

Sample Preparation, Storage, and Disposal

1. Scope:

This document provides a standard procedure for the preparation, storage, and disposal of feed and fertilizer samples.

2. Safety:

The grinders are dangerous. Read the manual and follow all safety precautions. Do not use without proper training. Debris can be propelled out of the grinder during grinding. Be sure a shield is in place and always wear proper PPE including safety glasses, dust mask, gloves, lab coat, and hearing protection. Long hair should be tied back and no items such as long necklaces or badge lanyards shall be worn when grinding. Other PPE such as a hair net may also be used.

3. Equipment:

Equivalents are acceptable

- 3.1. Large capacity hammer mill (Jacobson model P-88B)
- 3.2. Centrifugal mill (Retsch model ZM 200)
- 3.3. Vibrating sieve
- 3.4. Drying oven (VWR model 1390 FM)
- 3.5. Refrigerator
- 3.6. High capacity balance capable of weighing up to 150 pounds
- 3.7. Shop vac type vacuum cleaner
- 3.8. 16-ounce glass jars
- 3.9. 500mL HDPE bottle
- 3.10. Utility pans

4. Sample Preparation:

For all samples, download the corresponding sample sheet submitted by the inspector via email. See section 5 for gypsum samples, section 6 samples with a request for pesticide analysis, and section 7 for samples with split requests.

4.1. Liquid Samples

Prepare a label containing the lab number, inspector number, and date prepared. For small containers, place label directly on the sample. For large containers, mix the sample thoroughly and pour into a labeled 500mL bottle. Place the sample in the storage area. Samples that may spoil or ferment shall be stored in a refrigerator.

4.2. Solid Samples

For solid samples, label 16 oz. glass jar(s) with the lab number, inspector number, and date prepared. The number of jars depends on the analyses requested and if an unground portion of the sample needs to be retained. See the chart:

Sample type or analysis requested	Save an undried, unground portion	Save a dried, unground portion
Feed	No	Yes
Gypsum*	Yes	No
Microscopy	Yes	No
pH	Yes	No

* Gypsum may require air drying. If so, save an unground dried portion.

If mycotoxin and/or aflatoxin analysis is requested, retain a second ground portion in a labeled plastic deli cup. For microscopy samples where other analyses are requested, save one unground jar each dried and undried material.

- 4.3. Place the entire sample in a large plastic bag and mix thoroughly for ~1 minute. If the sample is too large to mix in a plastic bag, mix it in its original container. If the sample contains both solid and liquid portions, do not mix in a plastic bag. The sample must be pre-dried.
- 4.4. If an unground portion needs to be saved, from the mixed sample fill one of the labeled jars up to ~1 inch from the top of the jar. Place the unground portion in the sample storage area. Note: for small samples, it may not be possible to save an unground portion.
- 4.5. For samples that do NOT require pre-drying, skip to step 4.10 for feed and 4.11 for fertilizer.
- 4.6. Weigh a utility pan and record weight.
- 4.7. Add the remaining sample to the pan. If there is too much sample to fit in the pan, remix the sample and place a representative portion into the pan. Weigh the sample and pan together and record the weight.
- 4.8. Place in a drying oven at ~60°C and allow sample to dry overnight. If sample is dry enough to grind (not moist when stirred), remove from oven and cool the sample to room temperature. If it is not, allow to dry in oven until it is. Weigh the dried sample and pan.
- 4.9. Calculate the pre-dry moisture using the formula:

$$\% \text{pre-dry moisture} = \frac{W - D}{W - P} \times 100$$

Where: W = Weight of wet sample + pan

D = Weight of dry sample + pan

P = Weight of empty pan

4.10. Grinding Feed

Grind the sample (or a representative portion) in either the Jacobson P-88B Hammer Mill or the Retsch ZM 200 Mill using a 0.75mm screen. If the sample does not appear homogeneous, a 0.50mm screen may be used for achieving a finer grind. Feeds containing heat-sensitive analytes or clog the 0.75mm screen may require a larger screen.

4.11. Grinding Fertilizer

Grind the sample (or a representative portion) in the Retsch ZM 200 Mill. For dry, hard samples use a 0.5mm screen. For compost and similar materials, use a larger screen such as a 0.75mm or 1.0mm screen. If the screen becomes plugged during grinding, use the next larger screen.

4.12. Place the entire ground sample in a plastic bag and mix thoroughly.

4.13. Fill one of the labeled jars with ground material, leaving ~1 inch of space between the top of the material and the top of the jar and place the jar in the appropriate storage area.

4.14. The remaining material may be discarded.

4.15. Thoroughly clean the grinder between samples to prevent cross-contamination. Brush and vacuum the grinder and screens to remove any remaining sample. The screen may require washing with soap and water to remove sample. Dry screen thoroughly before use.

5. Gypsum Samples:

5.1. Follow step 4.2.

5.2. Gypsum samples needing pre-drying shall be dried at room temperature. **Do not dry gypsum samples in the oven.**

5.3. Place the entire sample in a large plastic bag and mix thoroughly for ~1 minute. If the sample is too large to mix in a plastic bag, mix it in its original container. Fill the jar labeled for the unground portion up to ~1 inch from the top of the jar and place in the appropriate storage area.

5.4. Proceed with step 4.11.

6. Samples with Requests for Pesticide Analysis:

- 6.1. Pesticide analyses are performed by the Pesticide Residue group. Label an additional jar or container.
- 6.2. For liquid samples, pour roughly 200mL into a labeled container.
- 6.3. For solid samples, after the entire sample is mixed in a plastic bag, coarsely grind ~400 grams of sample and place in labeled container.
- 6.4. Complete a pesticide request sheet and deliver sample(s) and sheet(s) to the Pesticide Residue group.

7. Split Sample Requests:

- 7.1. Split samples are requested by the Feed, Fertilizer, and Livestock Drug Regulatory Services (FFLDRS) Branch.
- 7.2. Label a small bottle with the lab number and inspector number and place ~200g of ground sample in the container and seal.
- 7.3. Fill out a chain of custody sheet with sample number, inspector number, date split prepared and initials of preparer.
- 7.4. Notify FFLDRS the split sample is ready so they can pick it up and have them sign the chain of custody sheet.

8. Sample Disposal:

- 8.1. Non-violative samples are retained for 3 months from the date the data is reported to FFLDRS.
- 8.2. Violative samples are retained for one year from the date the data is reported to FFLDRS.
- 8.3. A record is kept of sample numbers and discard date.
- 8.4. Samples may be discarded in the trash.

Approvals:

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