

Soil Sampling Guidance

Guidance Document 11

The following is Minnesota Department of Agriculture's (MDA) recommended guidance for collection of soil samples at agricultural chemical incident sites.

SAMPLE COLLECTION PROCEDURES

Two kinds of soil samples, composite and discrete, are usually collected in a typical agricultural incident investigation. In addition, there are sampling procedures specifically designed for sampling stockpiles.

I. COMPOSITE SAMPLES

A. Subsample Collection

During an investigation of agricultural chemical releases to soil, the MDA usually requires collection of composite samples to characterize a large area of near-surface soil in potentially contaminated areas (see MDA guidance document [GD9 Remedial Investigation and Work Plan](#)). A composite soil sample consists of several subsamples that are thoroughly mixed together to create one sample for analysis. For investigating agricultural chemical releases to soil, the MDA requests that composite samples be created from equal volume subsamples collected from:

1. Three to six equally spaced locations within a 15-foot diameter sampling area; and
2. A common 6-inch-thick vertical depth interval.

The exact size and shape of the sampling area may be adjusted to meet site specific conditions. The exact size and shape of each sampling area must be discussed in the work plan. Regardless of sampling area shape and size, exact subsample location must be well-documented as described in a subsequent section.

All subsamples used to create a given composite sample must be collected from the same 6-inch depth interval. Do not create a composite sample from subsamples collected over different vertical intervals (e.g., 0 to 6 inches, 2 to 2.5 feet, and 4.5 to 5 feet) or a long vertical depth interval (e.g., 0 to 2 feet or 1 to 4 feet, etc.). The MDA requires that surficial composite samples in non-graveled high-risk areas be collected from the surface to a depth of 6 inches, and in loose graveled areas from a depth interval of 0 to 6 inches below the base of the gravel. In addition, a subsurface composite sample must be collected from a 6-inch-thick depth interval of 2 to 2.5 feet below the ground surface or the base of gravel. All sample depths must be referenced from the ground surface for sample identification purposes.

The methods used to collect composite samples for characterizing soil stockpiles are discussed in Section III.

B. Creating a Composite Sample

Use a new pair of disposable gloves for each composite sample to prevent cross contamination of the sample. Create a composite sample from the subsamples using the following procedure:

1. Combine all of the subsamples in a large clean stainless-steel mixing bowl or disposable aluminum pan;
2. Decant or drain away any liquids;
3. Remove large stones, sticks and vegetation;
4. Thoroughly mix the subsamples together with a clean stainless steel or disposable spoon;
5. Transfer an adequate volume of the composite sample to a lab clean amber glass jar with a Teflon lined lid; and
6. Wipe the threads, then cover, label and seal the container.

II. DISCRETE SAMPLES

A discrete sample is a sample collected from one specific horizontal location and vertical interval. A 6-inch vertical sampling interval is recommended. If there is free liquid, large stones, sticks or vegetation in the sample, put the sample in a clean stainless-steel mixing bowl or disposable aluminum pan and remove them. Otherwise, a discrete sample may be transferred directly from the sampler or sampling location to the sample container using a clean stainless-steel or disposable implement. Wipe the threads of the sample container, then cover, label and seal the container.

Collect samples from split barrel samplers or hand augers starting at the bottom of the split barrel core or hand auger bucket and continuing upward only until the required volume of soil is obtained. In any case, be extremely careful that slough does not come in contact with or become part of the analytical sample, especially when sampling from a hand auger. Use a new pair of disposable gloves at each sampling location to prevent cross contamination.

As discussed in MDA guidance document GD9 Remedial Investigation and Work Plan, the MDA requires collection of a discrete sample from the 4.5 to 5-foot depth interval, near the center of composite sampling areas, in order to provide vertical delineation of contamination. Discrete samples may also be required for horizontal delineation of contamination when collecting samples from individual borings and for samples analyzed by a field screening procedure.

III. STOCKPILE SAMPLES

Sample stockpiles using the following procedure. First determine the number of composite samples required based on the volume of soil in each stockpile as follows:

| Volume of Soil in Stockpile (cubic yards) | Minimum Number of <u>Composite Samples</u> |
|--|---|
| <200 | 1 |
| 200 - 500 | 2 |
| 500 - 1000 | 3 |
| 1000 - 2000 | 4 |
| Each additional 2000 cu. yds. | 1 |

- A. Divide each stockpile into sections of approximately equal volume, with one section for each composite sample;
- B. Conduct 4-6 borings for each composite sample and collect 1-3 subsamples from each boring (the recommended locations for the borings and depths of the subsamples are shown on Figure 1, attached);
- C. Use Figure 1 as follows: each boring location is marked by an "x" and is identified by one or more of three letters, B, "M" or "T" (a "B" means collect a subsample from the bottom third of the soil pile, a "M" means collect a subsample from the middle third of the soil pile, and a "T" means collect a subsample from the top third of the soil pile);
- D. In general, ensure that all depths and locations of the stockpile are sampled (this may require digging into the center of the stockpile to collect samples from close to the bottom);
- E. Collect all subsamples from a depth of greater than one foot below the pile surface;
- F. All subsamples must be of equal volume; and
- G. Create the composite samples from the appropriate subsamples as described in the Section I under "Creating a Composite Sample" (above).

IV. DUPLICATE SAMPLES

The MDA generally requires collection of duplicate samples: one for every ten samples or less submitted for laboratory analysis. A duplicate sample must be submitted to the laboratory as a "blind" sample and be reported to the MDA as a duplicate sample. Also, the MDA will occasionally request split samples so that independent or additional analyses can be conducted by the MDA.

A duplicate sample may be created by splitting, collecting a field duplicate, or cutting a core down the vertical axis. Split samples are created by sieving the soil through a laboratory cleaned number ten (#10) slot sieve and thoroughly mixing the sieved soil prior to splitting. Duplicate soil samples created from soil that has been mixed but not sieved must be identified as "field duplicates" and are useful as an analytical confirmation method and should provide similar analytical results. It is often difficult to create totally homogeneous split soil samples in the field, particularly for wet or fine-grained soil and it may not be possible to split cohesive soils (clay) in the field. As an analytical confirmation method, cut clay cores down the vertical axis into halves for separate analysis. Core halves are not considered split samples.

EQUIPMENT AND DECONTAMINATION

Re-usable sampling equipment must be made of glass, stainless steel, Teflon, or other inert material. Be sure to clean reusable, shovels, picks, hand augers, split tube samplers, stainless steel bowls or spoons and any other equipment that comes in direct contact with the sample, between each sample. All subsamples collected for a single composite sample are considered one sample unless the subsamples are used for both discrete and composite samples.

Clean sampling equipment using the following procedure:

1. Using a non-phosphate soap and clean potable water solution, wash the equipment to remove all visible soil particles, changing the wash water at regular intervals or between borings when using a drill rig. Do not use water from contaminated or onsite wells. The wash basin must be steel or another inert material, not plastic;
2. Rinse with potable water to remove all soap;
3. Rinse with acetone (preferred) or methanol. Wiping the equipment with an acetone or methanol saturated towel is acceptable but dispose of the towel after each use;
4. Triple rinse with deionized water. Deionized water can usually be obtained from the laboratory. If deionized water is not available, distilled water may be used;
5. If time allows air dry; and
6. Wrap in aluminum foil or other suitable material, or store on a clean surface in a protected area until used.

Alternatively, disposable plastic and PVC materials may be used. Replace disposable equipment between samples.

For drilling equipment, clean all downhole sampling equipment (e.g., split spoon) as described above between samples. Other downhole drilling tools and auger flights must be cleaned as described above, or by steam cleaning or high-pressure hot water wash between each boring.

Laboratories can provide guidance on method appropriate sampling containers. Sampling containers may be purchased directly from laboratory equipment and supply vendors. However, most commercial laboratories will provide sampling containers when conducting the analyses. In general, canning jars, plastic jugs, paper bags, plastic bags, etc. purchased at local grocery stores, hardware stores, etc., are not considered appropriate sampling containers.

DOCUMENTATION PACKAGING AND SHIPPING

Keep a precise record of the distance from each sample location (including individual subsample locations within each composite sampling area) to two permanent immobile objects so that sampling areas can be easily and exactly relocated. Record the sample description including soil type, grain size, color, moisture content, density, and orders and classify soils according to MSTM D2487/D2488. In addition, photographs - annotated with the date, photographer, sample number and orientation - of the sample area, taken after the samples have been collected, are recommended.

Include the following information on the sample label:

1. The site name;
2. Sample location and depth;
3. Date collected;
4. Analysis requested; and
5. Name of the person collecting the sample.

For samples that will be submitted to an MDA approved commercial laboratory, MDA staff will usually approve a procedure whereby individual sample bottles are stored and transported to the laboratory in a second sealed container such as a cooler. Use a chain of custody procedure for all samples. Include the sample number, location and depth for all samples on the chain of custody form. Submit the chain of custody form to the laboratory with the samples.

Keep the samples cool. Clean freezer packs are recommended. If ice is used it must be double wrapped in plastic to keep the sample labels and seals from getting wet.

Soil samples which are not analyzed immediately, (i.e., within a few days), may be stored frozen for up to six months under proper chain of custody. Do not dispose of stored samples without MDA staff approval, including the portions of samples remaining after analysis.

All samples must be collected, transported and stored in accordance with all federal and state applicable rules, statutes or regulations. Any sample being shipped by common carrier or through the mail must comply with the United States Department of Transportation Hazardous Materials Regulation (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring compliance with applicable regulations.

GENERAL INFORMATION

Safety is always the highest priority at any site. If for any reason the procedures discussed in this or other MDA guidance documents cannot be implemented safely, MDA staff will consider proposed alternative procedures.

MDA staff must pre-approve all investigation or cleanup activities at agricultural chemical incident sites except for those activities immediately necessary to minimize or abate contamination related to a spill. The MDA's pre-approval process is discussed in MDA guidance document GD9 Remedial Investigation and Work Plan.

The MDA has prepared standard analytical lists for use in pesticide incident investigations (GD26 Analytical List for Pesticide Incident Investigations). These lists must be adapted to site specific analytical requirements. QA/QC plans and analytical methods for commercial laboratories must be approved by MDA (GD24 Fixed Base Laboratories Quality Assurance/Quality Control Plans). A List of commercial laboratories that have approved QA/QC Plans and analytical methods on file with the MDA is available (GD23 Pre-approved Commercial Laboratories: Fixed Base and Mobile).

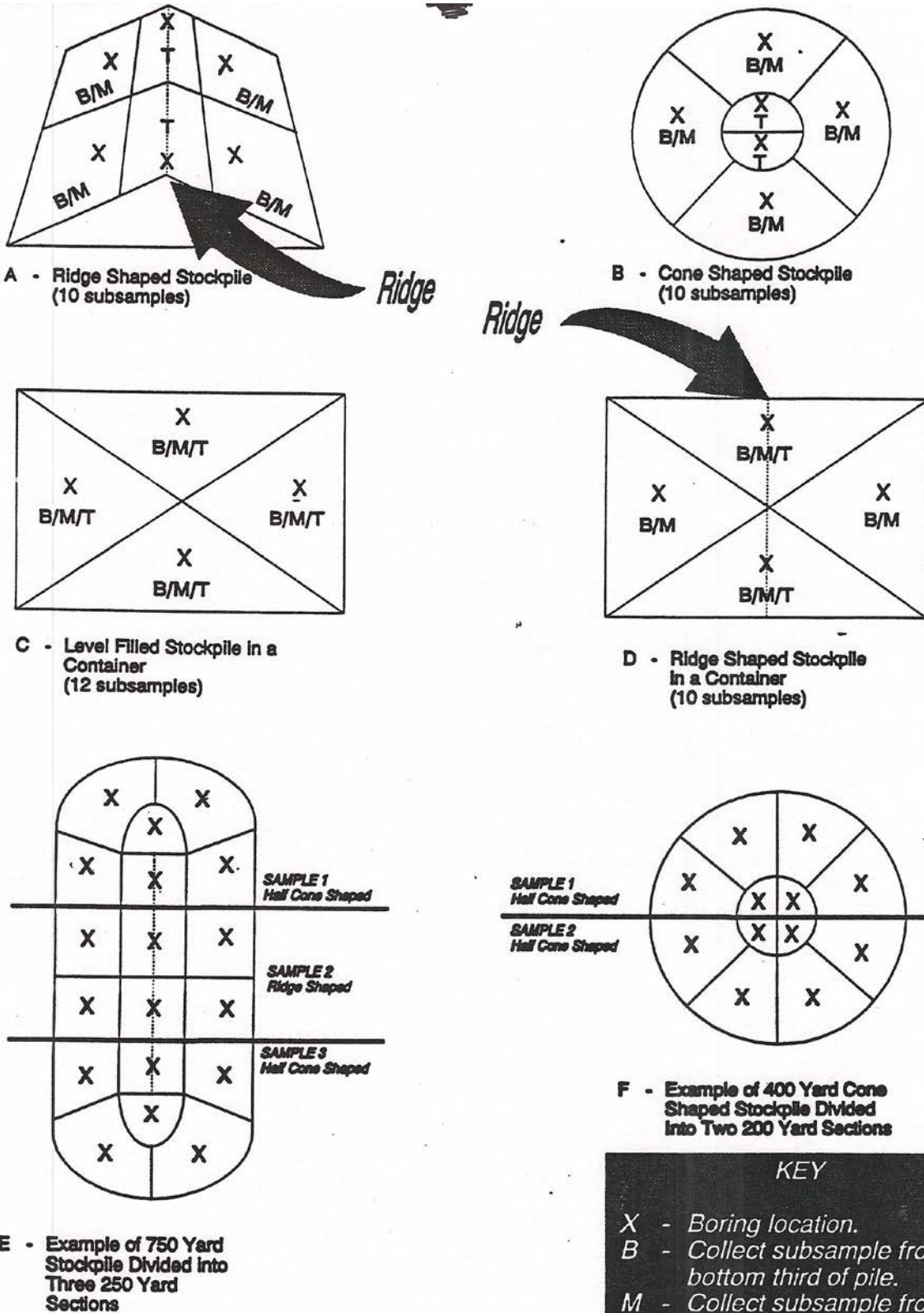


FIGURE 1. MDA Recommended Sampling Points for Stockpiles