



Sampling and Analysis of Organic Commodities for the Determination of the Presence or Absence of Pesticide Residues

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Sampling for Analytical Testing



- The reliability and relevance of any analytical test is totally dependent upon the integrity and representation of the sample tested.
- A laboratory can perform the most accurate, precise, and defensible analysis of a sample, but if the sample is not relevant or defensible, the analysis is worthless.
- Reliable laboratory results begin with and depend directly upon the quality and timing of sample collection.

It is imperative that Certifiers follow their standard operating procedures for sampling; otherwise *ALL results* may be considered suspect.

Communication is a key ingredient in a successful sampling program



The laboratory needs notification of sample collection prior to sample shipment in order to be ready to receive and process the sample.

Sample Collectors Shall Notify:

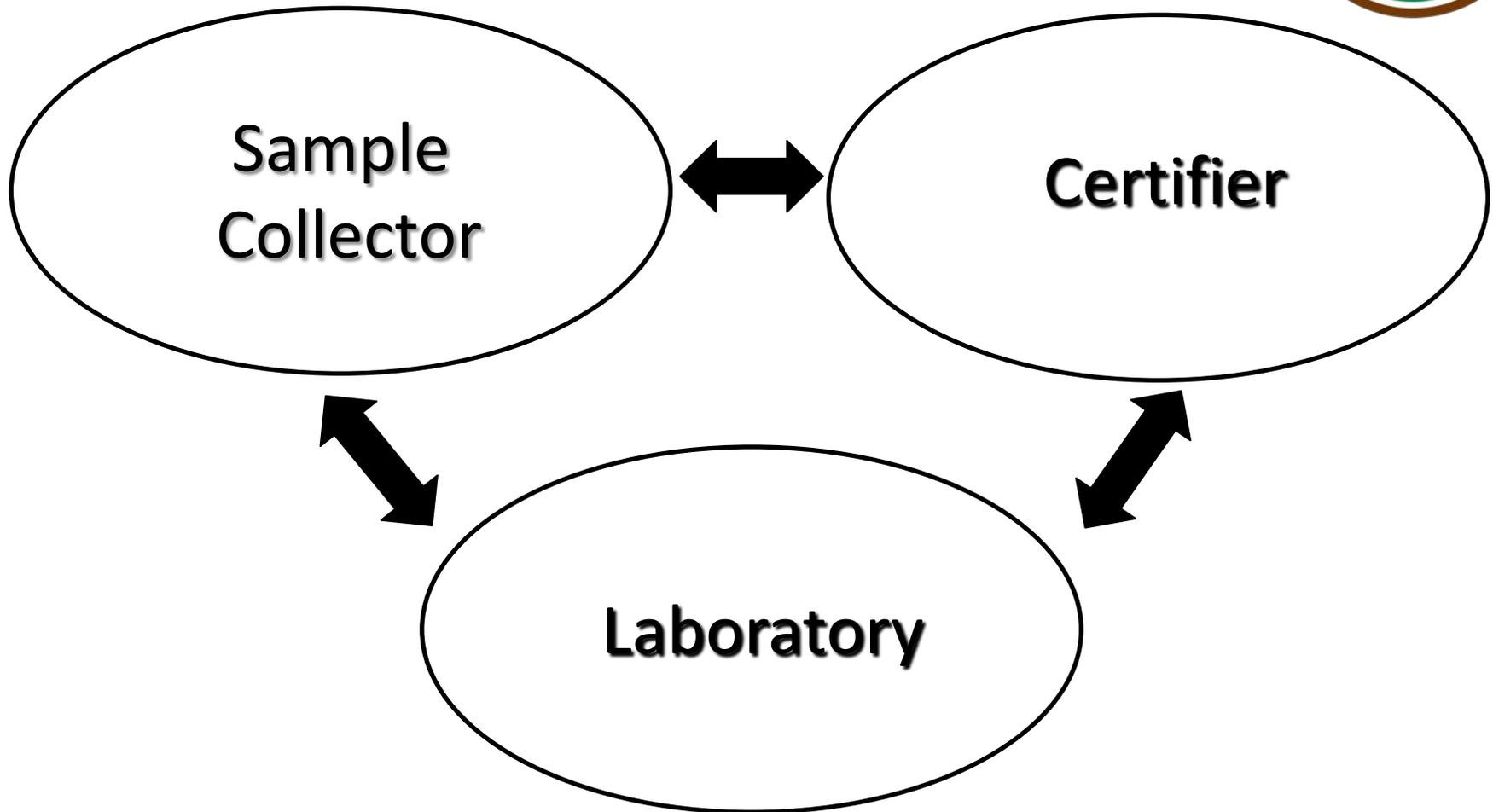
Certifier

- When scheduled samples are collected and shipped.
- When problems arise with sampling.
- Any other sampling-related problems.

Lab

- When scheduled samples are collected and shipped.

Diagram of Communications



Notification Procedures:



Certifier Should Notify:

Laboratory

- When sampling is planned or scheduled in advance to assist in laboratory preparation.

Laboratories Should Notify:

Certifiers

- Rotten, spoiled, or damaged samples
 - Samples did not arrive
 - Insufficient sample weights or numbers
 - Missing sample form
 - Any other sample receipt problems
- Lab requests resample**

Use of Field Notebooks



Field Notebooks are used by the collector for reference and documentation

Collectors should have sampling information in a notebook that includes:

1. Site information
2. Certifier or NOP sampling SOPs
3. Blank sampling forms
4. Packaging and shipping instructions
5. Certifier, Laboratory, and NOP Contact Information

Preparation for Sample Collection



Pre-frozen cold packs



Styrofoam cooler (with lid)
fitted conformably inside
cardboard box

OR, thick bubble wrap to
conform to inside of sturdy
cardboard box





Several ice packs placed inside box to pre-cool container

Checklist prior to sampling



- Insulated container with ice packs
Inside
- Field notebook with blank sampling forms
- Markers, pens, pencils
- Sample bags
- Gloves (optional)
- Tamper-proofing tape
- Sample identification stickers & envelopes
- Knife or scissors
- Laptop computer or PDA Portable scale (optional)
- Packing materials, tape, & shipping labels.

Timing of Sample Collection



- Attempt to call the sample collection site ahead of time to assure product availability and accessibility.
- Samples collected on Mon. – Thursday only with overnight shipment.
- Samples shipped the same day as collection, for arrival at labs the next day.
- Labs are notified the same day samples are collected for preparedness.
- Labs notify Certifier if samples do not arrive at laboratory.

Samples selected randomly from site



Samples selected that are NOT damaged, overripe, or wilted



Plastic bags should be used for samples

Bags should NOT be overstuffed



Target Sample Weights



Target Sample Weights:

3 lbs—small produce

5 lbs—large produce

A minimum of two units for large commodities are required --- to obtain representative samples



Non-perishable Products



Non-perishable products like processed foods, grains, dried fruit, spices, etc. can be collected in the finished container and shipped without being cooled. These types of samples may also be shipped ground instead of overnight to save on shipping costs.



Fiber, non-edible plant parts, soil and water samples



Fiber samples (cotton, wool, etc.) may also be handled like non-perishable food products.

Non-edible plant parts (leaves, stems, blossoms, etc.) can be collected, handled and shipped in the same manner as perishable fruits and vegetables.

Soil and water should be shipped overnight but do not require cooling. Water samples should be collected in screw top plastic bottles of 1 liter size or larger. Soil samples should be collected into plastic bottles or plastic bags and should be approximately 1000 grams or larger

Sample Information



1. Sample Documentation

Each sample shall be identified by the following information on the sample form - PLEASE WRITE LEGIBLY:

- **Certified operation name and mailing address (city/state/zip/country).**
- **Identification of sampling site (may include site maps or field).**
- **Grower and handler information (both grower and handler should have identification included if the sample is not collected at the farm).**
- **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.**
- **Name of Lab samples are shipped to for analysis**

Note:
The certified operation must also receive documentation (i.e. a receipt) when a sample is obtained for analysis.

Tamper-proofing and chain-of-custody



- Write sample ID on the sample bag either directly or with a sticker.
- Place the sample into the plastic bag and place tamper proof tape around the knot for chain-of-custody protection.
- Place sampling form in an envelope and place the envelope on top of the insulated container and under the cardboard box lid.



Sample info on the bag and tape placed around knot in bag that is initialed and dated

Preparation for Shipment



Frozen cold packs are placed around all sides, top, & bottom of bagged samples

Plenty of paper is used for cushioning & insulation

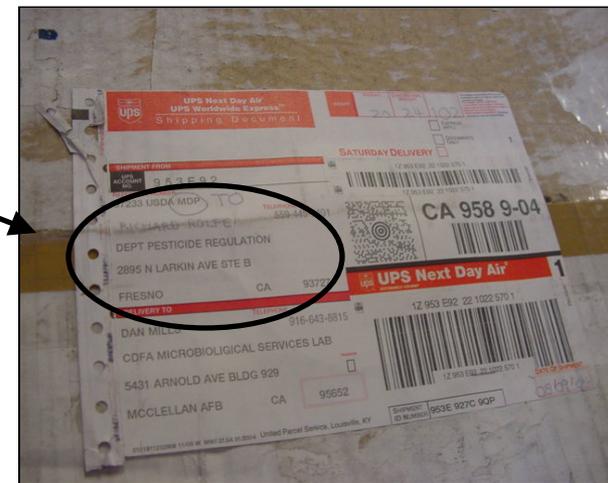




Box is securely taped



And properly labeled and shipped for overnight delivery



Collectors can reuse boxes to save on costs by placing a return request inside box



Sample Receipt at Laboratory



**Samples Should
Arrive at <math><15^{\circ}\text{C}</math>
(<math><60^{\circ}\text{F}</math>)**

**Ideal Temperature
is $4\text{-}7^{\circ}\text{C}$ ($39\text{-}45^{\circ}\text{F}$)**



Laboratory Requirements



Upon arrival at the laboratory, the following information shall be recorded by the laboratory on the sampling form:

- Date received.
- Name or initials of person receiving the sample.
- Explanation for what happened to a sample that is not analyzed (e.g., chain of custody breached, rotten sample, sample miscoded).
- Internal Sample ID: The laboratory shall generate an internal Sample ID for chain-of-custody.

Sample Login-

- The Lab shall login the sample information on the sample form to record the receipt and utilize the information when reporting results.
- A unique laboratory identification shall be assigned to each sample at login.
- Samples are then transferred to temporary storage prior to preparation for analytical testing



Sample Prep

- Samples are homogenized in a large, high speed food processor.
- Some commodity types (e.g. high fat or dry samples) require the addition of dry ice to the homogenization step.
- Samples are stored at $-20\text{ }^{\circ}\text{C}$ prior to analysis.

Dry ice
homogenization



Reporting of results



United States
Department of
Agriculture

Agricultural
Marketing
Service

Science and
Technology

National Science Laboratory
801 Summit Crossing Pl. Ste. B
Gastonia, NC 28054

Applicant Identifier: _____ Sample Description: **Organic Sample** Lot #: XXXXXX
 Contact Person's Name _____
 Company Name _____
 Company Address _____
 Company Phone Number _____

Sample Identifier: **A199999** Date Received: **07/18/11** Date Completed: **7/25/2011**

Pesticide Residue	Result (PPM)	LOD (PPM)	Pesticide Residue	Result (PPM)	LOD (PPM)
1-Naphthol	N.D.	0.050	Clofentezine	N.D.	0.020
2,4 Dimethylphenyl formamide (DMPF)	N.D.	0.004	Clothianidin	N.A.	0.001
3-Hydroxycarbofuran	N.D.	0.004	Coumaphos	N.D.	0.001
4,4-Dibromobenzophenone	N.D.	0.010	Cyazofamid	N.D.	0.002
5-Hydroxythiabendazole	N.D.	0.050	Cycloate	N.D.	0.025
Acephate	N.D.	0.010	Cyfluthrin	N.D.	0.004
Acetamiprid	N.D.	0.004	Cyhalothrin lambda	N.D.	0.001
Acetochlor	N.D.	0.025	Cypermethrin	N.D.	0.004
Aldicarb	N.D.	0.004	Cyprodinil	N.D.	0.004
Aldicarb sulfone	N.D.	0.003	Cyromazine	N.D.	0.050
Aldicarb sulfoxide	N.D.	0.020	DDD o,p	N.D.	0.040
Aldrin	N.D.	0.010	DDD p,p	N.D.	0.020
Allethrin	N.D.	0.008	DDE o,p	N.D.	0.020
Atrazine	N.D.	0.008	DDE p,p	N.D.	0.002
Azinphos methyl	N.D.	0.015	DDT p,p	N.D.	0.040
Azoxystrobin	N.D.	0.001	Deltamethrin	N.D.	0.010
Bendiocarb	N.D.	0.002	Diazinon	N.D.	0.001
BHC alpha	N.D.	0.002	Diazinon oxygen analog	N.D.	0.010
Bifenazate	N.D.	0.100	Dichlorvos (DDVP)	N.D.	0.011
Bifenthrin	N.D.	0.005	Dicloran	N.D.	0.003
Bifenthrin	N.D.	0.025	Dicofol o,p	N.D.	0.010
Boscalid	N.D.	0.020	Dicofol p,p	N.D.	0.003
Bromacil	N.D.	0.025	Dieldrin	N.D.	0.005
Buprofezin	N.D.	0.100	Difenoconazole	N.D.	0.050
Captan	N.D.	0.010	Diffubenzuron	N.D.	0.020
Carbaryl	N.D.	0.003	Dimethoate	N.D.	0.023
Carbendazim (MBC)	N.D.	0.005	Dimethomorph	N.D.	0.056
Carbofuran	N.D.	0.001	Dinotefuran	N.D.	0.030
Carfentrazone ethyl	N.D.	0.003	Diphenamid	N.D.	0.001
Chlorantraniprole	N.D.	0.010	Diphenylamine	N.D.	0.020
Chlordane cis	N.D.	0.001	Disulfoton sulfone	N.D.	0.025
Chlordane trans	N.D.	0.005	Diuron	N.D.	0.025
Chlorfenapyr	N.D.	0.003	Endosulfan I	N.D.	0.005
Chlorothalonil	N.D.	0.004	Endosulfan II	N.D.	0.001
Chlorpropham (CIPC)	N.D.	0.050	Endosulfan sulfate	N.D.	0.001
Chlorpyrifos	N.D.	0.003	Endrin	N.D.	0.020
Chlorpyrifos methyl	N.D.	0.001	Epoxiconazole	N.D.	0.003
Chlorthal (DCPA)	N.D.	0.010	Esfenvalerate	N.D.	0.005

LOD - Limit of Detection (Limit of Detection = Limit of Quantitation), N.D. - Not Detected, N.A. - Not Analyzed

The fee for the laboratory services provided above is \$324.00.

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Approved by:

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Roger Simonds, Laboratory Manager

7/1/11

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Thank You



For more information concerning pesticide residue testing services, please contact:

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- Gastonia, NC 28054

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QUESTIONS?