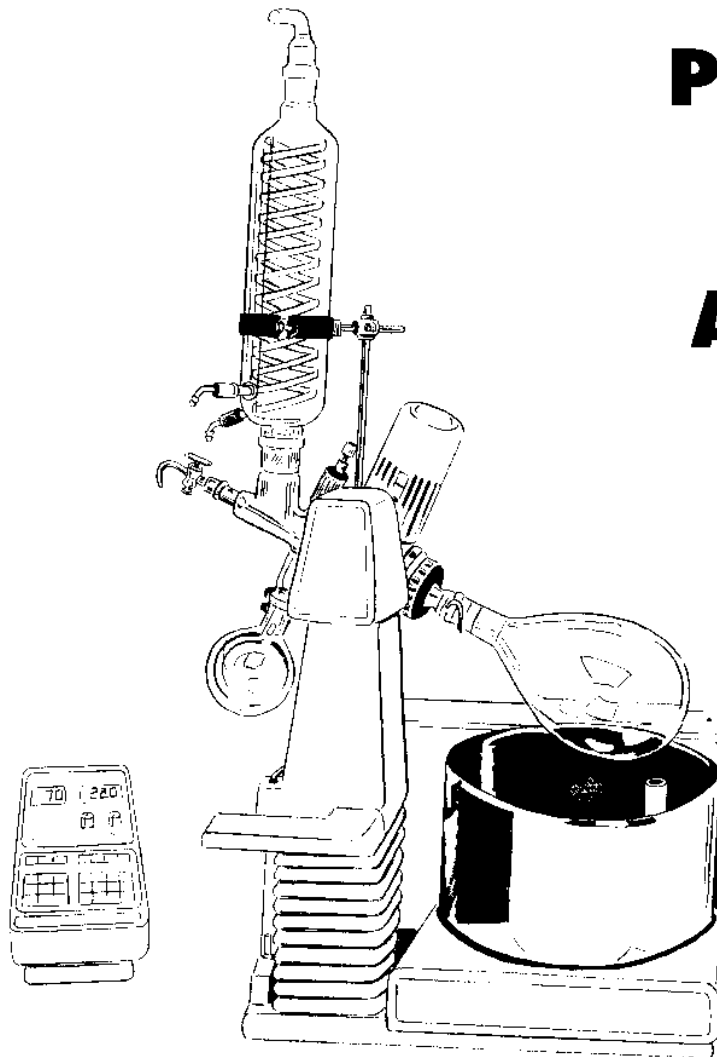


# A&L REFERENCE GUIDE



The Soil Experts.



## PESTICIDE RESIDUE ANALYSIS

### **A & L LABORATORIES**

**A & L Analytical Laboratories, Inc.**  
Memphis, Tennessee

**A & L Great Lakes Laboratories, Inc.**  
Fort Wayne, Indiana

**A & L Eastern Agricultural Laboratories, Inc.**  
Richmond, Virginia

**A & L Plains Agricultural Laboratories, Inc.**  
Lubbock, Texas

**A & L Western Agricultural Laboratories, Inc.**  
Modesto, California

**A & L Southern Agricultural Laboratories, Inc.**  
Pompano Beach, Florida

**A & L Canada Laboratories East, Inc.**  
London, Ontario, Canada

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Atlantic, Iowa

## INTRODUCTION

Pesticides are being used today to control a variety of insects, weeds and plant diseases. Some of these same pesticides can act as toxins to plants, animals and humans when found in sufficient concentrations as residues. Residues can be found in plant materials, water, soil, meat and dairy products. Samples to A & L Laboratories can be effectively analyzed for organic compound residues.

## GENERAL INFORMATION

When samples arrive, an appropriate amount is put through an extraction procedure using a variety of solvents and techniques to separate organic compounds from the sample material. After extraction the sample is passed through a chromatography clean-up column which removes most lingering contaminants. Evaporation is used to concentrate the extract and the sample is then ready for analysis by gas chromatography, high pressure liquid chromatography, or spectrophotometry.

## GENERAL SAMPLE COLLECTION INSTRUCTIONS FOR RESIDUE ANALYSIS

Good sampling techniques are extremely important when taking samples to be used for residue analysis. For liquids one sample is usually sufficient because we can assume the residue is mixed throughout the material. On the other hand in soils, feeds and plant tissues, it is important that the samples represent a **good cross-section of the material being sampled**, or of the area when you suspect the presence of pesticides. In such cases 15 to 20 samples from randomly selected areas should be thoroughly mixed and a generous sample drawn from this composite.

The table on page 2 shows amounts required of various types of samples. If possible, include more sample than is required. Should more than one test be desired you may need to submit additional amounts. Please inquire before shipping. **If potential litigation is involved, follow proper chain of custody procedures.**

As indicated in the table, some pesticides need special handling, and these samples should be frozen immediately after collection, and shipped in a styrofoam container packed in ice. Special containers for shipment of frozen samples are available from A & L Labs. Due to the cost of these

containers, we will bill you for these containers as they are shipped out from the lab and credit your account for the same amount when the containers arrive back at the lab with your samples.

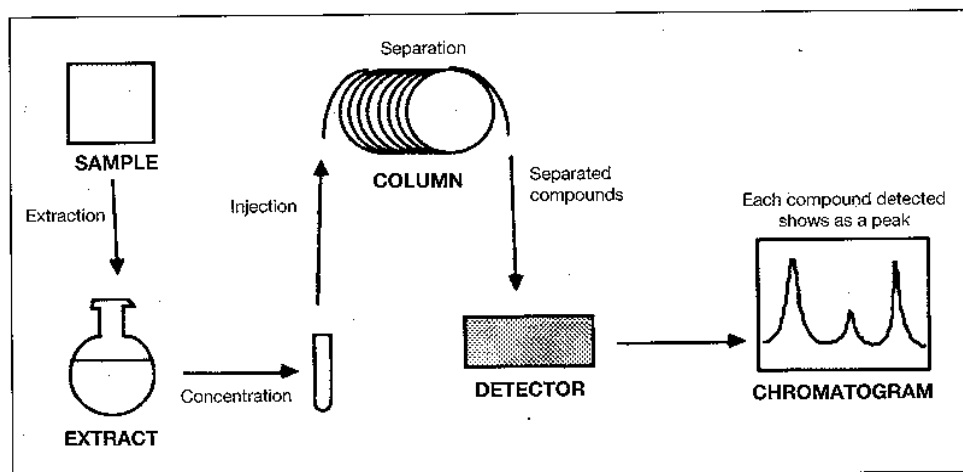
Label samples with the RESIDUE ANALYSIS INFORMATION STICKER provided by A & L (*see illustration*). The use of this sticker allows your sample to be routed and processed more rapidly, shortening turn-around time. The sticker includes three parts:

- The portion labeled RESIDUE ANALYSIS INFORMATION STICKER - A & L LABORATORIES should be attached to the shaded area of a completed Information Sheet.
- Attach the portion marked RESIDUE ANALYSIS (Special Handling) to the sample container.
- Place the portion titled RESIDUE ANALYSIS ENCLOSED on the shipping box.

**NOTE:** If a Basic Soil Test or Nematode Test is required, a **separate sample** should be submitted to insure that enough soil is received.

## SAMPLING PROCEDURES FOR RESIDUES IN SOILS

- Using a soil tube, obtain 15-20 cores. Be sure to take the cores to the **depth of incorporation** of the chemical in question. It is important to remember, the analysis is no better than the sample submitted. Samples taken too deep will, in effect, dilute the residue concentration. Samples too shallow may not include all of the residue present.
- Samples obtained with a shovel should include around 2 cups of soil taken to the depth of incorporation. However, this method is generally not recommended for taking residue samples because of the large amount of soil usually taken which must then be subsampled.
- Sample from several areas, but avoid any areas which might cause misrepresentation, such as "spill" areas and overlaps at the ends of the field.
- When a routine fertility analysis is wanted in addition to a residue analysis, **two separate soil samples should be sent**. Soils that have been allowed to dry cannot be used to accurately quantitate the presence of residue.



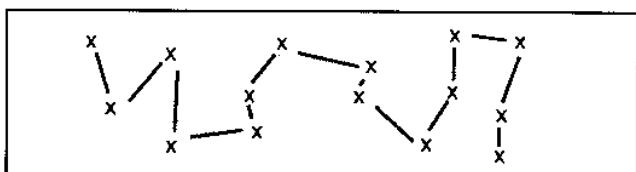
## AMOUNT OF SAMPLE NEEDED FOR COMMON HERBICIDE & INSECTICIDE ANALYSES\*

Plants	Soil	Water	Sludge	Fertilizer Dry	Fertilizer Liquid	Formulation Dry	Formulation Liquid	Animal Tissue	Feed
1 lb.	1 cup	1 quart	1 quart	1 cup	1 quart	1 oz.	1 oz.	1 lb.	2 cups

**Special Shipping Instructions:** Banvel, Bladex, Command, Chem Hoe, CIPC, Eradicane, Ramrod, Sencor, Treflan, Vernam, 2,4-D, 2,4,5-T, Bromacil and Furadan samples should be frozen immediately after collection (except water) and shipped in a styrofoam container packed in ice.

\* Analyses can be performed on smaller samples, but sensitivity may be reduced to an unacceptable level.

- Ship as soon as possible to the A & L Laboratory in your area. In the case of delayed shipping, keep the samples cool or in the freezer.



*Random Sampling For Best Representation*

### SAMPLING PROCEDURES FOR PLANTS AND FEEDS

- Plant samples to be tested for pesticide residues must be sampled immediately when contamination is suspected to have occurred. Plants metabolize some pesticides very rapidly, and residues may no longer be present after the plant has wilted or died.
- Metabolism of the plant or volatilization of the suspected pesticide should be stopped as quickly as possible. Chilling or freezing are preferable to drying. Samples should be shipped in ice in an insulated container unless they are dry and will not mold during shipment. Fresh plant material placed in sealed plastic bags will mold and decompose during shipment.

### SAMPLING PROCEDURES FOR WATER AND SLUDGE

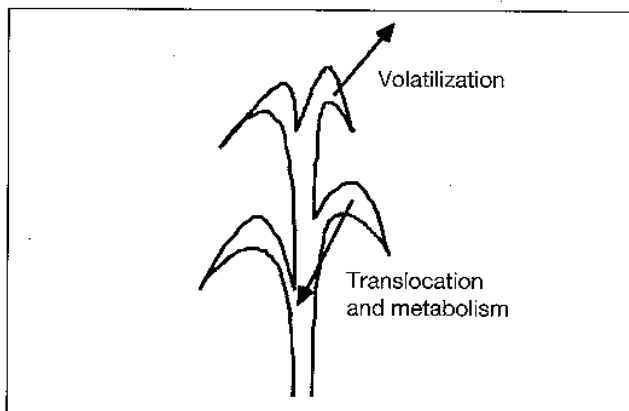
Sample collection should be done with widemouth glass bottles, equipped with screw caps fitted with teflon liners. Amber glass bottles are preferable to avoid the possibility of breakdown of some chemicals by light. The use of a screw cap without a teflon liner may cause contamination of the sample by the liner or adhesive used in sealing the liner to the cap.

Plastic bottles (polyethylene) are not used because traces of plasticizer may be leached from the plastic by the water, and can be a source of analytical interference. Moreover, organics from the sample may be absorbed into the plastic. All samples should be packed in ice and shipped as soon as possible.

At least one quart will be needed for each residue analysis or screen.

### SAMPLING PROCEDURES FOR FERTILIZER AND FORMULATIONS

- Sample collection should be done with a wide-mouth rigid walled container, equipped with a cap allowing a liquid tight seal. Sample containers are available from A & L Laboratories upon request.
- One pint of liquid or two pounds of dry material are sufficient. **Excessively large samples may require special disposal as hazardous waste, and if so will be returned to you by the laboratory.**
- Special care must be taken to obtain a **uniform, representative** sample. Liquids should be thoroughly agitated to eliminate layering, and dry materials should be thoroughly blended, especially if they contain non-uniform sized particles.



*Herbicide Disappearance From Plants*

### SAMPLING PROCEDURES FOR TRANSFORMER OILS

A small sample (1/2 oz.) should be drawn from the transformer and placed in a small vial obtained from the laboratory. Samples classified as hazardous waste due to PCB content will be returned to the submitter.

### FIELD SCREENING TESTS FOR PESTICIDES

A variety of screening test kits are available to test for the presence of pesticide residues. These can be useful for some purposes, but it is important to understand their limitations.

Each kind of screen is sensitive to only certain pesticides or classes of pesticides. It is important to know what

pesticides you are likely to encounter and choose an appropriate screen.

The sensitivity of some screens is much less than the sensitivity of laboratory tests. Many screens will fail to detect levels of pesticides which are highly significant in terms of plant growth, human health, or governmental regulation.

## REPORTING PROCEDURES

After a residue analysis has been completed, the client will receive a residue analysis report. The report will include the following information:

1. Residue analysis requested – the specific residue for which the analysis was done.
2. Amount of residue detected.
3. Sensitivity of the analysis – the lowest level which can be detected. When a report shows a level of “less than 0.02 mg/kg”, it means that the method could have detected 0.02 milligrams of residue in one kilogram of sample, but did not detect any. **Mg/kg is equivalent to parts per million (ppm).**
4. Method used for analysis. Usually this will be gas chromatography (GC).

If soil fertility recommendations are required, a basic soil analysis test S1A should also be run and a Soil Sample Information Sheet should be completed for the sample. A separate sample should be submitted for the Basic Soil Test S1A.

## WHAT LEVELS OF PESTICIDES ARE SIGNIFICANT?

The level of pesticide residue necessary to cause harmful effects depends on many factors:

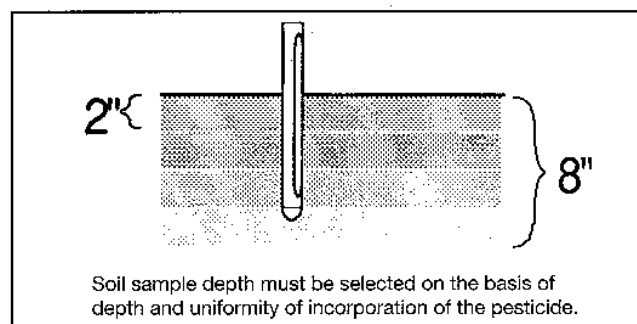
1. **Type of herbicide and intended crop:** For example, legumes, cereal grain crops, and sugar beets are very sensitive to Atrazine. Milo and wheat are very sensitive to Treflan.
2. **Soil Properties:** Soil texture, organic matter, and pH affect the activity and decomposition rate of herbicides. Very high or low pH can reduce activity of some herbicides, as do high levels of organic matter. On the other hand, soil pH 7.3 or above can enhance the activity of some herbicides, such as triazines. Most herbicides show greater activity in coarse-textured soils than in fine-textured soils.

3. **Tillage:** Herbicides near the surface of the soil are dispersed in the tillage layer. Distribution of the herbicide is affected by type and depth of tillage.
4. **Dissipation Rate:** Every pesticide breaks down over time at its own rate, which can be affected by temperature and microbial activity as well as the factors listed above.
5. **Human and Animal Toxicity:** Insecticides and other compounds can be toxic to humans. In many cases, there are legal limits allowed in food or feed products.
6. **Sampling Depth:** Since an acre of soil 6 inches deep contains approximately 2 million pounds of soil, the **level of herbicide in a 6-inch depth sample expressed as mg/kg or parts per million can be converted to pounds per acre by multiplying by two.** Residue in a 3-inch sample, expressed as parts per million, is **equivalent to the same number of pounds per acre** since the sampled layer would be equivalent to one million pounds of soil. An easy formula to convert ppm to pounds active ingredient is:

Sample depth X mg/kg = Lbs. active ingredient per acre

7. **Distribution of pesticides:** In the case of herbicides, shallow incorporation can, over time, concentrate the herbicide in limited areas due to drifting movement of soil. Banding of herbicides has the same effect. In these cases, a soil sample may show a residue level that is considered a marginal risk for a given group, but there could be areas of crop damage due to concentrated areas of herbicide residue.

Taking a greater number of core samples to make a composite will reduce these variations, but analyzing samples from several areas will help reveal whether such distribution variation exists. If all of the samples taken show the same level of residue, the results can be averaged. The sampling plan to follow for any kind of pesticide residue should take into account the risk involved if the residue is not distributed uniformly.



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