

# Recommended Methods for Manure Analysis:

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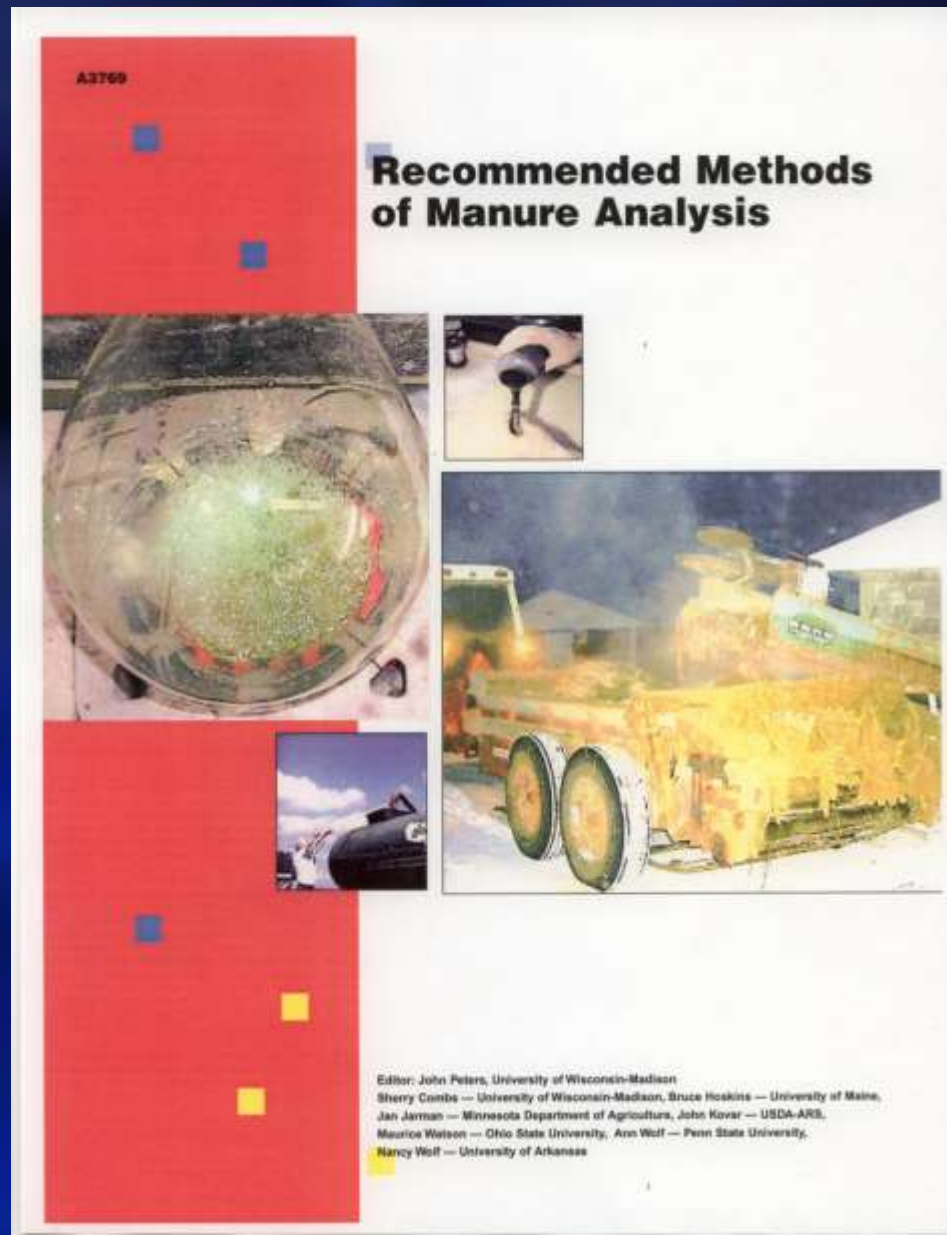
University of Wisconsin – Madison

# Background

- Multi-regional committee was established in September 1996 to work on the development of a manual for manure sampling, analysis and reporting.
- Committee members from NCR-13, SERA-6, NEC-67, and MN Dept. of Agriculture
- Publication is available on the web

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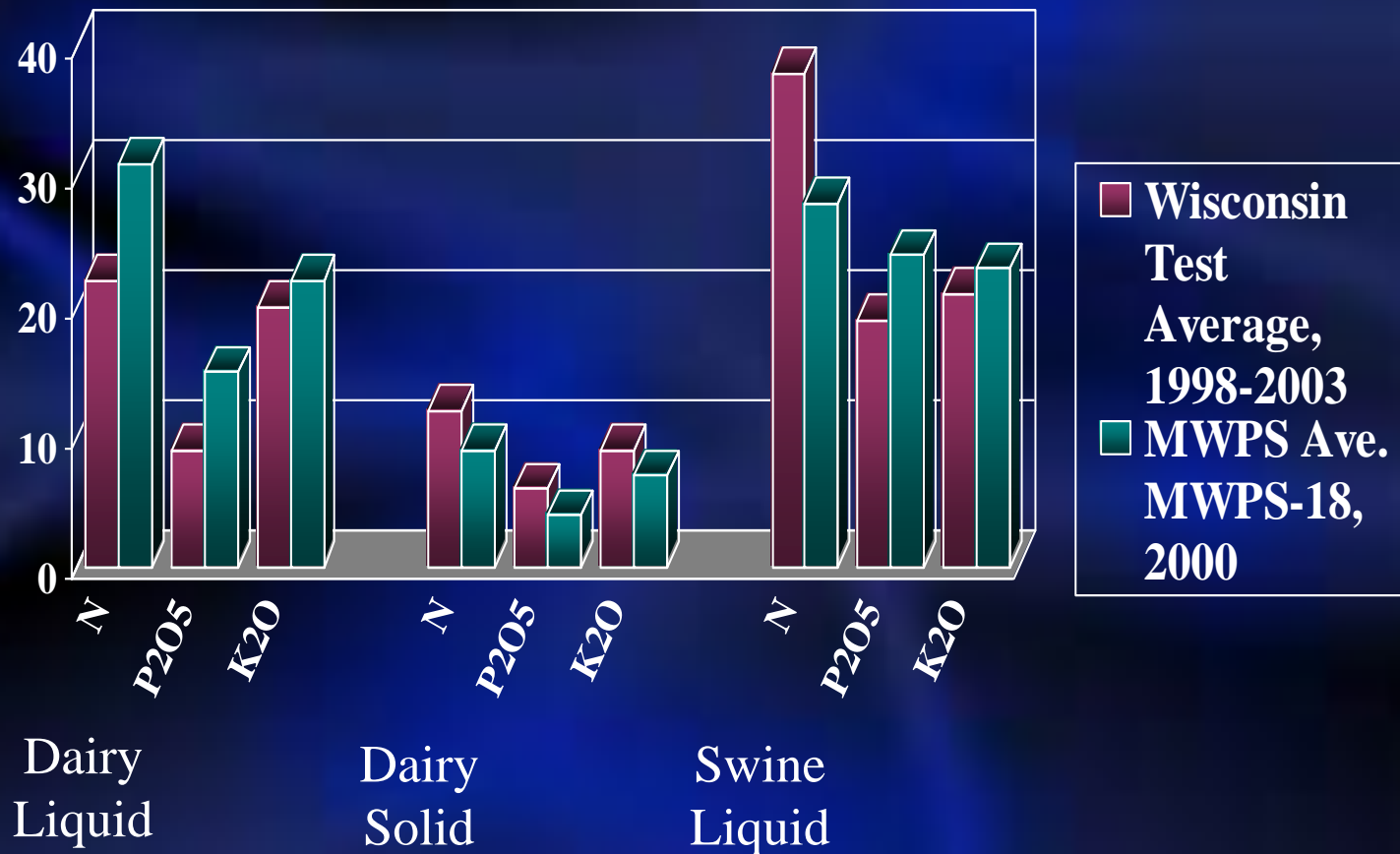
# Introduction

- Nutrient concentrations can be estimated using “book” values for available N, P<sub>2</sub>O<sub>5</sub>, and K<sub>2</sub>O
- Manure testing takes management practices into account and delivers more accurate values
- Sampling technique greatly influences test results
- Sample handling and testing methods also affect analytical results

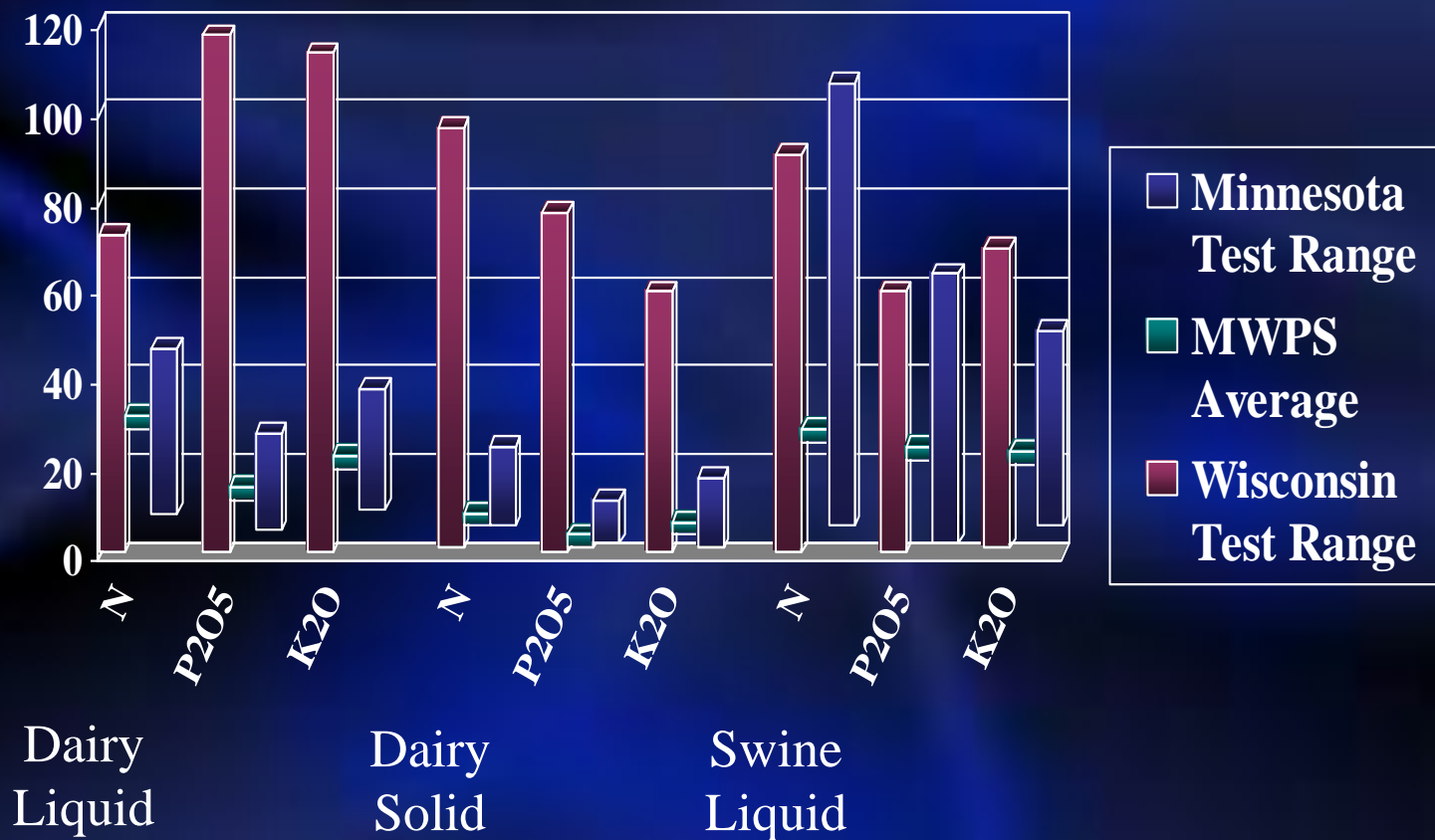
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# Comparison of analyzed and “typical” manure nutrient content



# Range of analyzed manure nutrient content



# Effect of In-Lab Variability on Total Nutrient Content of Manure

| Material               | No. of Analysis |      | DM   | Nutrient*     |      |      |
|------------------------|-----------------|------|------|---------------|------|------|
|                        |                 |      |      | 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 |

\* Dry Weight Basis, University of Wisconsin Soil and Forage Analysis Lab - Marshfield

# Effect of In-Lab Variability on Total Nutrient Content of Manure

| Material                       | No. of<br>Analysis |      | DM    | Nutrient*     |      |      |
|--------------------------------|--------------------|------|-------|---------------|------|------|
|                                |                    |      |       | N             | P    | K    |
|                                |                    |      |       | ----- % ----- |      |      |
| Poultry<br>(fresh)             | 8                  | Mean | 28.14 | 6.31          | 1.76 | 3.08 |
|                                |                    | SD   | 0.15  | 1.12          | 0.04 | 0.05 |
| Dairy<br>semi-solid<br>(fresh) | 8                  | Mean | 14.14 | 3.75          | 0.83 | 3.27 |
|                                |                    | SD   | 0.14  | 0.26          | 0.02 | 0.03 |

\* Dry Weight Basis, University of Wisconsin Soil and Forage Analysis Lab - Marshfield





# Nutrient Variability of Solid Dairy Manure.\* Marshfield, 1997

| Sampling Method   | No. of Analysis |      | DM    | Nutrient**    |      |      |
|-------------------|-----------------|------|-------|---------------|------|------|
|                   |                 |      |       | N             | P    | K    |
|                   |                 |      |       | ----- % ----- |      |      |
| 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 |

\* Wisconsin Farm Training Instructions used in this study.

\*\* Dry matter basis



# Nutrient Variability of Solid Dairy Manure.\* Marshfield, 1997

| Sampling Method   | No. of Analysis |      | Nutrient**    |      |      |      |
|-------------------|-----------------|------|---------------|------|------|------|
|                   |                 |      | DM            | N    | P    | K    |
|                   |                 |      | ----- % ----- |      |      |      |
| 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 |

\* Wisconsin Farm Training Instructions used in this study.

\*\* Dry matter basis



# Effect of Agitation on Analysis of Liquid Manure

## Minnesota

| Animal System           | Loads From Storage | DM  | Nutrient                 |                               |                  |
|-------------------------|--------------------|-----|--------------------------|-------------------------------|------------------|
|                         |                    |     | N                        | P <sub>2</sub> O <sub>5</sub> | K <sub>2</sub> O |
|                         |                    | %   | ----- lbs/1000 gal ----- |                               |                  |
| Dairy -<br>Metal Tank   | First              | 8.2 | 34                       | 16                            | 28               |
|                         | Mid                | 8.6 | 33                       | 18                            | 28               |
|                         | Last               | 8.0 | 30                       | 14                            | 27               |
| Swine -<br>Concrete Pit | First              | 6.9 | 45                       | 50                            | 15               |
|                         | Mid                | 8.5 | 46                       | 60                            | 16               |
|                         | Last               | 7.4 | 46                       | 57                            | 18               |

# Effect of Agitation on Analysis of Liquid Manure

| Animal System | Loads From Storage | DM      | Nutrient                 |                               |                  |
|---------------|--------------------|---------|--------------------------|-------------------------------|------------------|
|               |                    |         | N                        | P <sub>2</sub> O <sub>5</sub> | K <sub>2</sub> O |
| Minnesota     |                    | -- % -- | ----- lbs/1000 gal ----- |                               |                  |
| Dairy -       | First              | 4.4     | 24                       | 10                            | 23               |
| Earthen Pit   | Mid                | 6.0     | 23                       | 10                            | 22               |
|               | Last               | 8.7     | 27                       | 12                            | 23               |
| Wisconsin*    |                    |         |                          |                               |                  |
| Dairy -       | Early**            | 6.1     | 24                       | 15                            | 23               |
| Earthen Pit   | Late**             | 7.1     | 25                       | 14                            | 25               |

\* Dairy milking herd, Marshfield Agricultural Research Station

\*\* Average of four-subsample analysis





# Nutrient Variability of Liquid Dairy Manure.\* Marshfield, 1997

| Sampling Method  | No. of Analysis |      | DM   | Nutrient**    |      |      |
|------------------|-----------------|------|------|---------------|------|------|
|                  |                 |      |      | N             | P    | K    |
|                  |                 |      |      | ----- % ----- |      |      |
| 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 | 5.2  | 4.8           | 1.30 | 5.15 |
|                  |                 | SD   | 0.06 | 0.10          | 0.03 | 0.23 |

\* Wisconsin Farm Training Instructions used in this study.

\*\* Dry matter basis



How do you sample this?



Sampling while loading is a better choice

# Recommended Sampling Procedures: Solid Manure

- Sampling while loading
  - Take samples from several spreader loads
  - Combine samples to form one composite sample



# Recommended Sampling Procedures: Solid Manure

- Sampling during spreading
  - Catch manure from one pass on a tarp in field
  - Sample from several locations to create a composite sample



# Recommended Sampling Procedures: Solid Manure

- Sampling daily haul
  - Place five-gallon bucket under the barn cleaner 4-5 times while loading spreader
  - Repeat sampling 2-3 times and test separately



# Recommended Sampling Procedures: Solid Manure

- Sampling stockpiled manure
  - Take ten subsamples from different locations around the pile at least 18 inches below surface
  - Mix thoroughly in a five-gallon pail



# Recommended Sampling Procedures: Solid Manure

- Sampling poultry in-house
  - Collect ten samples from throughout the house to the depth litter will be removed
  - Sample near feeders and waterers proportionately to their space occupied in the whole house
  - Mix samples well in a five-gallon pail



# Sampling Procedures: Liquid Manure

- Sampling from storage
  - Agitate storage facility thoroughly (2-4 hrs minimum)
  - Collect at least five samples from storage facility or during loading using a five gallon pail



# Sampling Procedures: Liquid Manure

- Sampling during application
  - Place buckets around field to catch manure from spreader or irrigation equipment
  - Combine and mix samples



# Effect of Sampling Time on Content of Laying Hen Manure

| Month* | DM   | Nutrient**          |                               |                  |
|--------|------|---------------------|-------------------------------|------------------|
|        |      | N                   | P <sub>2</sub> O <sub>5</sub> | K <sub>2</sub> O |
|        | %    | ----- lbs/ton ----- |                               |                  |
| March  | 28.1 | 36                  | 23                            | 20               |
| August | 20.0 | 25                  | 7                             | 14               |

\* Average of four-subsample analysis

\*\* Laying hen barn, University of Wisconsin Arlington Agricultural Research Station

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# Quality Assurance

- Ensures consistent quality of analytical results through the application and documentation of appropriate quality control and quality assessment procedures
- Serves to promote client confidence in analytical results and documenting analytical uncertainty.

# Quality Control

- Comprised of laboratory practices undertaken specifically to achieve accurate and reliable analytical results.
- Quality assessment is comprised of the procedures undertaken to monitor and document the effectiveness of quality control practices.

# Components of a Quality Control Program

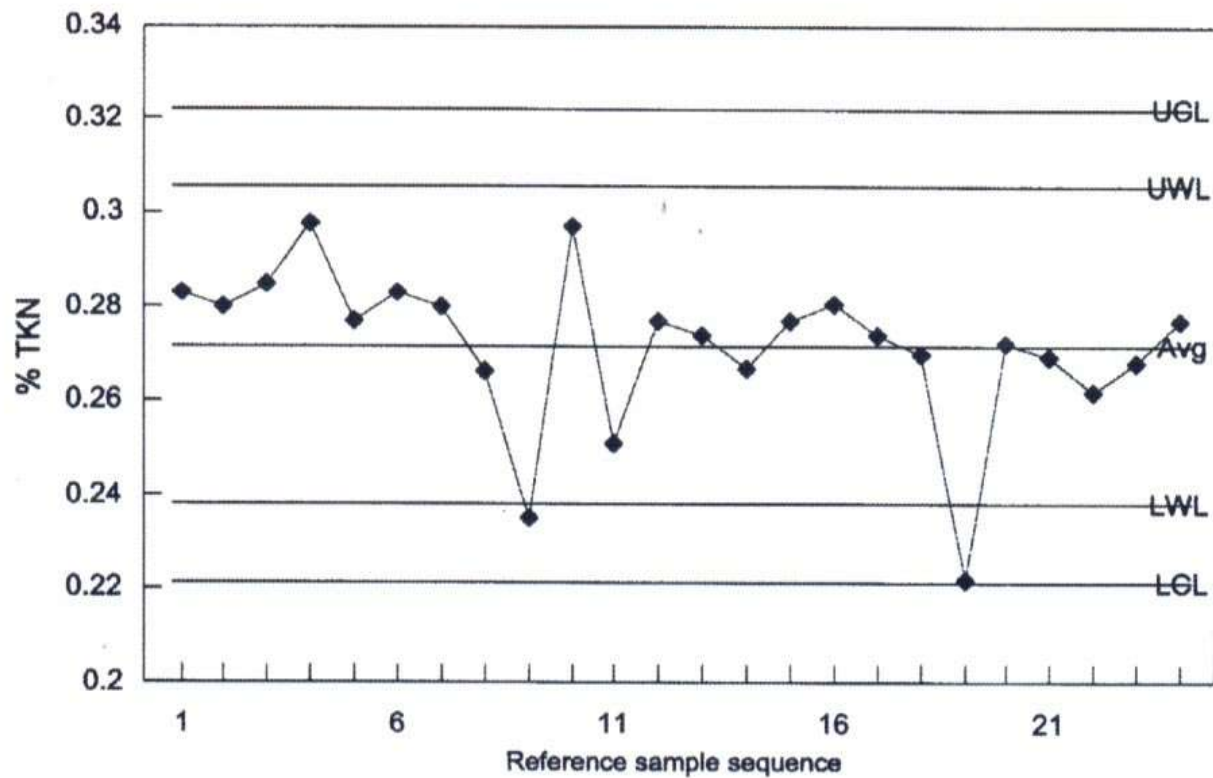
- Documentation of SOP (standard operating procedure)
- Training
- Implementation of good laboratory practices and procedures

# Components of a QA/QC Program

- Document precision
- Document accuracy
- Known vs. blind checks
- Control charts
  - X-charts – accuracy and precision
  - R-charts – replicate range

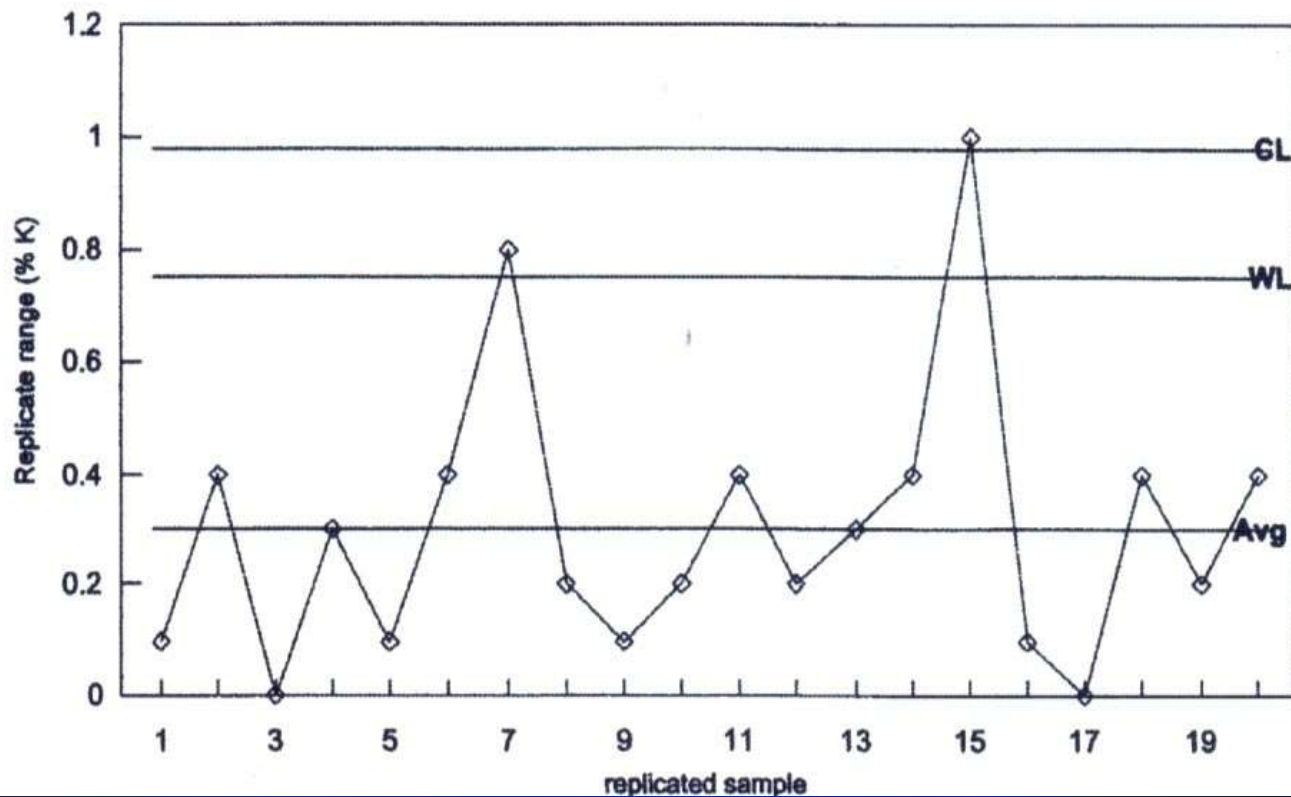
# X - Charts

Figure 1. Example X-Chart



# R - Chart

**Figure 2. Example R-Chart**



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# Sample Handling

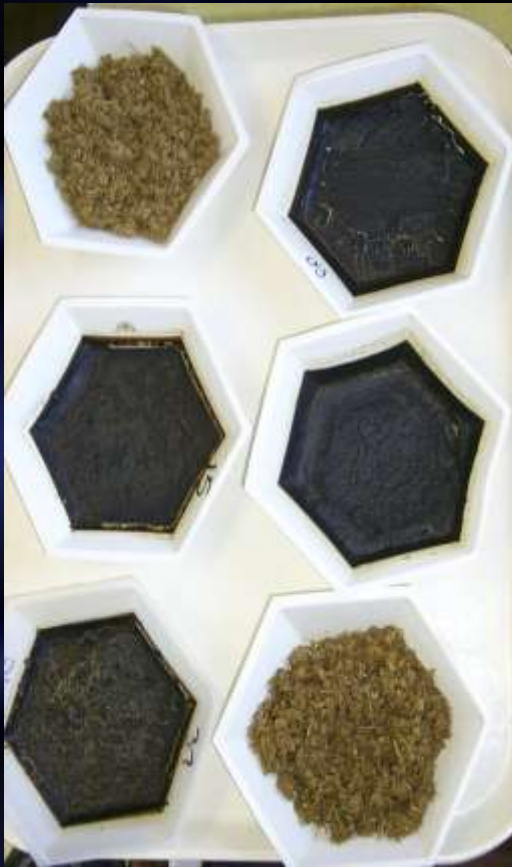
- Biohazards and laboratory safety
- Sample receiving, examination and transfer
- Sample stabilization and storage
- Sample holding times
- Homogenizing and subsampling
- Archiving and disposal

# Sample Holding Times

**Table 4. Maximum holding times for manure at 4° C before specific analyses.**

|   |          |
|---|----------|
| pH                                      | 7 days   |
| Dry matter/Total solids                 | 7 days   |
| Total nitrogen/Kjeldahl nitrogen        | 7 days   |
| Ammonia nitrogen                        | 7 days   |
| Electrical conductivity                 | 6 months |
| Minerals—<br>Total P, K, Ca, Mg, Cu, Zn | 6 months |

# Dry Matter Analysis



# Sample size for DM analysis

**Table 5. Maximum fresh sample size for dry matter determination in open vessels**

| <b>Drying Time</b> | <b>Drying temperature</b> |             |              |
|--------------------|---------------------------|-------------|--------------|
|                    | <b>50°C</b>               | <b>70°C</b> | <b>110°C</b> |
| 6 hr               | Not recommended           | 5 g         | 10 g         |
| 16 hr              | 5 g                       | 10 g        | 20 g         |
| 24 hr              | 10 g                      | 20 g        | 20 g         |

# Dry Matter Effect on Calculated Manure Nutrient Content

| DM% | Nutrient*               |                               |                  |
|-----|-------------------------|-------------------------------|------------------|
|     | N                       | P <sub>2</sub> O <sub>5</sub> | K <sub>2</sub> O |
|     | ----- lbs/wet ton ----- |                               |                  |
| 12  | 7.2                     | 5.5                           | 11.5             |
| 15  | 9.0                     | 6.9                           | 14.5             |
| 18  | 10.8                    | 8.3                           | 17.3             |

\* Based on dry matter analysis of 3.00% N, 1.00% P and 4.00% K

# Suggested Minimum Drying Times at Various Temperatures

|                                       | Temperature       |      |       |
|---------------------------------------|-------------------|------|-------|
|                                       | 50 C              | 70 C | 110 C |
|                                       | ----- hours ----- |      |       |
| Solids<br>(<85%<br>H <sub>2</sub> O)  | 24                | 16   | 6     |
| Liquids<br>(>85%<br>H <sub>2</sub> O) | 48                | 48   | 16    |

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# Laboratory Methods of Analysis

- Total Nitrogen determination
  - Kjeldahl
    - Advantages – low cost, large samples, wet or dry
    - Disadvantages – strong chemicals, labor intensive
  - Combustion
    - Advantages – fast and automated, no strong acids, etc.
    - Disadvantages – high cost of equipment and maintenance and small sample size requirement

# Ammonium Nitrogen

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# Ammonium Nitrogen Methods

- Distillation
- Electrode
- Colorimetry using an autoAnalyzer

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# Digestion and Dissolution Methods

- P, K, Ca, Mg & trace minerals
  - Digestion
    - Dry Ashing
    - Microwave assisted acid digestion
    - Nitric and hydrochloric acid digestion with peroxide
    - Nitric and hydrochloric with block digester

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# Methods of Determination for P, K Ca, Mg and trace elements

- Atomic absorption spectrophotometer
- Inductively coupled plasma spectroscopy
- Colorimetric method for P

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First year availability assumptions – Use  
the values appropriate for your state

Example - Wisconsin

- N – Variable; Dependant on animal species  
and type of application
- $P_2O_5$  – 60%
- $K_2O$  – 80%
- S – 60%

## WASTE ANALYSIS REPORT

Lab Number: 2 Date received: 8/25/02 Account #: 555901  
County: Wood Date processed: 8/25/02 Client: UW Soil & Forage Analysis Laboratory

### Send to:

UW Soil & Forage Analysis Laboratory  
8396 Yellowstone Drive  
Marshfield, WI 54449

### Sample Information

Sample Name: #2  
Material: Dairy Type of Storage: Lagoon  
Storage System: Liquid Type of Bedding:  
Comments:

### Laboratory Analysis

Moisture: 95.20 %

Dry Matter: 4.80 %

#### Estimated Available Nutrient Credits for Manure

|   | Total Nutrients<br>lbs/1000 gal | In 1st Year<br>of Application<br>lbs/1000 gal | If Applied 2<br>Consecutive Yrs<br>lbs/1000 gal | If Applied 3<br>Consecutive Yrs<br>lbs/1000 gal |
|---|---------------------------------|---|---|---|
| Total Nitrogen (Injected)   | 27.09                           | 10.84   | 13.55   | 14.90   |
| Total Nitrogen (Surface Applied)  | 27.09                           | 8.13  | 10.84   | 12.19   |
| Total Phosphorus as P <sub>2</sub> O <sub>5</sub>                             | 15.51                           | 9.31  | 10.86   | 11.63   |
| Total Potassium as K <sub>2</sub> O   | 28.68                           | 22.94   | 25.81   | 27.25   |
| Sulfur  | 1.27                            | 0.70  | 0.83  | 0.89  |
| Estimated Value of Available Nutrients in Surface Applied Manure <sup>1</sup> |                                 | \$7.08  | \$8.43  | \$9.10  |

### Additional Tests

NH<sub>4</sub>-N

Ash

### Additional Information

<sup>1</sup> Value based on commercial fertilizer costs as of 3/1/2002:  
N (urea) \$0.21/lb  
P<sub>2</sub>O<sub>5</sub> (Triple Superphosphate) \$0.24/lb  
K<sub>2</sub>O (Potash) \$0.13/lb  
S (Elemental Sulfur) \$0.23/lb

# WASTE ANALYSIS REPORT

Lab Number: 1 Date received: 9/25/02 Account #: 555001  
County: Wood Date processed: 9/25/02 Client: UW Soil & Forage Analysis Laboratory

Send to:  
UW Soil & Forage Analysis Laboratory  
8396 Yellowstone Drive  
Marshfield, WI 54449

## Sample Information

Sample Name: #1

Material: Dairy Type of Storage: Stack

Storage System: Solid Type of Bedding: hay/straw

Comments:

## Laboratory Analysis

Moisture: 81.50 %

Dry Matter: 18.50 %

### Estimated Available Nutrient Credits for Manure

|   | Total Nutrients<br>lbs/ton | In 1st Year<br>of Application<br>lbs/ton | If Applied 2<br>Consecutive Yrs<br>lbs/ton | If Applied 3<br>Consecutive Yrs<br>lbs/ton |
|---|----------------------------|--|--|--|
| Total Nitrogen (Injected)   | 11.50                      | 4.60                                     | 5.75                                       | 6.33                                       |
| Total Nitrogen (Surface Applied)  | 11.50                      | 3.45                                     | 4.60                                       | 5.18                                       |
| Total Phosphorus as P <sub>2</sub> O <sub>5</sub>                             | 5.75                       | 3.45                                     | 4.03                                       | 4.31                                       |
| Total Potassium as K <sub>2</sub> O   | 11.32                      | 9.06                                     | 10.19                                      | 10.75                                      |
| Sulfur  | 0.51                       | 0.28                                     | 0.33                                       | 0.36                                       |
| Estimated Value of Available Nutrients in Surface Applied Manure <sup>1</sup> |                            | \$2.79                                   | \$3.33                                     | \$3.60                                     |

## Additional Tests

NH<sub>4</sub>-N

pH

Ash

## Additional Information

<sup>1</sup> Value based on commercial fertilizer costs as of 3/1/2002:  
N (urea) \$0.21/lb  
P<sub>2</sub>O<sub>5</sub> (Triple Superphosphate) \$0.24/lb  
K<sub>2</sub>O (Potash) \$0.13/lb  
S (Elemental Sulfur) \$0.23/lb

# Summary

- The value of manure testing is highly dependant on sampling technique
- Laboratory methods – a variety of methods seem to be acceptable for N, P and K
- Standardizing dry matter analysis methods is important for calculating proper nutrient credits of fresh (as applied) manure