on the fourth.

TABLE 1

SAMPLE AMOUNTS -TABLE 1

Collectors should obtain sufficient sample to ensure the laboratories will have adequate amounts for processing and reanalysis if necessary (Table 1).

Commodity Type	Recommended Sample Amount
Most fresh fruit and vegetables	3-5 pounds (approximately 1.5-2.5 kg); A single large melon or squash exceeding 5 pounds (approximately 2.5 kg) is acceptable.

TABLE 1

SAMPLE AMOUNTS (Table 1)

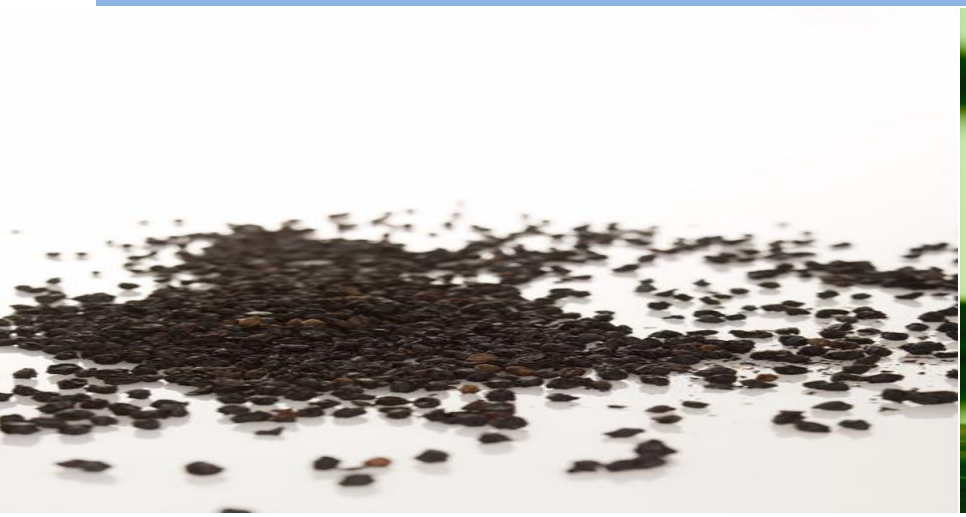
Blended commodities or those smaller than a strawberry	1 pound (approximately 500 g)
Berries	
Cherries	
Coffee beans	
Dried Commodities	
Flours	
Grains	
Herbs	
Garlic	
Legumes	
Mushrooms (small)	
Nuts	
Teas	
Seeds	
Small jars/packages (i.e. baby food sized)	
Spices	
All liquids and semisolid foods (i.e. juices, oils)	16-32 ounces (approximately 500 mL to 1000 mL)
Canned/jarred foods	

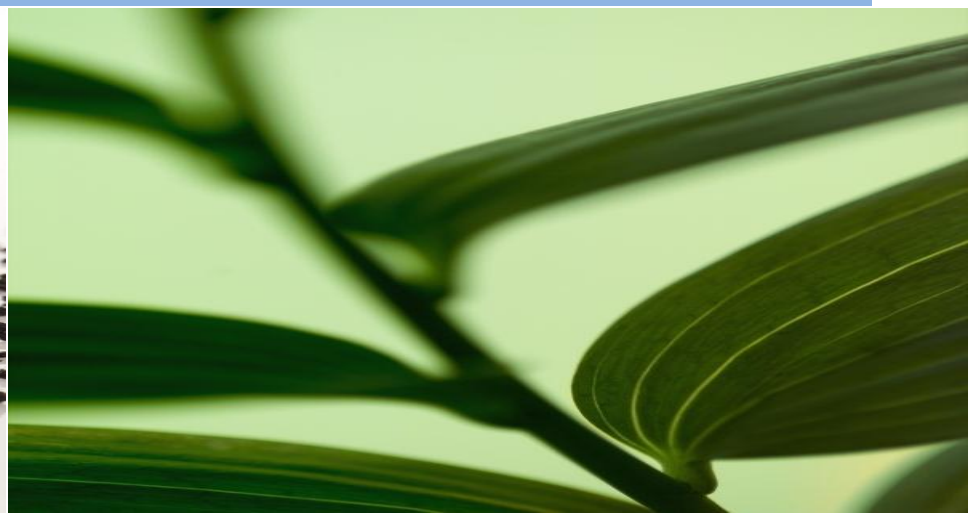
SAMPLE AMOUNTS

- If collecting from multiple containers is needed to obtain the suggested amounts, (Table 1), sample collectors should confirm the products being sampled are from the same lot.
- For raw commodities, the portion which should be sampled is generally the whole commodity.

SAMPLE AMOUNTS

Adhering soil, decomposed outer leaves, and inedible root and tuber vegetable tops should be excluded from the sample





Documentation
-on site

SAMPLE DOCUMENTATION

Each sample should be identified by the following information:

- Certified operation name and mailing address (city/state/zip/country).
- Identification of sampling site (may include site maps or field identification).
- Grower and handler information (both grower and handler should be included if the sample is not collected at the farm).

Additional Sample Documentation continued on next slide

SAMPLE DOCUMENTATION

- Sample identification, including commodity information, variety, brand name and lot number (if applicable), or other identification.
- Certifier name.
- Collector's name & signature.
- Date collected and date shipped.

DOCUMENTATION at the LAB

Upon arrival at the laboratory, the following information should be recorded by the laboratory and included with the sample results:

1. Date received.
2. Name of person receiving the sample.

Additional Lab documentation continued on next slide

DOCUMENTATION at the LAB

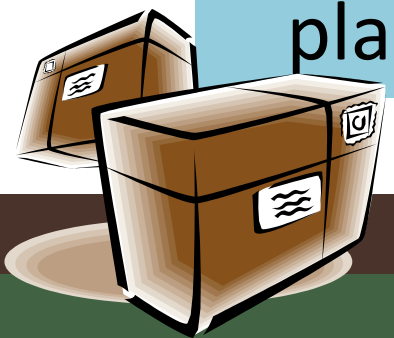
3. Explanation for samples if they are not analyzed (e.g., chain of custody breached, sample rotted, sample miscoded).
4. Internal Sample ID: The laboratory should generate an internal Sample ID.

MAINTAINING CHAIN OF CUSTODY

The chain of custody ensures the chronological possession of samples as they pass from the sample collectors, to the shipping carriers, to the laboratories.

SAMPLE INTEGRITY

- Each sample shall be packed by the sample collector using precautions to prevent sample contamination from commingling or contact with prohibited substances.
- Samples of fresh commodities must be taken using gloved hands (latex or clean rubber gloves) and removed from the plant or storage bins using a clean utensil.



SAMPLE INTEGRITY, cont.

- Sample collectors should avoid including excess dirt and foliage (as appropriate) from field samples.
- Samples should be placed into a clean plastic bag (or other receptacle required by a given laboratory) and sealed with tape to provide a tamper-proof seal.

Sample Integrity-labeling

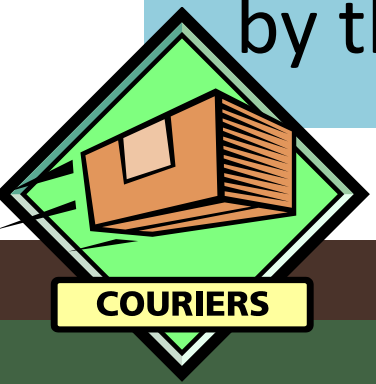
SAMPLE INTEGRITY, cont.

- Samples should be initialed and dated by the sample collector who has bagged the sample.
- In addition to the initial and date, the outside of each sample bag should be permanently marked with a unique identification code.

Sample Integrity-labeling

SAMPLE INTEGRITY, cont.

- A shipping label with time and date will be acceptable as evidence of transfer to the carrier and delivery to the laboratory.
- Sample collectors should ensure that the shipping container is properly sealed, labeled for perishable goods, and ship the container by the appropriate means of transportation.



Sample Integrity-shipping

SAMPLE INTEGRITY, cont.

Sample collectors should avoid shipping samples that will arrive during a weekend or holiday when laboratories are not open to receive and process the samples for analysis.

Sample Integrity-shipping

SAMPLE INTEGRITY, cont.

It is important to note that many samples will require refrigerated temperatures for shipping and should be placed in a pre-cooled, insulated shipping container with an adequate number of frozen cold packs.

Sample Integrity-shipping

SAMPLE INTEGRITY, cont.

If samples are transported away from the collection site to be packed at a later time, then the samples must be maintained in a cooled container until they are packed for shipment.

Sample Integrity-shipping

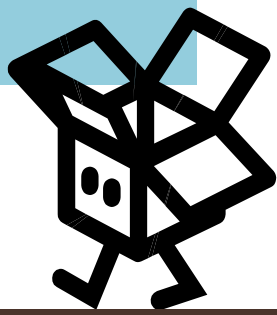
SAMPLE INTEGRITY, cont.

- Sufficient packing materials (i.e., bubble wrap) should be used to prevent movement during transit.
- Fresh and frozen samples should be shipped overnight.
- Processed foods normally stored at room temperature (i.e. canned vegetables,) can be shipped at ambient temperature by ground.

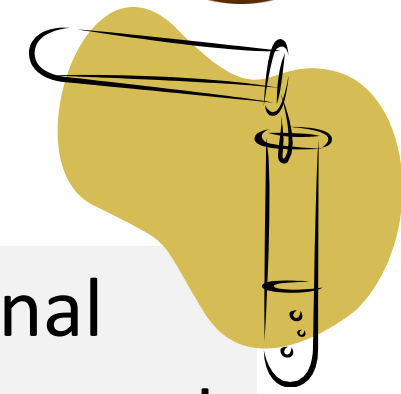
Sample Integrity-shipping

SAMPLE INTEGRITY, cont.

- In cases where the shipping container will not change hands (i.e. if the sample is being delivered directly to the laboratory by the sample collector) it is not necessary for the packing box to be sealed.



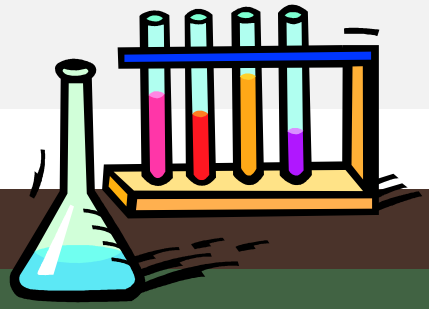
Laboratory Selection



The NOP recognizes that an international harmonized method for residue testing may not be possible at this time, but that sufficient policies and procedures must be in place to ensure that false positives and false negatives are not reported.

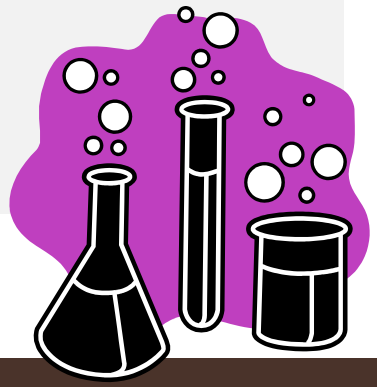
Laboratory Selection

- In collaboration with the USDA AMS Science and Technology Program, NOP created a "target" analyte list (NOP 2611-1) by examination of all pesticides/metabolites/environmental contaminants that have been detected in samples analyzed for the USDA Pesticide Data Program.



Laboratory Selection

Laboratories employed by certifying agents should attempt to analyze as many compounds on this list as possible. If certifying agents suspect a prohibited substance was used that is not included on the NOP "target" list, they should initiate sampling/testing and investigation.



Laboratory Selection Criteria

Certifying agents should consider the following when selecting a laboratory for residue analysis of their samples:

5 levels of criteria

Laboratory Selection Criteria

1. Laboratories should hold current accreditation to the international standard, ISO/IEC 17025:2005, *General Requirements for the Competence of Testing and Calibration Laboratories.*

A copy of the accreditation certificate should be provided to the certifying agent prior to shipping samples and should be attached to laboratory results when they are reported back to the certifying agent.

Laboratory Selection Criteria

2. Laboratories should participate in an international proficiency test program. A proficiency testing program is the determination of the calibration or testing performance of a laboratory by means of inter-laboratory comparison.

A copy of the results from the most recent round of proficiency testing should accompany results.

Laboratory Selection Criteria

3. Laboratories should be capable of screening for the "target" analyte list of pesticides included on the document NOP 2611-1, analyzing the samples using gas chromatography (GC) and/or liquid chromatography coupled to a mass spectrometer (MS) or tandem mass spectrometers (MS/MS).

Laboratory Selection Criteria

4. Laboratories should provide evidence that their analytical method is appropriate for the submitted sample and that suitable validation data are available.

Correspondence should be available to the certifying agent documenting that the method meets the laboratories' minimum internal quality assurance requirements

Analytical Results

5. Certifying agents should direct the laboratory to provide analytical results as follows:
   - **<u>If no residue is detected</u>** the result should be provided as ND (not detected). The limit of detection should be provided.

Analytical Results

- If some residue is detected below the limit of quantification (LOQ), the result should be provided as "Trace" or "BQL" (below quantifiable level).
- If residue is detected at or above the LOQ, the result shall be reported in parts per million (ppm)[1]. *Parts per million (ppm) is equivalent to milligrams per kilogram (mg/kg).*

Review of Analytical Results:

If no residue is detected -the result should be provided as ND (not detected). The limit of detection should be provided

If some residue is detected below the limit of quantification (LOQ)-the result should be provided as "Trace" or "BQL" (below quantifiable level).

If residue is detected at or above the LOQ-

the result shall be reported in parts per million (ppm)[1].

Suggested Laboratory Practices

- Laboratories should use a unique identifier to track the sample throughout the handling and analysis.
- Before homogenization, the sample may be stored at 4 degrees Celsius for up to 72 hours, if fresh, or stored at ambient temperature in the case of samples normally stored at room temperature.
- If a sample was previously frozen and shipped on ice packs, then it should be homogenized upon receipt at the laboratory

Suggested lab practices- continued

Suggested Laboratory Practices

- The entire sample as received (up to 5 pounds (~2.5 kg)) should be homogenized by the laboratory to obtain a suitable representative portion for analysis.
- Homogenized samples should be stored at less than -20 degrees Celsius.
- Violative sample homogenates should be retained (preferably stored at -80 degrees Celsius) until the contamination issue is resolved by the certifying agent.

Suggested lab practices- continued

Suggested Laboratory Practices

- Samples should not normally be washed or peeled (e.g. bananas, oranges). However, certain commodities may be hulled (e.g. hazelnuts, fresh soybeans), and/or pitted (e.g. mango, avocado) prior to homogenization.
- In some cases a sample will have to be reconstituted (frozen concentrated juices). It should be noted that U.S. EPA establishes tolerances on specific raw agricultural commodities (RACs) and feedstuffs derived from crops listed in Table 1 of the Residue Chemistry Guidance that may not apply to the submitted sample.

QUESTIONS