

Sample Preparation

Following proper procedures in obtaining and preparing a sample is essential for an accurate analysis on any given lot of feed, hay, silage, etc. No analysis can be more accurate than the sample used for making it. **No information is better than misinformation.** Steps must be taken to correctly obtain and prepare a sample for analysis.

Basic Sampling Rules:

1. The gross sample is supposed to be a replica in composition and particle-size distribution of the entire lot. If it does not represent the entire lot, all further work to reduce it to a suitable size for analysis are a waste of time.
2. Size of the sample must be adequate depending on what is being measured as well as the level of contaminants.
3. The sample must be representative and reproducible. In static systems, multilevel sampling must be made.

Sampling Nonhomogeneous Solids:

1. A gross sample is taken.
2. The gross sample is reduced to a representative laboratory sized sample.
3. The sample must be prepared for analysis.

Coning and quartering:

This method is used when very large lots are to be sampled. The first sample is formed into a cone, the next sample poured on the apex of the cone, mix and flatten, reform cone, and repeat as often as necessary. After all samples have been mixed by coning, the mass is flattened and a circular layer of material is formed. Quarter this, and discard three quarters. Repeat until a suitable size for analysis is obtained.

Rolling and quartering:

A representative sample is obtained from the coning procedure, and this sample is placed on a large sheet of paper. The cone is flattened and the entire sample is repeatedly rolled by first pulling one corner of the paper to the opposite corner, then another corner to its opposite corner. The number of rolls depends on the size of the sample, the size of the particles and the condition of the sample. To collect the sample, lift all four corners of the paper and collect the sample in the middle of the paper. Then the rolled sample is flattened and quartered until a suitable size for analysis is obtained.

Sample Sorter/Separator:

A sample is loaded into the top of a metal device with channels pointing in opposite directions. The sample is split into two sections, each falling into pans on the opposite side. This process is repeated by removing one of the pans, redistributing the sample across the top of the separator, and allowing it to divide again. Repeat until the sample size is suitable to work with.

Handling the sample in the Laboratory:

Each sample submitted to the laboratory should be completely labeled so that no question as to its source can arise. Information may include:

1. The number of the sample
2. The experiment identification number
3. The date
4. The origin (technician or graduate student's name)
5. The analyses to be determined

Grinding the sample:

Most solid samples will require some sort of pretreatment before it can be analyzed. It is necessary to produce a material so homogeneous, that any small portion removed for analysis will be identical to any other portion. Another benefit of pretreatment is that the particle size must be converted to a form that it can be readily "attacked" by the reagents used in a particular analysis. This is accomplished by reducing the particle size to a few tenths of a millimeter followed by thorough mixing. The ground sample should resemble a fine powder.

The Nutrition Lab has four grinding mills for sample preparation use. They are:

1. Standard Wiley Mill, No. 3 (Screen sizes 0.5, 1.0, 2.0, 3.0 mm)

The Wiley #3 should be used for grinding large amounts of dry forages (hay, silage, haylage), dried fecal samples, and hard pellets (beet pulps, etc.)

2. Tabletop Wiley Mill (Screen Sizes 10, 20, 40 mesh)

The tabletop Wiley mill is used to grind small amounts of dried forage samples, such as grass clippings, stems, roots, etc.

3. Cyclotec Sample Mill (Screen sizes 0.5, 1.0 mm)

The Cyclotec is used for most feedstuffs, such as grains, supplements, etc.

4. Coffee mill

Coffee mills are used to grind high oil samples, such as sunflower seeds.

Care of Grinders:

These general procedures should be utilized when commencing any sample grinding.

1. Before introducing a sample, the entire grinder must be clean. Spatulas, brushes, and vacuums are available for this purpose. **Failure to clean between samples results in contamination.**
2. When clean, assemble grinder parts. Secure sample bag (poultry bag, Whirl-Pak, glass jar, etc.) to the collection hopper.
3. Turn the motor on (**before adding sample**), then place sample into the upper hopper. Feed sample through the grinder slowly, so as not to overload the motor. It may be necessary to grind some samples through a larger screen first and then through a smaller screen.
4. When grinding is complete, open door and brush all residual material into the catch pan and regrind is necessary.
5. **Thoroughly** clean all part of the grinder. Remove all caked material, paying special attention to the knives, screens, and/or grinding rings. **When finished for the day, clean the machine and the surrounding area.**

Subsampling:

1. Thoroughly mix entire ground sample by:
 - a. Rolling and quartering
 - b. Use a sample separator
 - c. Trap air in the sample bag, rotate and mix sample for 1-2 minutes.
2. Remove portion of sample (50-100 g) to a carefully labeled Whirl-Pak. The remainder may be saved or discarded.
3. Most samples may be stored at room temperature while performing analyses.

Reference

Shugar, G. J., and Ballinger, J. T., Chemical Technician's Ready Reference Handbook. Third Edition, McGraw-Hill, Inc., 1990.