

4 Manure Sampling and Analysis

To calculate manure application rates, an estimate of the nutrient content of the manure must be known. Because manure nutrient concentrations are highly variable (see Section 2), actual manure nutrient analyses are strongly recommended. The only way to determine the level of each nutrient being applied to a field is to submit a representative manure sample for analysis.

Field kits are also available that can provide estimates of N and P contents in manure.

When a manure analysis is not available, published average values (“book” values) for a given region are available. Book values can be obtained from the various provincial departments of agriculture.

The biggest challenges in manure sampling are:

- getting a representative sample from a manure pile or liquid manure storage structure;
- getting a laboratory analysis that can be applied to the calculation of this year's manure application rate.

4.1 When to Sample Manure

It is recommended that manure be sampled just prior to the actual application. If manure is applied in different seasons (such as in spring and in fall), a manure sample should be taken during each application period. Sampling at the time of application will account for changes in nutrient concentrations due to nutrient transformations, atmospheric losses, evaporation and dilution during storage.

Unfortunately, sampling at the time of application does not provide enough time for the nutrient analysis to be completed prior to application. This presents a problem for producers who are in the early stages of manure management planning. Book values do provide estimates that can be used in the absence of laboratory values. However, as shown in previous sections, manure nutrient concentrations are variable. As a result, book values may not represent actual manure nutrient concentrations for a given operation. Therefore, although it poses several challenges, sampling prior to application should be considered when the first manure sample is taken for an operation.

Due to the variability in manure, a single manure nutrient analysis can be unreliable. Manure samples should be taken over a number of sampling seasons to provide more reliable nutrient estimates for an operation. The greater the number of samples in a farm's database, the more accurate the information will be to calculate the annual manure application rates. Once established a farm's database can be used to calculate application rates if significant changes in the livestock production system have not been made. The current year's analysis can then be used to refine the manure nutrient estimates for the coming years.

4.2 How to Sample Liquid Manure

Because the solids tend to settle out of liquid manure over time (stratification), the manure in the storage should be well agitated before samples are taken. Samples of well-agitated manure will contain both liquid and solid fractions and, therefore, will provide a better average nutrient estimate for the entire manure storage. If the nutrient analysis represents all of the manure in the storage, then only one application rate is required per field to satisfy crop nutrient requirements.

Liquid manure samples should be representative of the manure being applied, or about to be applied, to the field. A one-litre (one quart) plastic sample bottle, no more than three-quarters full, is normally adequate for nutrient analysis. The plastic

bottles should be clearly labelled, placed in a plastic bag and sealed with a twist tie. All manure samples should be kept cool and transported immediately to the lab for processing. If immediate transport is not possible, the manure samples should be frozen until transport is possible.

4.2.1 Sampling from the Liquid Manure Storage

The most convenient time to take manure samples is when the manure is being pumped from the manure storage for land application. Several samples should be taken from the manure outlet over a period of several hours, mixed and sub-sampled to create one representative, composite sample. If micronutrient analysis is desired, the manure samples should be mixed using plastic equipment, as galvanized steel contains zinc and can interfere with the results.

Very large manure storages are difficult to fully agitate. In order to get an estimate of the nutrient variability within the storage, several samples can be taken while the storage is being emptied, and samples from the top, middle and bottom of the storage can be sent to the lab for separate analysis.

It is also possible to sample the manure storage prior to land application. However, if the manure has not been agitated, much more care must be taken to obtain a representative sample. Sampling equipment is available that can take samples at various depths within the storage. These samples should be thoroughly mixed, sub-sampled and transported to the lab for analysis.

There are safety issues related to sampling liquid manure. Liquid manure contains dangerous manure gases, such as hydrogen sulphide (H_2S). **Never enter an enclosed area that contains manure without wearing proper respiratory equipment.** Respiratory equipment is also recommended in the vicinity of all manure storages during agitation, as manure gases will be released in higher concentrations at this time.

4.2.2 Sampling Liquid Manure During Field Application



Catch Sample from Injector

It is also possible to take manure samples directly from the manure application equipment. If a manure tanker is being used, single samples can be taken from each of several tanker loads, mixed and sub-sampled in order to obtain a representative sample. A clean, plastic pail should be used to collect the sample from the unloading port or the opening near the bottom of the tank.

If applying with a drag hose system, several samples can be taken from the tap near the pump or from the injectors when they are lifted out of the ground.

Manure samples can also be taken during manure irrigation. Catch cans, such as aluminum roasting pans or clean plastic buckets, can be placed randomly in the field to catch the liquid manure. Although messy, this method of collection provides a real estimate of the N lost through volatilization during irrigation. Therefore, additional volatilization losses during irrigation (Table 6.2) do not need to be factored into the manure application calculation.

4.3 How to Sample Solid Manure

Obtaining a representative solid manure sample can be very difficult. A single composite sample from a manure pile may not provide an accurate estimate of the nutrient content. Therefore, a number of composite samples are recommended to get an estimate of the variability. For each solid manure sample that is taken, visible variations in moisture and bedding content should be taken into account.

For mixing and sub-sampling solid manure, the following method is recommended:

1. Combine all of the solid manure samples on a plastic sheet or cement pad and mix thoroughly. Large clumps should be chopped with a fork or spade.
2. Divide the well-mixed manure into four portions.
3. Discard two of the four portions.
4. Combine the remaining two portions and mix.
5. Repeat steps 2, 3 and 4 until the remaining sample is small enough to send for analysis.

A well-mixed sample (approximately one litre) should be placed in a freezer bag or tightly sealed plastic container. The sample should be placed in an outer bag that can be sealed to prevent leaks and kept cool or frozen until transported to the lab for analysis.



Sampling Solid Manure from a Truck

4.3.1 Sampling from Manure Piles or Housing Areas

Manure can be sampled directly from manure piles, packs or housing areas. In order to get an estimate of the variability, more than one composite sample should be sent for analysis. Manure should be taken from several depths and locations throughout the pile or pack using a fork or spade. If only a portion of the stockpile is to be spread, only that portion of the pile requires sampling. The objective is to take as many samples as necessary to obtain a reasonably representative sub-sample for analysis. It is often difficult to obtain samples from the middle of a large solid manure pile. It may be necessary to access the centre of the pile using a front-end loader. A sample should then be obtained from the bucket of the loader. All of the manure samples can be mixed and sub-sampled for analysis.



Solid Manure Pile

4.3.2 Sampling Solid Manure During Field Application

Solid manure can be sampled directly from the manure spreader just prior to field application. Single samples can be taken from each of several spreader loads using a fork or spade. The number of spreader loads that must be sampled in order to obtain a representative sample will depend on the total amount of manure that is to be spread. The spreader loads which are sampled should represent the beginning, middle and end of the application process. As the samples are being collected, they should be placed in a bucket and, if possible, temporarily stored where it is relatively cool (such as in the shade). The samples must be well mixed in order to obtain a representative sub-sample for analysis.

4.4 Handling and Shipping Samples

It is recommended that the manure-testing laboratory be consulted regarding specific requirements for sample size and shipping, turn-around times, analytical options, costs and any additional instructions it may have. As well, some labs provide containers, labels and submission forms for manure samples.

When handling and shipping samples, leakage, nutrient loss to the air (volatilization), moisture loss and nutrient transformations must be prevented as much as possible. To accomplish this:

- Liquid manure containers should be no more than three-quarters full in order to provide air space in the container for manure gases.
- All samples should be kept cool, either by refrigeration or by placing on ice, until they are transported to the lab. Cool conditions will reduce nutrient transformation and minimize odours.
- If the samples cannot be transported within a day, they should be frozen until shipping is possible. If liquid manure is to be frozen, plastic containers should be used and should be no more than three-quarters full to leave room for the liquid to expand while freezing.
- Samples should spend no more than two days in transit. Shipping should be arranged to ensure that samples are not held over weekends or holidays or in warm locations.
- Manure samples should be tightly sealed to prevent leakage. Secondary containment, such as double bagging, should be used as an added precaution.



Sampling Liquid Manure

4.5 Laboratory Analysis for Manure

Laboratories that offer manure analysis often provide a variety of tests and analysis packages. Costs can vary, depending on the laboratory and the extent of the analysis.

The following manure analyses are recommended:

- moisture content or dry matter content
- total N
- ammonium N
- total P
- total K
- EC (required in Alberta)

Optional tests could include: electrical conductivity (total salts), pH, chloride (Cl), sulphur (S), sodium (Na), calcium (Ca), magnesium (Mg) and micronutrients, such as copper (Cu), manganese (Mn), zinc (Zn) and iron (Fe).

4.6 Estimating Manure Nutrients in the Field

Sampling manure for laboratory analysis at the time of application provides the most accurate measurement of the nutrient content. However, due to the delay between sample collection and return of the lab analysis results, this information cannot be used in the calculation of the current application rate.

Estimates of manure nutrient concentrations can be obtained quickly and inexpensively in the field using rapid-test kits. Although not as accurate as laboratory results, these estimates can be used to calculate approximate application rates prior to application. Laboratory analysis of a representative sample can then be used to confirm the result obtained from the rapid-test kit. Although adjustments can sometimes be made to the testing procedure, rapid-test field kits are designed mainly for manure with relatively low solids content (i.e. liquid or semi-solid manure).

Caution: Rapid field-testing of manure can only provide rough estimates of nutrient concentrations. It should not replace regular laboratory analysis.

Most rapid tests provide estimates, through either direct or indirect measurement, of ammonium N in manure. Tests kits that provide ammonium N estimates include:

- Agros N meter
- ammonia electrode
- conductivity meter
- conductivity pen
- Quantofix-N-volumeter
- Reflectometer

These rapid analytical methods are useful since manure is typically applied to meet the crop's requirements for N, and ammonium N is the most readily available form of N in manure.

Some rapid testing procedures, such as those of the conductivity meter and conductivity pen, can also provide indirect estimates of potassium ion (K^+) concentrations in solution. However, because these methods involve only indirect determinations of K^+ levels, interference by other ions in solution can reduce the accuracy of results.

Estimates of total N and total P in manure can be determined using a hydrometer, an instrument that measures the specific gravity, or density, of a liquid. This method is based on two premises: that the density of manure varies with its dry matter, or solids, content; and that total N and total P concentrations vary with dry matter content.

The accuracy of rapid test kits in the field can be reduced due to a number of factors, including manure variability and inconsistent operation of equipment. Consequently, on-farm calibration over multiple manure applications is recommended. Calibration is the adjustment of field test kit readings to improve their accuracy. The adjustment is typically based on laboratory results. Keeping complete records of field and laboratory results over time is critical to effective calibration. Calibration of the rapid-test equipment for certain nutrients can also be done on a regular basis (even prior to each use) using standard solutions, each of known concentration.