



## TESTING FOR HERBICIDE RESIDUES IN SOILS

Herbicides vary in their potential to persist in soil. Herbicides that can persist to the next season may injure subsequent crops and require close monitoring. Two methods used to determine if injurious herbicide residues might exist are a soil chemical test conducted at a laboratory and a bioassay done either in the suspect field or in a warm, sunny indoor location (such as a greenhouse). These tests help predict potential herbicide-residue problems so the grower can make better decisions about crop rotation, herbicide selection, planting date, and other cultural practices.

### SOIL COLLECTION AND PREPARATION

With the lab analysis or indoor bioassay, proper sampling of soil is the first step. The procedures for submitting a soil for laboratory analysis and for conducting an indoor bioassay are similar. These guidelines should be followed:

1. In early to midspring or before planting time, collect representative soil samples from the suspect field. Take samples from several locations in the field. For the bioassay or laboratory analysis, take 15 to 20 soil cores and combine them to make a composite sample. This sample should represent no more than 15 to 20 acres. Enough areas must be sampled to avoid missing locations with high herbicide-residue content. Take separate samples from areas where excessive residues are suspected, such as sprayer turnaround points and end rows. Do not mix these samples with the others. Sample the soil to a 6-inch depth, and divide the samples into 0-to-3-inch and 3-to-6-inch sections for greater accuracy. Be sure to mark on the bags the depths

from which the samples came. About 8 pounds of soil (about 4 quarts) are needed for each bioassay and 2 pounds of soil (about 1 quart) for each laboratory analysis.

2. Sample an area that is not suspect for use as a "check" soil. This soil may be taken from a nearby fencerow or another untreated area. Keep this sample separate from the others. Many laboratories require a check soil.
3. Submit the samples to the laboratory as soon as possible after sampling. If bioassays are to be performed, they should be run on the soil samples as soon as possible after they have been obtained from the field. If samples cannot be assayed immediately, store the soil in a refrigerator or freezer that is not used for food. If samples are stored in a warm environment, herbicide residue may decrease with time.

### BIOASSAY

The bioassay can help predict potential crop injury. The test is inexpensive and can be done with a few simple supplies. A bioassay does not measure the amount of herbicide residue present in the soil, but it may indicate whether or not enough residue is present to injure a sensitive crop.

### FIELD BIOASSAY

A field bioassay is conducted by planting one or more strips of a species sensitive to the suspect herbicide in the field. This procedure can be done in the fall or spring, but it is more accurate if performed closer to the planting of the intended crop. Before planting the

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*The information in this chapter is provided for educational purposes only. Product trade names have been used for clarity, but reference to trade names does not imply endorsement by the University of Illinois; discrimination is not intended against any product. The reader is urged to exercise caution in making purchases or evaluating product information.*

*Label registrations can change at any time. Thus the recommendations in this chapter may become invalid. The user must read carefully the entire, most recent label and follow all directions and restrictions. Purchase only enough pesticide for the current growing season.*

desired crop, allow the test plants to grow and develop symptoms of injury from any herbicide residues. Plant the strips in several locations, if possible, and include an area that is most suspect and an area that can serve as a check. Choose an appropriate species for the bioassay, such as one of the more sensitive ones listed in this chapter. Include several species of differing sensitivity for greater accuracy.

#### **INDOOR BIOASSAY**

The procedures for conducting an indoor bioassay vary, depending on what herbicide residue is of concern. However, for the indoor bioassay, the procedures for soil collection and preparation are the same.

1. For an indoor bioassay, collect the samples and allow them to air dry if needed until they can be worked readily. Do not overdry. If the soil is cloddy, crush the clods into pieces (the size of a pea or smaller). If the soil contains a high amount of clay, the addition of coarse sand (50 percent by volume) improves its physical condition. If sand is added, mix it thoroughly with the soil.
2. Tin cans, milk cartons, and cottage cheese containers are appropriate containers in which a bioassay can be conducted. Punch holes in the bottoms of the containers to allow water drainage. Fill two or more containers (a set) with soil from each sample. Additional containers increase the accuracy of the test. Place the soil samples obtained from the 0-to-3-inch depth in one set of containers; and, in another set, place the soil obtained from the 3-to-6-inch depth. Follow this procedure for the composite sample and the sample taken from areas where excessive residues are expected. In addition, fill a final set of containers with the check soil.

### **TESTING FOR SPECIFIC HERBICIDE GROUPS**

#### **TRIAZINE RESIDUES**

For suspected carryover from triazine herbicides, such as atrazine and Princep (simazine), an oat plant bioassay works best. Place about 15 oat seeds in each container of soil and cover the seeds with about 1 inch of soil. Wet the soil with water, but do not saturate it.

Place the containers in a warm location (70° to 75°F) where they can receive ample light. Sunlight is essential for the development of the plant as well as for inducing symptoms of triazine injury. The container should be watered as needed.

Injury symptoms should become apparent within 10 to 14 days after emergence. Triazine injury is characterized by chlorosis (yellowing), then necrosis

(browning) of leaf tissue. As injury symptoms start at the leaf tip and develop toward the base, a comparison with the plants in the check soil is essential.

If injury appears on the oats, enough herbicide residue may be present to injure a susceptible crop. Planting a more tolerant crop is suggested. In general, the order of susceptibility from most to least susceptible to triazine herbicides is as follows:

Ryegrass > Alfalfa > Oats > Wheat >  
Soybean > Sorghum > Corn

#### **DNA RESIDUES**

If residues from dinitroaniline (DNA) herbicides, such as Treflan (trifluralin) or Prowl (pendimethalin), are suspected, a different assay technique is used. A sorghum or corn-root bioassay is relatively quick and easy to perform.

Wrap a number of sorghum or corn seeds in a moist paper towel and store them at room temperature for 2 to 3 days. This procedure allows the seed to imbibe water and germinate. Once the seed has germinated, carefully place three to five seeds into containers with the suspect soil and the check soil. Cover the seeds with soil to a depth of about 1 inch and leave them for 10 to 14 days, depending on the air temperature. Water the plants as needed but do not saturate the soil.

At the end of the 10-to-14-day period, carefully remove the plants and observe the root formation. DNA herbicides inhibit root development. Symptoms include stunted plants, stubbed roots, inhibited root-hair development, thickened hypocotyls on broadleaf species, and leaves that fail to unroll. If the plants in the suspect soil display any of these symptoms in comparison to the check plants, DNA residues may be present at concentrations high enough to injure susceptible crops. In general, the order of susceptibility from most to least susceptible to DNA herbicides is as follows:

Annual rye > Oats > Sorghum > Corn >  
Wheat > Alfalfa > Soybean

#### **IMAZAQUIN, IMAZETHAPYR, AND CHLORIMURON RESIDUES**

Imazaquin, the active ingredient in Scepter and a component of Squadron, Detail, Steel, and Tri-Scepter; imazethapyr, the active ingredient in Pursuit and a component of Pursuit Plus, Steel, and Lightning; and chlorimuron, the active ingredient in Classic and a component of Canopy, Canopy XL, Concert, and Synchrony STS, have the same mode of action. These herbicides affect root and shoot growth and development. Symptoms of plant injury include inhibited

root development, stunted plants, and interveinal chlorosis or leaf striping. Therefore, a sorghum or corn-root bioassay performed according to the procedure outlined for suspected DNA residue is appropriate. Corn is more sensitive to imazaquin, and sorghum is more sensitive to imazethapyr and chlorimuron. In addition to making root observations, look for stunted shoot growth and interveinal chlorosis or yellowing. Bioassay plants should be grown for 14 to 21 days. The order of crop susceptibility from most to least susceptible to imazaquin, imazethapyr, and chlorimuron is as follows:

*Imazaquin*: Canola > Alfalfa = Corn = Sunflower > Sorghum > Oats > Wheat > Soybean

*Imazethapyr*: Canola > Sorghum > Sunflower > Oats > Wheat > Corn > Alfalfa > Soybean

*Chlorimuron*: Canola > Alfalfa > Sunflower > Sorghum > Corn > Oats > Wheat > Soybean

Introduction and commercialization of Clearfield (CF) corn hybrids (formerly, IMI—IT/IR) resistant to the imidazolinone herbicides provide producers with a viable option for corn production in fields suspected of having soil-residue levels (carryover) of imidazolinone herbicides high enough to cause injury to conventional hybrids. Resistant hybrids that are homozygous (IR) for the imidazolinone-resistance trait have also demonstrated resistance to potentially injurious levels of the sulfonylurea herbicide chlorimuron. If bioassay results show residue levels of imidazolinone herbicides are high enough to cause potential injury to conventional hybrids, you may wish to consider planting one of the imidazolinone-resistant or -tolerant hybrids if corn is the rotational crop of choice.

#### **COMMAND (GLOMAZONE) RESIDUES**

Clomazone, the active ingredient in Command, inhibits the production of photosynthetic pigments in susceptible plants, causing them to emerge lacking green color (that is, they are white or albino). Lower levels of Command residue may appear as a chlorosis or mild bleaching of the plants. Oats or wheat can be used to detect Command residues using the same procedure as was outlined for detecting triazine residues. Bioassay plants should be grown for 10 days to 2 weeks. Susceptible plants that are exposed to significant levels of Command residues will be white, while untreated or tolerant plants will be green. Keep in mind that oats and wheat are usually more susceptible than corn to injury from Command. The order of susceptibility from most to least susceptible to Command residues is as follows:

Oats = Wheat = Alfalfa > Sunflower = Sorghum = Corn > Soybean

#### **OTHER RESIDUES**

Bioassays may be made for other herbicides using similar techniques. If the mode of action of a specific herbicide is known, then a procedure for detecting the herbicide can be developed. For example, if the herbicide is a root meristematic inhibitor (that is, if it stops cell division in the roots), then a root bioassay is the appropriate test. If the herbicide inhibits photosynthesis, then injury symptoms first appear in the leaves. Choose a species that is moderately susceptible to the suspected herbicide, and always include a check soil. Wheat and oats are very good indicator plants for many herbicides but may be more sensitive than the desired crop. Include several species in the bioassay to give a better range of susceptibility. The desired rotational crop is a good bioassay plant to include.

#### **LABORATORY ANALYSIS**

Laboratory analysis involves extracting herbicide from the soil with the use of specialized equipment to detect very small amounts. The amount is expressed in parts of herbicide per million parts of soil (ppm). This measurement can be transposed into pounds of herbicide active ingredient per acre (lb a.i./A) if we assume that an acre of soil weighs 1 million pounds in the top 3 inches and 2 million pounds in the top 6 inches. For a soil sample taken to a 3-inch depth, 1 ppm = 1 lb/A of residue. For a soil sample taken to a 6-inch depth, 1 ppm = 2 lb/A of residue.

A lab report of 0.2 ppm atrazine, then, means that there is 0.2 pound of atrazine per acre if the samples were taken to a 3-inch depth, and 0.4 pound per acre if taken to a 6-inch depth.

The location and concentration of the chemical depend on the herbicide used, the soil type, whether the ground was tilled, and the amount of rainfall since application. In most medium-textured soils (silt loams, silty clay loams, sandy clay loams), the herbicide remains primarily in the top 3 inches unless there was excessive rainfall, the ground was plowed, or the herbicide was deeply incorporated. If the soil has a high sand content (coarse texture), then herbicide leaching may be greater. Movement of the herbicide from the surface soil zone by tillage or by rainfall decreases the likelihood of crop injury. The risk of injury is greater when the herbicide residue is concentrated in the top 3 inches rather than distributed throughout a 6-inch soil depth. Therefore, it is best to sample the 0-to-3-inch and 3-to-6-inch sections separately.

Whether parts per million or pounds of active ingredient of herbicide per acre are used, it is difficult to

translate these units of measure into potential crop injury. Many variables affect crop susceptibility or tolerance, including soil type, crop sensitivity, and environmental conditions after planting. Crop injury is more likely on more coarsely textured soils or under cool, wet weather conditions. Additionally, high soil pH increases the potential of triazine or chlorimuron injury. General guidelines are provided in Table 1, although you are cautioned that crop injury may still occur below these levels.

Laboratories may differ in available tests and in the prices for analysis. The cost can range from \$20 to \$200 per sample for herbicide analysis. Most laboratories can analyze a sample and have the results in 5 to 7 days. Contact your local Extension office for more information on laboratory selection.

### CORRECTING FOR HERBICIDE RESIDUES

If the lab test or bioassay indicates a potential herbicide-residue problem, several steps can be taken.

1. First select a tolerant crop or variety. This selection depends on what herbicide is of concern. Check current herbicide labels for more information on crop tolerance.

2. Tillage can help dilute herbicide in a problem field.
3. Plant the field that concerns you last. Delaying planting allows more time for the herbicide to dissipate.
4. If the triazine herbicides or chlorimuron is suspect, be sure to check the soil pH and adjust your management practices accordingly.
5. If imazaquin or imazethapyr is suspect, check for low soil pH (<5.5). Liming would both benefit crop growth and minimize carryover of these herbicides.

In summary, a bioassay or laboratory test is not 100 percent accurate in predicting herbicide-residue problems. Crop response to herbicide residue depends on various factors, including species and variety, soil type, and environmental conditions after planting. So, predicting crop injury is often difficult. However, using a soil chemical test or bioassay can help in deciding whether a potential problem exists and in choosing the appropriate crop or variety.

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**Table 1. General guidelines for interpreting laboratory analysis**

Herbicide	Safe level*		Crop
	Parts per billion	Parts per million	
Triazine	150–250 <100	0.150–0.250 <0.100	Soybean Alfalfa, oats, wheat
Dinitroaniline	100–200 200–300	0.100–0.200 0.200–0.300	Corn Wheat
Clomazone	50–200 15–100	0.050–0.200 0.015–0.100	Corn Wheat, alfalfa
Imazaquin	2–10 10–30	0.002–0.010 0.010–0.030	Corn Wheat
Imazethapyr	10–30 4–15	0.010–0.030 0.004–0.015	Corn Sorghum
Chlorimuron	1–2 2–5	0.001–0.002 0.002–0.005	Corn Wheat

\*Due to differences in herbicide availability from the soil, "safe" values for herbicide residues differ according to soil type. Low-range values are for coarsely textured soils with low levels of organic matter; higher-range values are for finely textured soils with higher levels of organic matter. 1 ppm = 1,000 ppb.