4.1 INTRODUCTION

This Guideline provides a description of the techniques and sampling strategies commonly used to characterize pesticide dissipation on lawns and turf. Such dissipation data are used in conjunction with concurrent human exposure data to establish chemical transfer coefficients which in turn are used to determine restricted-entry intervals for agricultural situations (e.g., sod farms). Further, for turf pesticides, characterizing residue dissipation rates provides valuable information for posting and notification requirements, where applicable. Lawn surface residue data are also used to determine whether or not a given pesticide may be used without appreciable risk in a residential setting.

4.2 SAMPLE COLLECTION

4.2.1 Test Substance

As stated at 40 CFR 158.390, the test substance to be used for inhalation exposure measurements must be a typical end-use product. Where metabolites, breakdown components, or contaminants of pesticide end-use products pose a potential toxicological concern, investigators may need to consider sampling for them on a case-by-case basis.

In choosing the typical end-use product, factors to consider include (but are not limited to): formulation, irrigation practices (i.e., "watering in"), and concentration of active ingredient in the spray solution. For example, several researchers have found that lawn surface residues are greater following applications of liquid formulations than after granular application (Sears et al., 1987; Cowell et al., 1993). Therefore, for a pesticide available in several different formulations, a liquid formulation (e.g., emulsifiable concentrate) should be chosen for testing. Furthermore, it has been demonstrated that "watering in" immediately after application may move pesticide residues into the thatch where they are less available (Niemczyk and Krueger, 1987). Therefore, a product for which "watering in" is not prescribed for efficacy should be selected for testing. Similar to "watering in," applying a pesticide in a large volume of water may carry the residues into the thatch. Therefore, products which can be applied in a minimal amount of water should be used.
4.2.2 **Timing of Application**

Sampling should be conducted during the intended use season or under climatic conditions that are essentially representative of those encountered during the activity being studied. Applications should be made after mowing and watering. Weather forecasts should be studied, as much as possible, to avoid initiating the testing immediately (e.g., within 24 hours) before a precipitation event. (See Part B, Chapter 2 - Study Design for more information.)

4.2.3 **Pesticide Application Rate and Frequency**

Generally, the end-use product chosen for the study should be applied at the maximum rate specified on the label. In addition to applying the product at the maximum label rate, it is suggested that the product be applied using a lower application rate, if possible. For example, typical rates are often used in cancer assessments (U.S. EPA, 1997). Monitoring at more than one rate will also provide additional information about the relationship between the application and deposition rates. Also, testing at a lower rate may prove to be beneficial in the event that the data from use of the product at the maximum application rate results in an unacceptable risk.

Where multiple applications are recommended, the minimum time interval between applications should be used. Also, testing at a longer time interval between applications may prove to be beneficial if unacceptable risks may result. Registrants should note, however, that if the maximum rates or minimum application intervals are not monitored, subsequent label changes may be necessary. Also, the potential accumulation of residues from multiple applications should be considered. The application method and equipment typical for the selected test substance and intended end-user (i.e., private resident versus commercial lawn care applicator) should be used.

4.2.4 **Sampling Parameters**

The following paragraphs provide a description of: where geographically sampling should occur (i.e., site selection); how long the dissipation must be characterized (i.e., the sampling period); the times within the sampling period that samples should be taken (i.e., sampling intervals); and the number of samples that should be taken at each sampling interval along with a description of where at the sampling location to sample.
4.2.4.1 Number of Geographic Locations

Samples are typically collected from at least three geographically distinct locations per formulation type. This is necessary to ensure that varying climatic conditions, crops, and pest types are represented. For the purposes of selecting these locations for a study, investigators should consider differences in species, climate, and cultural practices within different geographic regions. For example, St. Augustine grass in Florida has a different growth habit than Kentucky Bluegrass in Missouri, or Bermuda grass in California. In addition, the amounts of rainfall, air and soil temperatures, mowing intervals, and other factors differ within these regions. (See Chapter 2, Study Design, for more guidance.)

4.2.4.2 Sampling Period

Data should be collected in a manner that characterizes the dissipation mechanisms for the compound (e.g., three half-lives). Further, the sampling period should be reflective of the exposure conditions and toxicological endpoint of concern (i.e., acute or chronic). Typically, dissipation rates are characterized for at least 72 hours postapplication unless the compound has been found to fully dissipate in less than this time period. EPA has observed that this sampling period is usually adequate for characterizing pesticide dissipation under most use conditions. Please note, however, that for more persistent pesticides, a longer sampling period may need to be used. In addition, the recent passage of the Food Quality Protection Act may result in the need for monitoring over longer periods to allow for the aggregation of long-term exposures from multiple sources (i.e., dietary plus dermal). Registrants should present the proposed sampling period in the study protocol prior to initiation of the study to ensure that it is agreeable to the Agency.

4.2.4.3 Sampling Intervals

Generally, the length of time between sampling days should be relatively short in the beginning and should lengthen as the study progresses. EPA recommends that samples be collected prior to application on the day of application; and on the application day at various intervals after application. For example, sampling at 1, 4, 8, 12, 24, 48, and 72 hours after application may be appropriate for residential turf. Please note that for certain pesticides (e.g., one that degrades quickly) shorter sampling intervals may suffice. However, the sampling should continue for 72 hours, but all of the samples may not need to be analyzed if the pesticide has fully dissipated (i.e., nondetects for two sampling intervals). On the other hand, sampling may need to continue beyond 72 hours for more persistent pesticides. Special consideration should also be given to pesticides that exhibit biphasic dissipation kinetics.
The proposed sampling intervals should be presented to EPA for review in the study protocol prior to initiation of the study to ensure that they are agreeable to the Agency.

4.2.4.4 Number of Samples and Sampling Positions

The Agency recommends that at least three samples be collected at each sampling interval. Each of the replicate samples should be taken at random from different areas within the treated location. For example, on a given lawn, the triplicate samples should be collected to account for differences in turf density, pesticide application variation, and environmental factors. Control plots should also be established. Sufficient control samples should be collected to ensure that the same bulk sample can be used as a negative control matrix throughout all sample analyses. Additionally, samples from the control plots should be collected at each interval for assessment of the field sample collection and storage procedures.

4.2.5 Sampling Techniques

The measurement of lawn surface residues is a relatively new area in the field of exposure assessment. The various techniques that have been employed by researchers since the early 1980s are mostly modifications of agricultural dislodgeable residue techniques or techniques being developed for indoor surface sampling.

Presently, EPA does not have sufficient information to recommend one sampling technique as a better measure over the others. However, with the research currently underway, the Agency will continually improve its ability to assess each method. As research on the following methods (especially as they apply to lawn surface residue sampling) is an ongoing effort within EPA and the chemical industry, the importance of reviewing the most recent published literature before selecting a method cannot be over emphasized. Regardless of the residue sampling method selected, a relationship must be proposed between the residue data and dose during one or more defined activities. Such a relationship is usually obtained via the transfer coefficient generated from the concurrent collection of residue and exposure data, as described in Part B, Chapters 7 through 10. Also, study investigators are encouraged to propose new methodologies for estimating exposure from lawn surface residues and to validate existing methods. The selected methods must satisfy specific performance criteria as described in Part C, Quality Assurance and Quality Control.

The following is a brief description of each available method. Detailed descriptions of these methods may be found in the literature and in "Methods for Assessing Residential Exposure to Pesticides," (U.S. EPA, 1994).
4.2.5.1 *Dislodgeable Residue Technique*

In the dislodgeable residue technique, all grass is obtained from randomly selected defined areas of the treated plot. The grass clipping samples may be weighed, but the residues must be dislodged in the field within 4 hours of sampling. The residues are dislodged by washing (i.e., consistently shaking for a set period of time, for example 10 minutes) the grass clippings in a detergent or surfactant solution (see Section 3.2.8 for further details) (Goh et al., 1986a; Goh et al., 1986b). Prior to the study, multiple grass clipping samples (a minimum of three samples) must be obtained from the test plot to establish a correlation between leaf surface area and weight. This correlation is established by weighing fresh grass clippings that have been placed on a template of known surface area. Multiple surface areas (and, therefore, multiple weights) must be tested to establish the correlation. The weights tested should bracket the anticipated sample size for the dislodgeable residue testing. While determining the weight/surface area correlation, it may be necessary to correct for moisture losses occurring while grass leaves are being arranged on the templates (Hurto and Prinster, 1993). Refer to Part B, Chapter 3 for further information on DFR dissipation study techniques.

4.2.5.2 *Cheese Cloth Wipe Technique*

There are various approaches to wipe techniques for determining transferable lawn surface residues. One technique described in the published literature involves a person scuffling forward and backward over a designated area of treated turf (Sears et al., 1987). More specifically, the sampler dons a pair of boots. The boots are then covered; first with protective plastic and then with multiple layers of cheese cloth that have been moistened with distilled water. Another technique that may provide better reproducibility is to use platforms (e.g., cooking pans) attached to the bottom of the feet. The sampling material (e.g., cheese cloth) can be attached to the underside of the platforms. The spreading of weight allows for greater distribution of weight (i.e., less pressure) and more uniform results among investigations. The sampler then scuffles forward and backward over a 1 m² area for a specified amount of time (e.g., 1 minute). One replicate consists of triplicate samples (i.e., three 1 m² samples) per sampling interval. The cheese cloth is then removed from the sampler, the excess, unexposed material is cut away, and the remainder is transported, on ice, to the laboratory for extraction and analysis.

4.2.5.3 *Polyurethane Foam Roller*

The polyurethane foam roller (PUF roller) was designed to measure transferable residues from contaminated surfaces that a child may contact during various activities (i.e., crawling) (U.S. EPA, 1994; Hsu et al., 1990). Originally, the device was designed and tested on indoor surfaces (e.g., vinyl flooring). More recently, the design of the PUF roller has been altered to facilitate use on turf. (See Figure B4-1.) In general, the PUF roller device consists of a PUF ring (8.9 cm outside diameter x 8 cm long) that is fitted around an
aluminum or stainless steel roller. This roller is attached to the end of a wheeled, forked handle. The device is weighted (via the stainless steel roller or weights attached to the forked handle) to exert a pressure of 7,300 Pa while rolling (approximating the pressure of a crawling or standing child). The roller is pushed over a specified area of treated turf to sample for surface residues. After sampling, the PUF ring is transported, on dry ice, to the laboratory for extraction and analysis. To simulate the moistness of the human skin, the PUF may be moistened with water.

4.2.5.4 California Cloth Roller

As is the case for the PUF roller, the California cloth roller was originally designed to measure residues that may be dislodged by a child in contact with indoor surfaces (U.S. EPA, 1994; Ross et al., 1990). (See Figure B4-2.) However, this sampling technique may be applied to turf with minimal modifications. In general, a sheet of percale cotton/polyester cloth is placed over a specified area of the treated lawn. A sheet of protective plastic (or clean cotton toweling) is then placed over the cloth. When the sheets are in place, a weighted foam covered roller (similar to a baker's rolling pin or a paint roller) is rolled over the entire covered area 10 times. The percale cloth is then collected and transported to the laboratory, on ice, for extraction and analysis.

Recently, the Outdoor Residential Exposure Task Force developed a modification of the California Cloth Roller technique for use outdoors on turf (Johnson, 1998). The roller is a 4-inch diameter by 24-inch long PVC pipe. The outside of the roller is wrapped with half-inch polyurethane foam sheet, or equivalent pipe insulation, for cushioning and traction. Enough weight is added to the inside of the roller to bring the total weight (excluding the handle) to 32 pounds, which is evenly distributed across the length of the roller. A handle is added to the roller. A rectangular-shaped frame is made of PVC material with inside dimensions of 24.5 x 36 inches. A 27 x 39 inch piece of 100 percent cotton cloth with a 200 thread count is secured to the frame with clamps. A piece of clear plastic, large enough to completely cover the cloth, is then placed completely over the cloth and also secured with the clamps. The frame assembly is placed in the plot with the cloth sampling media in contact with the turf. The frame is secured in place with spikes in each of the four corners of the frame to keep it from moving during sampling. The roller is placed just inside of the frame. Using the frame as a guide, the roller is pushed to the far end of the frame and pulled to the original starting point a total of five times. No downward pressure is exerted on the roller itself. The roller and frame are removed. Visible debris such as grass, thatch, granules, etc. are removed from the cloth sheet because this technique is designed to measure chemical residue which transfers to the cloth, not residue that adheres to particulate matter. The cloth is analyzed for chemical residue and the plastic is discarded. The roller and frame are reusable, provided they are decontaminated between sampling events.
Figure B4-1. PUF Roller Sampling Instrument
Figure B4-2. California Cloth Roller Sampling Device
4.2.5.5 Drag Sled

As with the two roller techniques, the drag sled method (also called the Dow sled) was originally designed for sampling indoor surfaces (U.S. EPA, 1994). (See Figure B4-3.) However, it has also found application in outdoor grassy areas (U.S. EPA, 1994; Vaccaro et al., 1993). This technique consists of dragging a weighted plywood block through a fixed area of treated turf. The block is 9 in² (3” x 3”) in area and contains a removable denim pad attached to the underneath side. The weight placed on the block (usually a lead ball) can be varied, but the original testing (as with the roller techniques) has focused on the pressure exerted by a crawling or standing child. After sampling, the denim pad is removed and transported, on ice, to the laboratory for extraction and analysis.

4.2.6 General Considerations for Field Sample Collection

Control or background samples should be collected from the test plot prior to application of the test substance. Sufficient control samples should be collected so that fortified controls can be prepared on each sampling day. These fortified controls should be packaged, transported, stored, and analyzed concurrent with the dislodgeable residue samples. Please see Part C for detailed considerations on Quality Assurance and Quality Control recommendations.

4.3 SAMPLE STORAGE

Lawn surface residue samples and extracts should be stored in a manner that will minimize deterioration and loss of analyte between collection and analysis; more detailed information on sample storage is provided in Part C, Quality Assurance and Quality Control. The stability of other residues collected on sampling materials (i.e., cheese cloth, denim) should be determined and extractions of them should be conducted within their holding time limitations. The study investigator is responsible for demonstrating the stability of the samples under the storage duration and conditions used.

4.4 SAMPLE ANALYSIS

Pesticide residues should be dislodged from grass clippings within 4 hours of sample collection. Other transferable method samples should be stored appropriately until analysis. Validated methods of appropriate or sufficient sensitivity are needed for all sample analyses. See Part C, Quality Assurance and Quality Control for more detailed information on sample analysis.
Figure B4-3. Dow Drag Sled Sampling Device
4.5 CALCULATIONS

Refer to Part D of this document for a description of the calculations needed for estimating dissipation rates, exposure, and risk.

4.6 DATA PRESENTATION

Lawn surface residues from studies using the dislodgeable foliar residue and transferable residue techniques should be expressed as the amount of pesticide per leaf surface area, and as the amount of pesticide per area of lawn sampled as appropriate (i.e., leaf surface area is only for dislodging approach). For the other techniques described in this chapter (i.e., wipe, roller, and drag sleds), results should be reported as mg or µg of pesticide active ingredient per m² or cm² of lawn sampled. These data should be reported in tabular form for each sampling day. In addition, the best fit dissipation curve should be plotted (typically log-linear) with lawn surface residues on the Y-axis and time on the X-axis. Also, if the dislodgeable residue technique is used, the weights of various surface areas of grass clippings should be reported in tabular form followed by the regression analysis conducted to establish the surface area to weight correlation. Distributional data should be provided, to the extent possible.
REFERENCES FOR PART B, CHAPTER 4


