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Manure Sampling and Testing

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Biographies for most speakers are in alphabetical order after the last paper.

Background

In September 1996, a joint NCR-13 and SERA-6 soil testing work group meeting was held in Raleigh, North Carolina. Earlier in that year, a manure sample exchange was conducted with NCR-13, SERA-6 and NEC-67 member laboratories. Results from that sample exchange were presented at the Raleigh meetings and sparked interest in joining efforts to develop a manure-testing manual, which could be used in all regions. A multi-regional committee was formed to share expertise in the development of a common manual. Members of the multi-regional committee working on the publication include John Peters and Sherry Combs (NCR-13, WI), Ann Wolf and Doug Beegle (NCR-13 and NEC-67, PA), Maurice Watson (NCR-13, OH), Jan Jarman (MN Dept. of Agriculture), Nancy Wolf (SERA-6, AR), John Kovar (SERA-6, LA), and Bruce Hoskins (NEC-67, ME). The author wishes to acknowledge the significant editorial contributions of members of this committee in the sampling section of this paper and to Sherry Combs for her assistance in the laboratory methods portion of this paper.

Introduction

There are essential pieces of information required to determine the proper application rate and nutrient credits for livestock waste to meet crop needs. These include the acreage of the field, capacity of the spreader and nutrient concentration of the manure. Nutrient concentration can be assigned by using estimated "book" or average available N, P_2O_5 and K_2O concentrations. However, testing manure may better indicate how factors such as animal management affect manure nutrient content. Using good sampling technique is critical for maintaining confidence in manure nutrient analysis results. Appropriate sample handling and laboratory methods are also important to ensure accurate results.

Sampling Livestock Waste

Data in the livestock waste facilities handbook (MWPS, 1985) provides "typical" or average nutrient contents for manures of several animal types. These values probably give an acceptable estimate for "typical" producers, especially if current sampling methods used do not represent the pit, pack or gutter adequately. However, an analysis of a well-sampled system may give a better estimate of manure nutrient concentrations for individual farms than book values, especially if herd and manure management are not "typical". The MWPS total nutrient estimates are compared in Table 1 to actual manure analysis of 51 farms in Minnesota (Wagar et al., 1994) and from 1128 manure samples submitted to the University of Wisconsin Soil Testing Labs between 1986-1991 (Combs, 1991). On average, the actual farm values compare well to the MWPS estimates. Note however that the actual analysis values range widely from the MWPS estimates, indicating poor sampling, management or other on-farm differences. Lindley et al., (1988) also found actual manure analysis values to be highly variable and ranged from 50 to 100% of published values.

Table 1. Comparison of analyzed manure total nutrient concentrations to "typical" nutrient concentrations.

Animal type	System	Nutrient	Minnesota * Avg.	Minnesota * Range	Wisconsin** Avg.	Wisconsin** s.d.	Wisconsin** Range	MWPS *** Avg.
			lbs/1000 gal					
Dairy	Liquid	N	29	10-47	28	11	1-71	24
		P_2O_5	15	6-28	13	9	1-118	18
		K_2O	24	11-38	29	16	1-171	29
			lbs/t					
Dairy	Solid	N	13	7-25	10	3	3-33	9
		P_2O_5	6	3-13	6	3	0.2-35	4
		K_2O	8	2-18	11	5	0.2-24	10
			lbs/1000 gal					
Swine	Liquid	N	48	7-107	41	40	1-281	36
		P_2O_5	28	3-64	17	19	1-141	27
		K_2O	21	7-51	21	15	2-83	22

* Nutrient levels in manure samples taken from 51 farms.

** Nutrient levels in 388 solid/semi-solid dairy, 380 liquid dairy and 260 liquid swine manure samples submitted to the University of Wisconsin Soil Testing Labs 1986-1991.

*** Livestock Waste Facilities Handbook (MWPS, 1985).

Technique

In virtually any type of agricultural analytical work the results are greatly influenced by sampling. For solid manure, it is generally recommended to sample from loaded spreaders rather than from the actual manure pack. A Wisconsin study (Peters and Combs, 1998) showed that even when well trained professionals sampled dairy manure, variability was much higher when samples were collected directly from the barnyard and pack compared to

those collected from the loaded spreader (Table 2). The data also indicated that taking several samples would help minimize potential variability.

In this same study, several samples of liquid manure were taken from a thoroughly agitated lagoon while being pumped into a spreader tank. The results of multiple samples taken by different individuals from a well-agitated liquid dairy manure lagoon indicate that variability is much lower than in the solid manure/barnyard system.

Table 2. Variability in dairy manure nutrient content when sampled by multiple individuals.* Marshfield 1997.

Material	Sampling Method	No. of Samples	DM	N	P	K
			%			
Dairy - Solid	Barnyard- hand					
		6				
		Mean	35.02	1.87	0.42	2.48
		SD	2.81	0.22	0.04	0.27
	Barnyard- shovel					
		7				
		Mean	31.37	2.10	0.50	3.45
		SD	4.50	0.40	0.09	1.16
	Spreader- hand					
		6				
		Mean	34.35	1.98	0.42	2.60
		SD	1.41	0.17	0.03	0.39
	Spreader- shovel					
		6				
		Mean	34.60	1.98	0.41	2.30
		SD	4.82	0.31	0.04	0.31
Dairy - liquid	Pump-direct	8				
		Mean	5.11	4.66	1.27	5.23
		SD	0.08	0.32	0.09	0.66
	Pail- subsample					
		4				
		Mean	5.20	4.80	1.30	5.15
		SD	0.06	0.10	0.03	0.23

* Wisconsin Farm Training Instructions used in the study.

Further, variability can exist among different samplings even when they are taken by the same individual under ideal conditions. This occurred when samples of liquid and semi-solid dairy manure were collected. In this Wisconsin study, five-gallon samples were mixed as thoroughly as possible before being split into twenty-four subsamples. The results indicate that the variability between liquid samples was quite low, but higher with semi-solid dairy samples. This was particularly apparent with total N and dry matter measurements (Peters and Combs, 1998).

Time

An evaluation of long-term sampling of solid/semi-solid manure showed little variability occurred in nutrient concentration over a three-year period at the University of Wisconsin Arlington Agricultural Research Station (Combs, 1991). Sampling from a stanchion barn pack periodically for three years showed that all samples had similar total nutrient values. The least variation occurred for N while the greatest variation was associated with K. These results seem to indicate that with good representative sampling and no significant change in herd management, consistent results, even for solid manure, are possible.

On the other hand, results from sampling solid manure in a poultry-laying barn at the University of Wisconsin Arlington Agricultural Research Station indicated inconsistent results over time (Peters and Combs, 1998). These poultry manure samples taken from the same barn approximately five months apart show a significant difference in all parameters measured. This could be partially a result of seasonal changes in the feed ration, feed contamination or differences in individual sampling technique. Commonly, 5-6 batches of birds are grown out before the litter is removed. Poultry houses are normally sampled when the last batch of birds is removed from the house. Sampling earlier is not recommended, since the nutrient content in poultry litter will change over time.

Due to these variations over time, manure nutrient concentration values used to determine field nutrient credits should ideally be based on long-term farm averages, assuming herd and manure management practices have not changed significantly.

Storage Management

The segregation of manure that occurs in liquid storage requires that special care be taken to ensure that a homogeneous mix is sampled. In a Minnesota study, manure agitated for 2-4 hours before application had highly consistent results for total N, P, K concentrations and percent solids when individual tanks (first to last) were analyzed (Wagar et al., 1994). Samples taken at various stages during the storage system emptying process at Wisconsin also showed very little variability providing the material was thoroughly agitated (Peters and Combs, 1998).

Sampling Recommendations

The number of manure samples tested by public and private labs has increased from approximately 6,220 in 1988 to almost 16,000 in 1996 (Soil, Plant and Animal Waste Analysis Status Report, 1992-96). However, the majority of animal producers still do not sample manure. Reasons for not doing so include sample heterogeneity and the inherent difficulty of taking a representative sample.

Several states have developed guidelines for sampling manure to minimize the sample heterogeneity problem. This information was used to help develop the sampling guidelines presented here. It is generally not recommended to attempt to sample bedded packs or unagitated liquid manure storage facilities. In fact, using nutrient analysis results from

poorly sampled systems will not improve the accuracy in estimating N or P loading to a field and may in fact be detrimental.

Taking an adequate number of subsamples is critical for getting a good estimate of nutrient value. In order to characterize N content of a beef manure stockpile within 10%, it took a Colorado State researcher 17 subsamples (Successful Farming, August 1998). However, getting that level of accuracy for P required 20 subsamples and for K it required 30. As a minimum, take a composite sample three independent times and subsample at least five times per composite.

Recommended Procedures for Sampling Livestock Waste for Analysis

Recommended procedures for sampling liquid and solids waste are given below. Producers may choose from these methods as appropriate.

Solid manure – Dairy, Beef, Swine, Poultry
Obtain a composite sample by following one of the procedures listed below. Thoroughly mixing the composite sample is important. One method is to pile the manure and then shovel from the outside to the inside of the pile until well mixed. Fill a one-gallon plastic heavy-duty ziplock bag approximately one-half full with the composite sample, squeeze out excess air, close and seal. Store sample in freezer if not delivered to the lab immediately.

Sampling while loading – *Recommended method for sampling from a stack or bedded pack.* Take at least five samples while loading several spreader loads and combine to form one composite sample. Thoroughly mix the composite sample and take an approximately one pound subsample using a one-gallon ziplock plastic bag. *Sampling directly from a stack or bedded pack is not recommended.*

Sampling during spreading – Spread tarp in field and catch the manure from one pass. Sample from several locations and create a composite sample. Thoroughly mix the composite sample and take a one-pound subsample using a one-gallon ziplock plastic bag.

Sampling daily haul – Place a five-gallon pail under the barn cleaner 4-5 times while loading a spreader. Thoroughly mix the composite sample and take a one-pound subsample using a one-gallon ziplock plastic bag. Repeat sampling 2-3 times over a period of time and test separately to determine variability.

Sampling Poultry In-house - Collect ten samples from throughout the house to the depth the litter will be removed. Samples near feeders and waterers may not be indicative of the entire house and subsamples taken near here should be proportionate to their space occupied in the whole house. Mix the samples well in a five-gallon pail and take a one-pound subsample, using a one-gallon ziplock bag.

Sampling Stockpiled litter – Take ten subsamples from different locations around the pile at least 18 inches below the surface. Mix in a five-gallon pail and take a one-pound subsample using a one-gallon plastic ziplock bag.

Liquid Manure - Dairy, Beef, Swine
Obtain a composite following one of the procedures listed below and mix thoroughly. Using a plunger, an up-and-down action works well for mixing liquid manure in a five-gallon pail. Fill a one-quart plastic bottle not more than three-quarters full with the composite sample. Store sample in freezer if not delivered to the lab immediately.

Sampling from storage – Agitate storage facility thoroughly before sampling. Collect at least five samples from storage facility or during loading using a five-gallon pail and mix. Place subsample of the composite sample in a one-quart plastic container. *Sampling a liquid manure storage facility without proper agitation (2-4 hrs. minimum) is not recommended.*

Sampling during application – Place buckets around field to catch manure from spreader or irrigation equipment. Combine and mix samples into one composite sample. Place the subsample of the composite sample in a one-quart plastic container.

Sample identification and delivery

Identify the sample container with information regarding the farm, animal species and date. This information should also be included on the sample information sheet along with application method, which is important in determining first year availability of nitrogen.

Keep all manure samples frozen until shipped or delivered to a laboratory. Ship early in the week (Mon.-Wed.) and avoid holidays and weekends.

Analyzing Livestock Waste

The ability of a laboratory, or individual using a quick test, to accurately analyze the nutrient value depends not only on sampling technique and handling but how a sample is prepared for analysis and which method is selected. In many cases, different methods can be used depending on instrumentation available and staff expertise. Even when only one method is available, how an individual executes its steps can be important to the overall results.

Laboratory Methods

Methods used to determine manure nutrient content were surveyed among public and private soil testing labs in order to develop suggested standardized methods for manure and other organic waste analysis.

Twenty-five of the 32 labs surveyed reported using the Kjeldahl technique for total N analysis. However, to arrive at 'total N' the nitrate-N must be determined and added to the Kjeldahl N value or the Kjeldahl test must be modified for total N analysis to include nitrate-N by use of salicylic acid. Nitrogen was also determined by the Dumas type of dry combustion analyzers. The Dumas type of analyzer measures total N in one single value that includes all forms of N (organic-N, ammonium-N, nitrate-N and nitrite-N). Most labs used

distillation if NH4-N was reported, but the Nessler color technique or an ion selective electrode were also identified.

Total P and K were determined in 19 labs by wet digestion techniques using a variety of acid combinations. Several used a microwave system instead of the traditional beaker /tube and hot plate. Eight labs dry ashed samples. The Inductively Coupled Plasma or Atomic Absorption Spectrometers were the most common detection systems but some labs reported using a colorimeter or Directly Coupled Plasma Spectrometer.

The choice of drying times and temperatures used to determine dry matter content varied substantially among all labs. Samples were dried anywhere from 4 hrs to 4 days at temperatures ranging from 50°C to 140°C. Overnight (16-24 hrs) was the most common drying time (15 labs). Most public labs used temperatures < 100°C while many private labs dried at temperatures ≥ 100°C.

Method Performance

Because of the variety of methods found to be used by soil testing laboratories to determine nutrient content, the NCR-13 Waste Analysis Subcommittee conducted a manure sample exchange to evaluate performance of various laboratory analytical methods. This exchange included 18 public laboratories in the NCR-13, NEC-67 and SERA-6 working groups. Solid and semi-solid manures were spread on a concrete floor and thoroughly mixed with manure forks and then subsampled. Liquid manure samples were taken from the lagoon at the Marshfield Agricultural Research Station while it was being agitated and field spread. Two sets of liquid samples were taken two days apart when the lagoon was approximately one-quarter and three-quarters empty.

Variability

To measure the variability present in the samples, four liquid and eight solid subsamples were analyzed by the University of Wisconsin Soil and Forage Analysis Laboratory. The variability in the liquid samples was quite low, but was 2 to 20 times higher for total N and DM in poultry and dairy semi-solid samples (Table 3). Because of this variability between samples, despite extensive mixing, solid manures were dried and ground prior to the NCR-13, NEC-67 and SERA-6 distribution. Bulk samples of poultry and sheep manure were dried in large forced air forage dryers at 55°C and ground through a 4-mm Wiley mill. The ground material was then thoroughly mixed in a large pail and subsampled into zip lock bags.

Table 3. Effect of in-lab variability on total nutrient content of liquid and solid/semi-solid manure.

Material		No. of Analyses	Nutrient* (dry weight basis) DM	N	P	K
			%			
Liquid Dairy Manure #3		4				
	mean		7.13	4.25	1.04	3.63
	SD		0.08	0.09	0.03	0.04
Liquid Dairy Manure #4		4				
	mean		6.05	4.65	1.28	4.07
	SD		0.09	0.05	0.05	0.04
Poultry (fresh)		8				
	mean		28.14	6.31	1.76	3.08
	SD		0.15	1.12	0.04	0.05
Dairy Semi-Solid (fresh)		8				
	mean		14.14	3.75	0.83	3.27
	SD		0.14	0.26	0.02	0.03

* University of Wisconsin Soil and Forage Analysis Lab - Marshfield

To assess the in-lab variability for all labs in the exchange program, a duplicate sample of the sheep manure was included in the five samples distributed. The mean and standard deviations for the individual samples were in close agreement with the overall values indicating that there was, generally, relatively low in-lab variability.

Five manure samples were sent to soil testing labs in the NCR-13, NEC-67 and SERA-6 regions and included:

Exchange Sample	Material
1	Poultry manure, no bedding, dried & ground
2	Sheep manure, bedded pack (same as #5)
3	Liquid dairy manure, lagoon ¾ empty
4	Liquid dairy manure, lagoon ¼ empty
5	Same as #2

The methods used by soil testing laboratories in the NCR-13, NEC-67 and SERA-6 regions generally fall into two categories for N, P and K. For total nitrogen, either Kjeldahl (micro and macro) or combustion were used. Total P and K were determined by either a dry ash (5 labs) or wet digestion (6 labs) using varying mixes of acids and detection systems (ICP, AA, colorimeter). Sample dry matter was determined by weight loss by all labs with drying temperatures ranging from 50 to 110°C and times from overnight to 72 hours.

Analytical results were received from 14 of the 18 labs who received samples. Slightly lower standard deviations from the mean were shown by using the Kjeldahl method for total N analysis compared to combustion, however, differences between methods were small. The liquid dairy manure samples showed higher variabilities by both methods than when samples were analyzed after drying and grinding. The combustion method means were higher than the overall mean, except sample #1, whereas Kjeldahl means showed no consistent pattern. Both methods seemed equally capable of producing reasonable results, especially for dried/ground manure.

Phosphorus analysis was extremely consistent between the dry ash and wet digestion methods used. This low variability occurred between methods in spite of differing acid mixes for ash dissolution or digestion and detection systems.

Potassium analysis was more variable than phosphorus. Samples prepared by dry ashing tended to result in slightly lower total K contents than by wet digestion. Wet digestion methods had highest standard deviations for the liquid dairy manure (samples 3 and 4) perhaps because of subsampling difficulties.

Dry Matter

The variability associated with dry matter determinations depended on whether a single lab or multiple lab results were considered and if manure was solid or liquid. Standard deviation for liquid dairy manure was <0.09% with only somewhat higher levels found for solid fresh dairy and poultry manure when analyzed by a single lab. Colorado researchers found that they could achieve within 10% accuracy for dry matter when only 3 subsamples were tested by the same lab (Successful Farming, August, 1998). However, when results were pooled across the varied times and temperatures used by labs to determine dry matter, the associated standard deviations increased especially for solid manure. Consistent results were most difficult to achieve for pre-dried poultry and sheep manure. Liquid manure variability was less but not as consistent as the single lab results.

Rather small differences in dry matter determinations can have substantial effects on reported fresh weight nutrient contents. The values in table 4 show how a difference in DM from 12 to 18% can change 'as is' N, P_2O_5 and K_2O values. Since producers use the 'as is' values for determining fertilizer equivalence, this could translate into an inappropriate application rate. Therefore, a more consistent method for determining dry matter is needed.

Table 4: Dry matter effect on calculated manure nutrient content.

	N	P_2O_5	K_2O
DM %	lbs/wet ton		
12	7.2	5.5	11.5
15	9	6.9	14.5
18	10.8	8.3	17.3

*based on dry weight analysis of 3.00% N, 1.00% P and 4.00% K

An in-depth study was initiated in 1998, among public labs at Arkansas, Maine and Pennsylvania to explore the effects of drying time and temperature on manure dry matter content (Wolf, Hoskins, and Wolf, Multi-regional Committee, 1998). Three temperatures (50, 70 and 110°C) and 4 drying times (6, 16, 24 and 48 hrs) were chosen to be used on 3 or 4 manure samples routinely submitted for analysis. A representative subsample was suggested to be approximately 10 g fresh weight for solid manures (>85% DM) or 20 g fresh weight for liquid manure (<85% DM). Subsample size was found to have a significant effect on drying time to constant weight for some samples dried at lower temperatures. Dairy manure samples, larger that 10g, required 24 hrs at 50°C to reach constant weight but only 16 hrs for more moderately sized samples. This effect was not shown for poultry manure or for those samples dried at higher temperatures. Liquid and solid dairy manure samples dried to constant weight at the lower (50-70°C) temperatures had ≤ 1-2% absolute relative moisture remaining after 48 hrs compared to drying at 110°C. However, poultry manure did not follow this trend and had relatively high moisture content remaining at 50 and 70°C compared to 110°C.

Table 5: Suggested minimum drying times at various temperatures.

	50°C	70°C	110°C
	hrs		
solids (<85% H_2O)	24	16	6
liquids (>85% H_2O)	48	48	16

On-farm Testing

In addition to using data from a commercial laboratory or book values, a third option for determining manure nutrient credits is the use of rapid on-farm testing. There are several quick tests that are commercially available for on-farm nutrient analysis. These measure either one or more of the following: ammonium N, total N, total P, and total K. Analysis takes less than 10 minutes and the equipment is, typically, relatively simple to use. Some of the instrument methods, which were reviewed by Van Kessel, et. al., 1999, include the hydrometer, conductivity meter and pen, ammonia electrode, reflectometer, and hypochlorite reaction meters. The hydrometer is used to indirectly measure total N and total P in slurry samples. The conductivity meter and conductivity pen are commonly used to measure ammonium N and K in slurry samples. The other instruments are mainly used for measuring ammonium N only. In all cases, a properly taken, representative sample is required. The skill of the individual operator is critical to obtaining consistently accurate results. The on-farm or "quick" test should be calibrated to traditional laboratory methods. In most situations, more information will be necessary than can be obtained from the somewhat limited scope of this type of instrumentation.

These "quick" on-farm methods are less accurate and should not be considered as a replacement for traditional laboratory testing. If used in conjunction with standard laboratory testing, these quick tests may have a value in monitoring nutrients in well-agitated liquid manure systems.

Summary

Obtaining a representative sample is critical for any method to be of value. Results of on-farm or laboratory testing are limited by how well the sample represents the farm's manure. Recommended sampling protocol must be followed to ensure a representative sample is obtained.

Results of the NCR-13 exchange tend to emphasize that laboratory analysis method and execution of the method influence results. In view of the consistency of the public labs' results with varying methods, standardizing methods may not result in better analysis. However method standardization may be a good first step in gaining producer confidence in using laboratory results. It is difficult to identify a 'best' method and suggests that probably of greater importance is good execution of individual methods. The exception is dry matter. The importance an accurate estimate of dry matter has on determining fertilizer equivalence makes development and use of a standardized method important.

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