

# The Establishment of a National Manure Analysis Proficiency Testing Program

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# Overview

- In October 2002 the Minnesota Department of Agriculture received a grant from the U.S. Environmental Protection Agency (EPA) to establish a Manure Analysis Proficiency (MAP) Program.

# Overview -- Partners

- Dr. Robert Miller, Colorado State University, Fort Collins, CO
  - Workshops and statistical analysis
- Minnesota Department of Agriculture
  - Provide manure proficiency samples
  - Certify laboratories for manure testing based on the laboratory performance in the Manure Analysis Proficiency (MAP) Program

# Program Goals

- Provide quality manure proficiency check samples to labs
- Develop an on-going program
- Minimize the program cost to laboratories through other funding sources:
  - Grants
  - Commodity groups
  - Meat producers/packers
  - Other state's agricultural or environmental agencies
  - Sell manure proficiency samples to entities other than laboratories (researchers, crop consultants, livestock producers)

# Manure Check Sample Preparation

# Check Sample Preparation Depends on Manure Type

- Solid
- Liquid
- Slurry

# Solid Manure Preparation

- Collect the manure
- Acidify the manure
- Dry the manure if necessary
- Freeze the acidified manure
- Chop frozen manure in a 60 quart Vertical Chopper Mixer with dry ice
- Screen the chopped and dried manure
- Mix manure in a cement mixer
- Place manure in sample bottles

Enough solid manure for one  
sample set



Lower pH by adding acid while mixing in a cement mixer



# Acidified manure in drying trays





# The dryer



# Trays loaded in the dryer



The dryer has a heater, blower, and vent



Thermostat ranges from 55 to 175 degrees F



The vent controls the amount of fresh air entering the dryer



Manure can also be dried outside



# Not all solid manure samples are dried

- If the solid manure samples are moist enough that they clump together in the cement mixer, they must be dried.
- However, many solid samples are relatively dry and clumping is not a problem. These samples are usually not dried.

# 60 Quart Vertical Chopper Mixer





Serrated blades work better than plain blades



The blades are replaced each year



The blades can also be repositioned for better chopping



# Processing with dry ice



# Pre-chop the dry ice into chunks

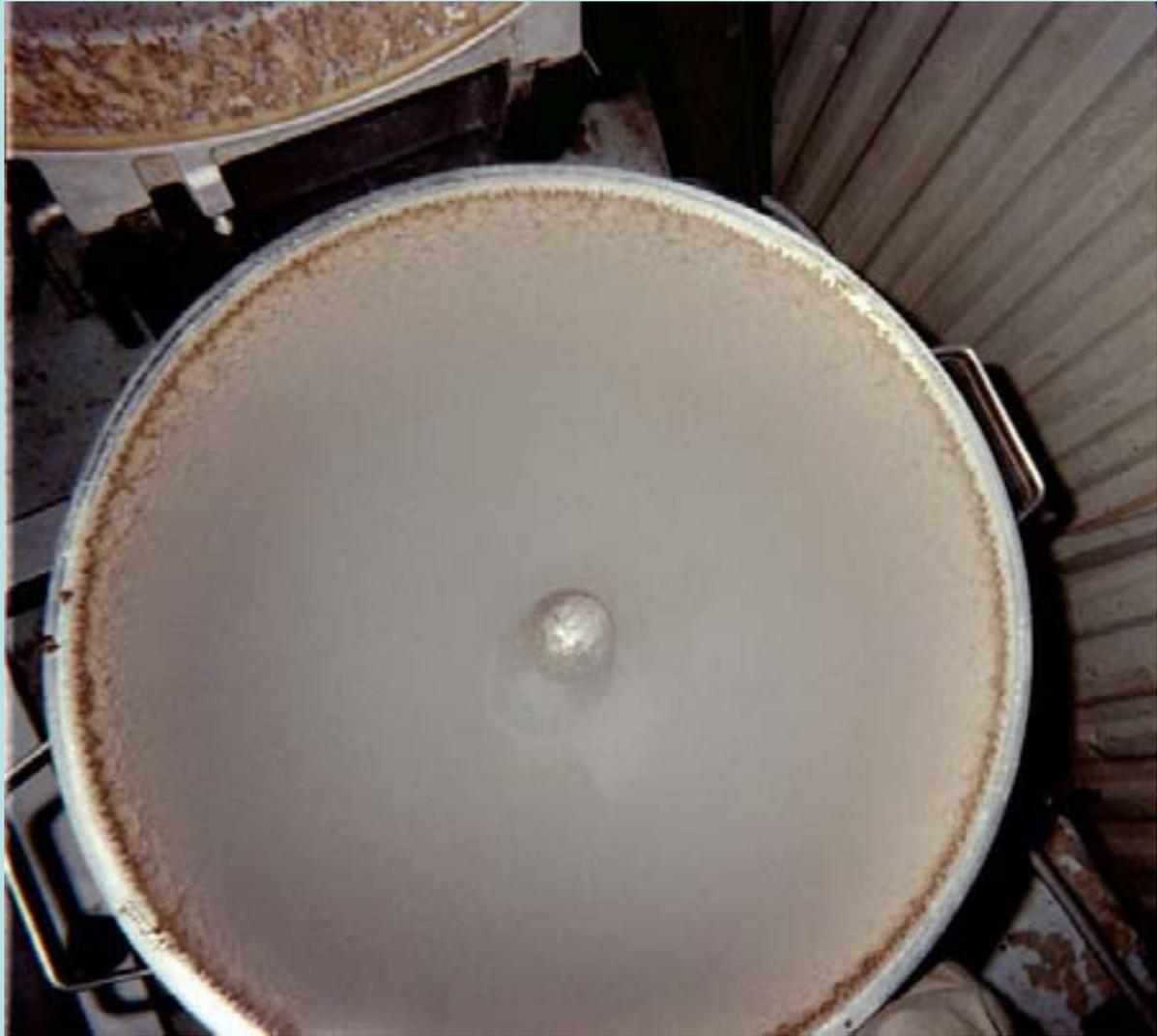


Add dry ice to frozen manure



Note frost on bowl's side







# Screening the manure





# Screening



# Material to discard



# Mixing with the cement mixer



# Plywood cover contains dust



Mix for at least 30 minutes



A smaller mixer is also useful



Large particles “float” to the top



Discard the large “floaters”



# Discarded material



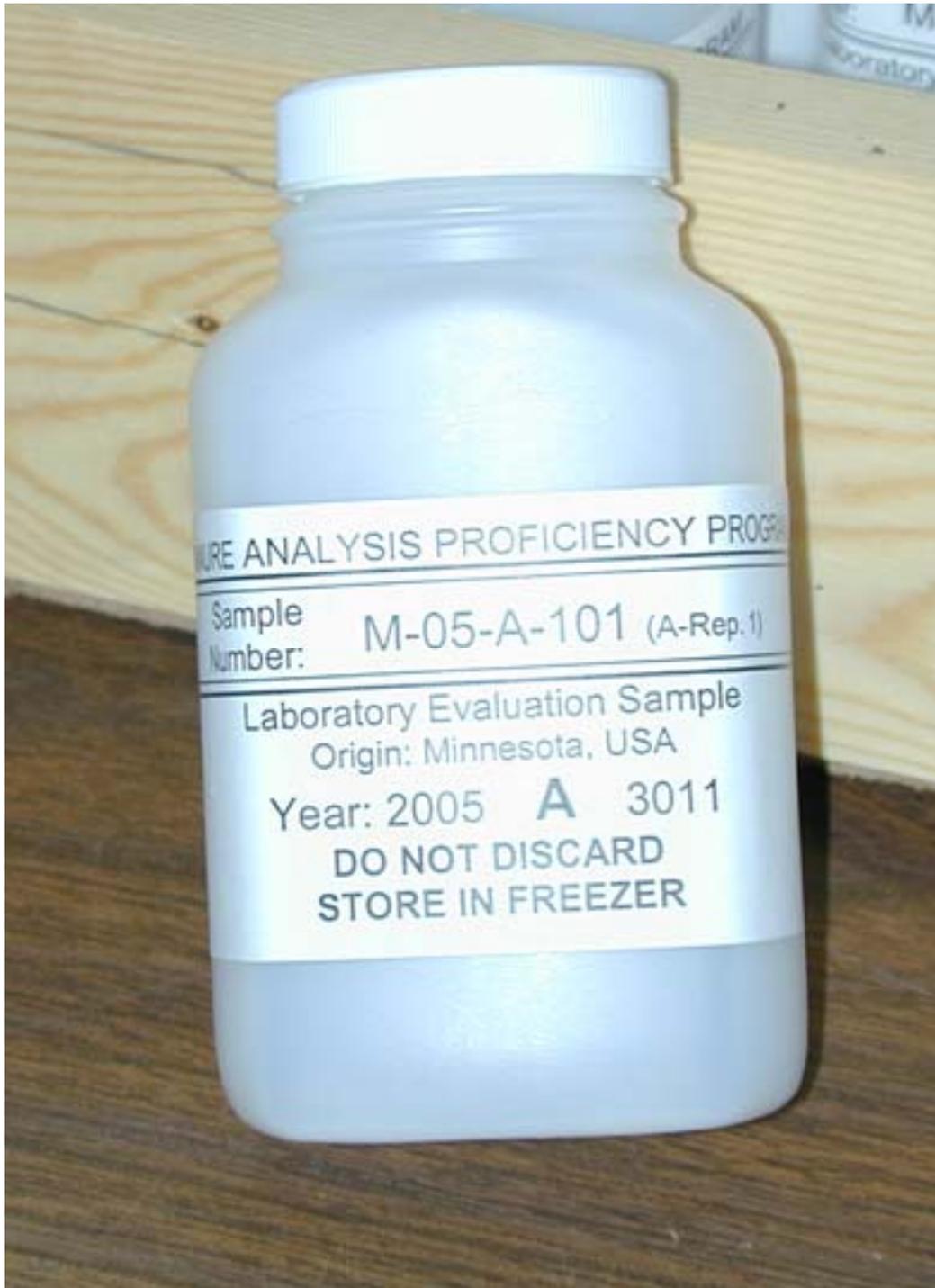
# Filling sample bottles



Labels are placed on sample bottles before filling with manure.







# Sample bottle

Each tray holds 50 bottles



Make four passes with a  $\frac{1}{4}$  cup measuring cup



Fill cup to heaping, tap three times,  
then strike level









# Tap the funnel to dislodge fines

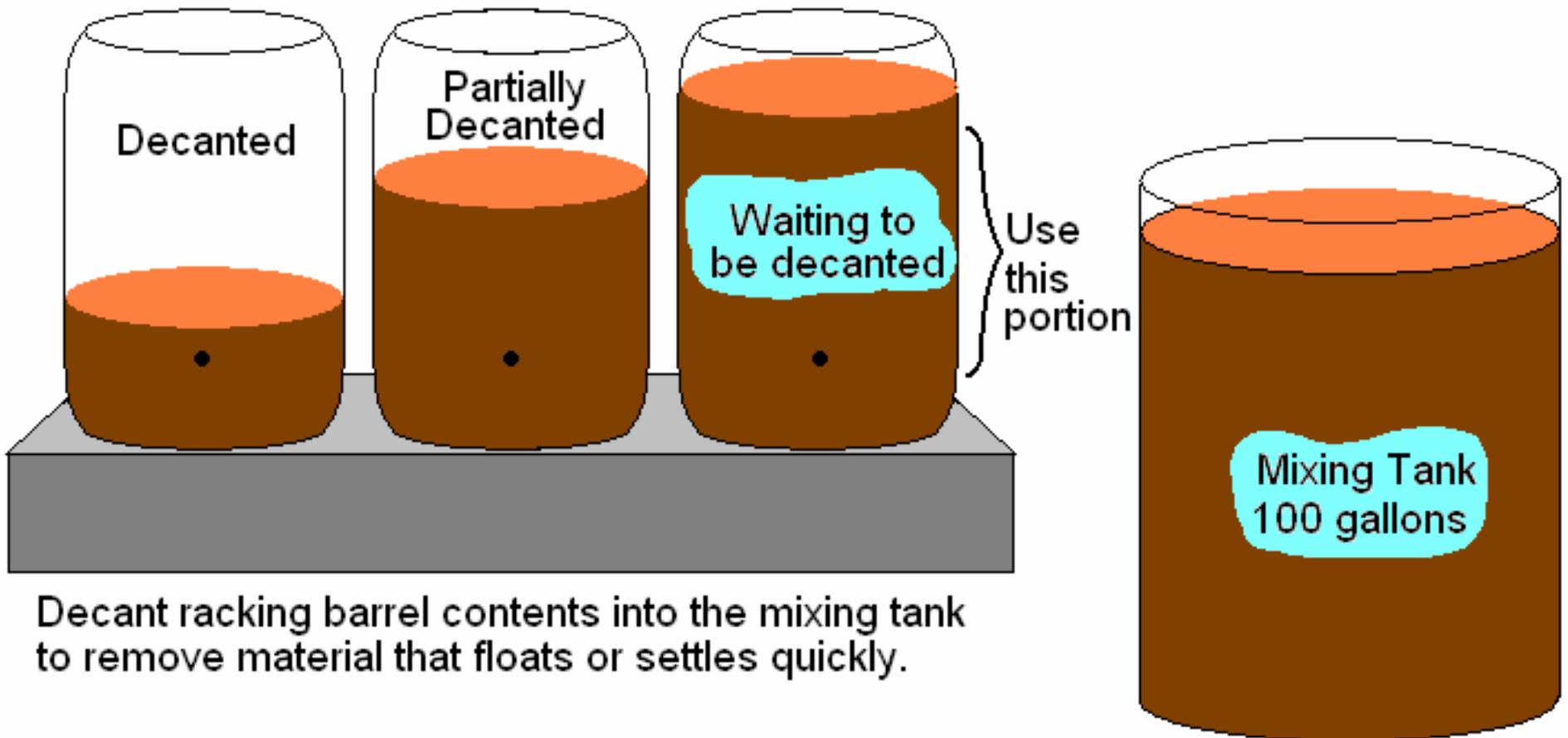


# Preparing liquid samples

- Add water to prepared solid manure, mix well, and let sit in “Racking” barrels.
- Decant liquid from racking barrels and place in 100 gallon mixing tank.
- Add more prepared solid material if desired.
- Agitate with the  $\frac{3}{4}$  HP prop mixer in a baffled tank.
- Use siphon tube or ladle to fill sample bottles.

# Racking Barrels

Three, 55 gallon, Racking Barrels



Decant racking barrel contents into the mixing tank to remove material that floats or settles quickly.

# Racking barrel for decanting



A Robot Coupe hand mixer is useful for the initial mixing



# 100 gallon mixing tank on stand



# 100 gallon tank and $\frac{3}{4}$ HP mixer



Baffles placed inside the mixing tank improve mixing



# Inside of baffled mixing tank



In 2004 a  $\frac{3}{4}$  HP, gear driven mixer with a variable speed controller replaced the  $\frac{1}{20}$  HP mixer that had been used previously.

$\frac{3}{4}$  HP Mixer

$\frac{1}{20}$  HP Mixer





Prop on  $\frac{3}{4}$  HP mixer

Prop on  $\frac{1}{20}$  HP mixer

$\frac{3}{4}$  HP prop mixer (variable speed)



# Tank on stand



# Filling the sample bottles



A siphon tube is faster and neater than using a ladle to fill the bottles.









As the total solids content increases, the siphon tube is more likely to clog. Then a ladle is used.

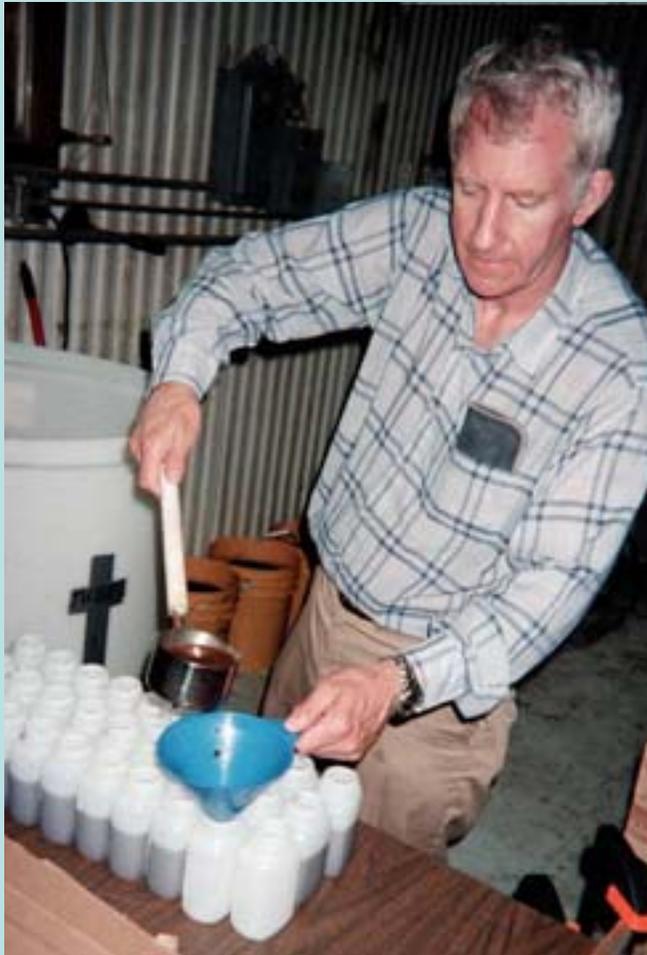
If a ladle is used, solids can stick to the ladle and funnel while filling the sample bottles.



To reduce solids clinging to the ladle, it should be streamlined and smooth (avoid rough edges).



# The funnel should have steep sides



Sides too flat



Steeper sides



A ladle makes it possible to prepare liquid samples with more total solids than when using a siphon tube.

The cardboard behind the funnel catches splatters to keep the sample bottles cleaner.



Starting in 2006, this method will be used to prepare liquid samples with more total solids than in previous exchanges.



# Preparing Slurry Samples

This appears to be a good mix for a slurry type of manure



# Slurry manure just after removal from the Robot Coupe VCM



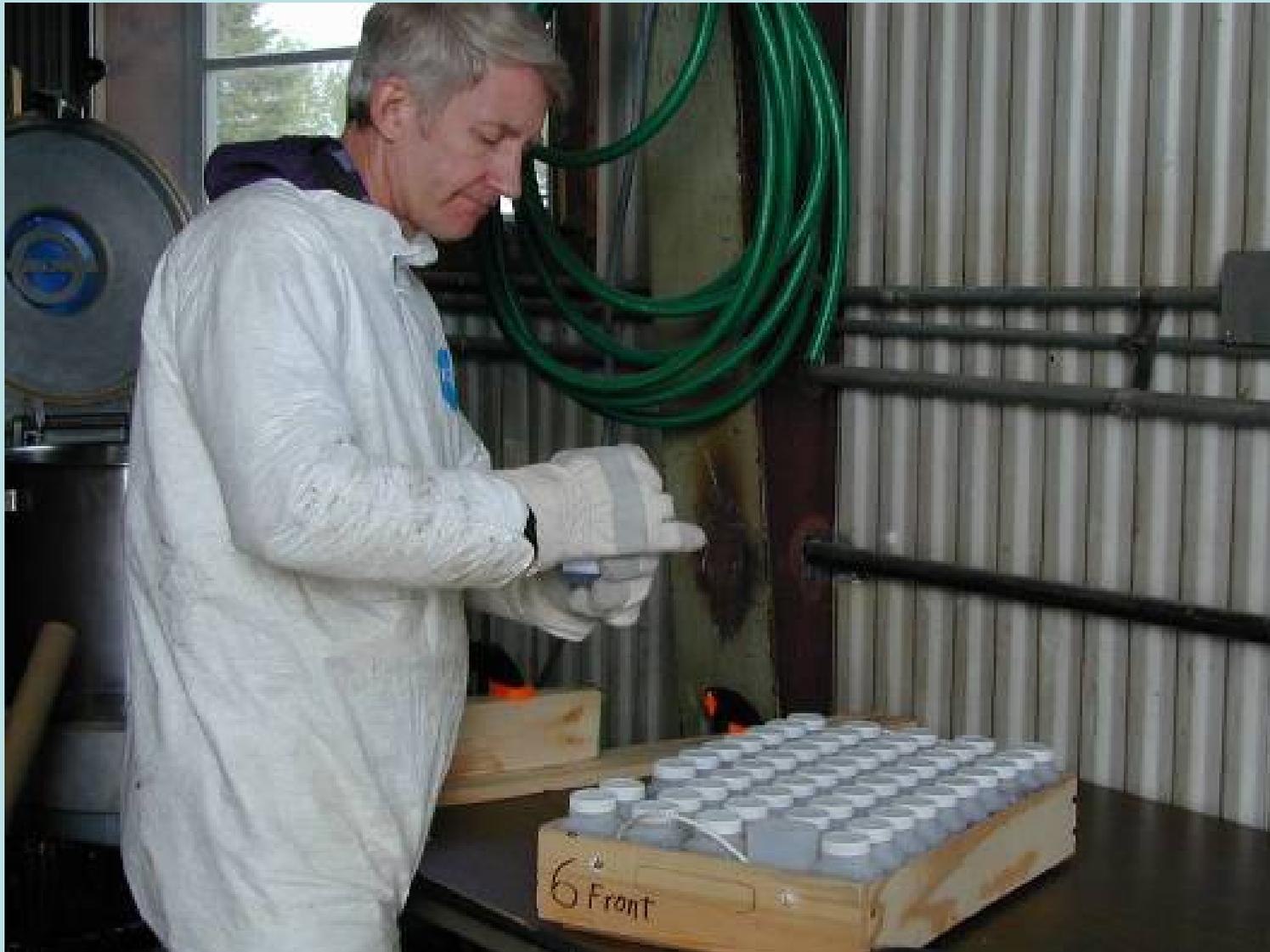
Note the solid/liquid separation after sitting for five minutes.



Because of this separation, I have not yet found a good method to prepare slurry manure.



Gloves make it easier to tighten the lids on the sample bottles





# Shipping samples

The sample trays are designed to facilitate shipping and storage



A full freezer with over three sample sets (1,090 bottles)



70 boxes waiting to be filled



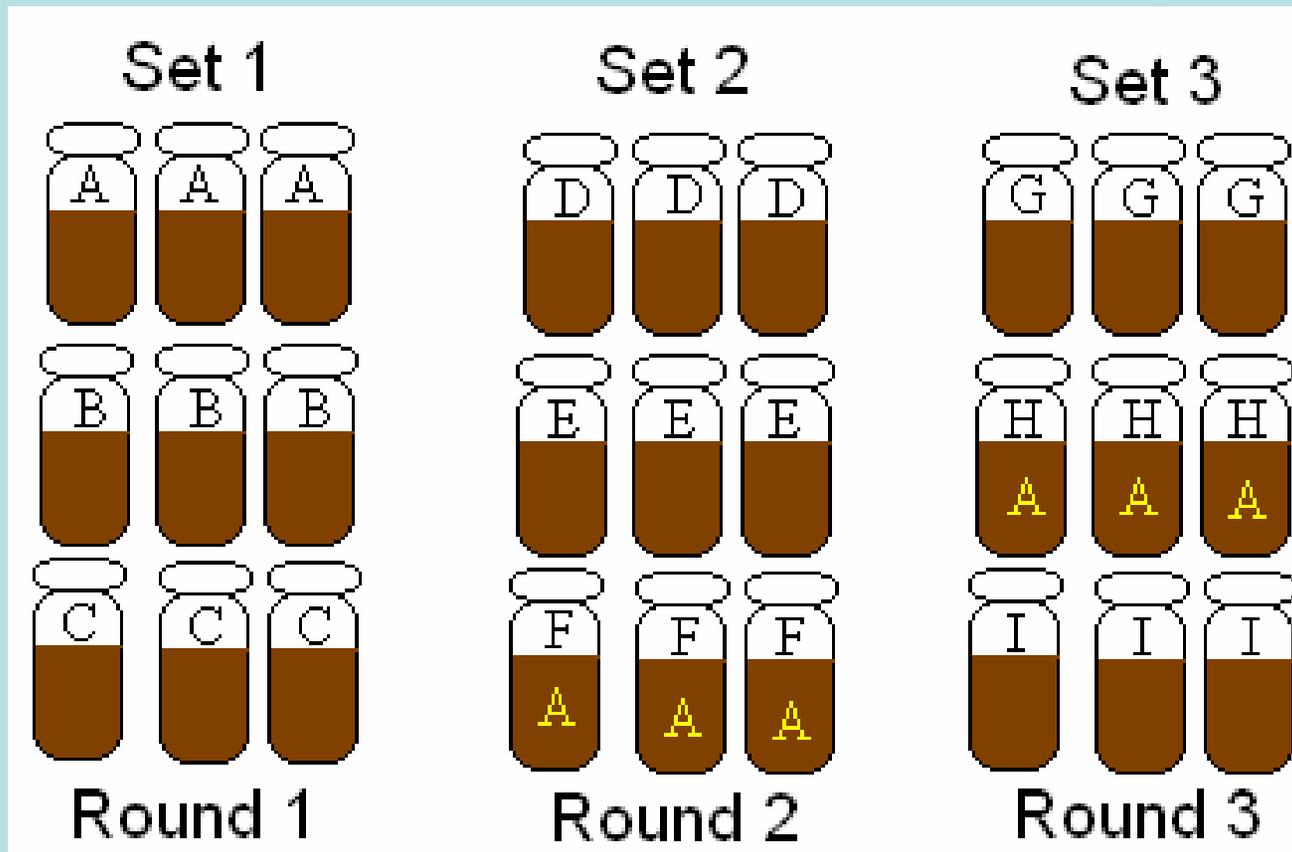
# Nine trays of different samples



Each box has three different manure types in triplicate



# Three rounds of check samples



One set (e.g. "A") is repeated for the three shipments.

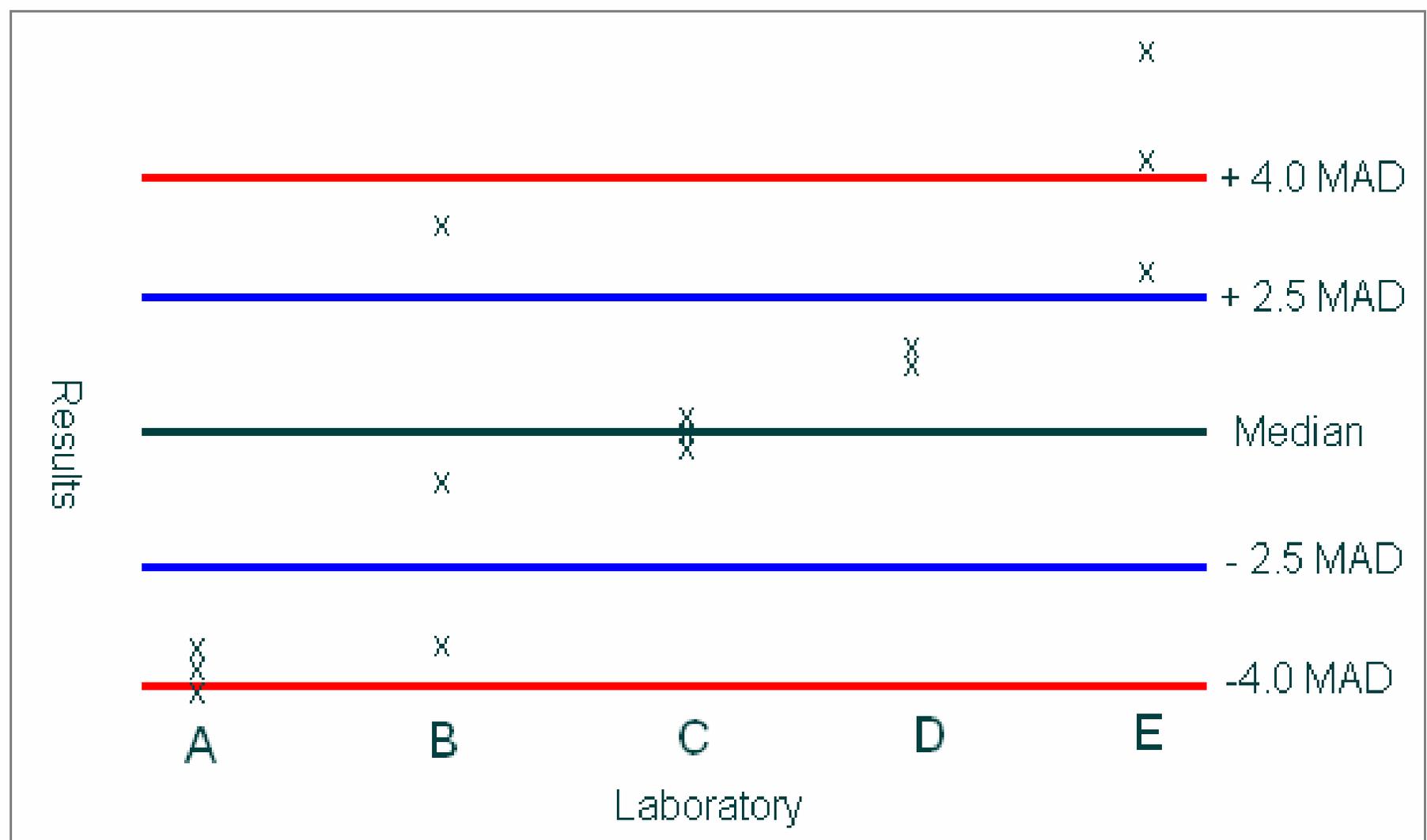
# Ready to ship (overnight FedEx)



# Statistical Analysis

- Dr. Robert Miller analyzes the data using median and Median Absolute Deviation (MAD) units.
- Comparing your lab's results with the median results from the other labs gives you an indication of your lab's accuracy for each test.
- Triplicate samples also allow you to compare your lab's precision with the precision obtained by other labs.

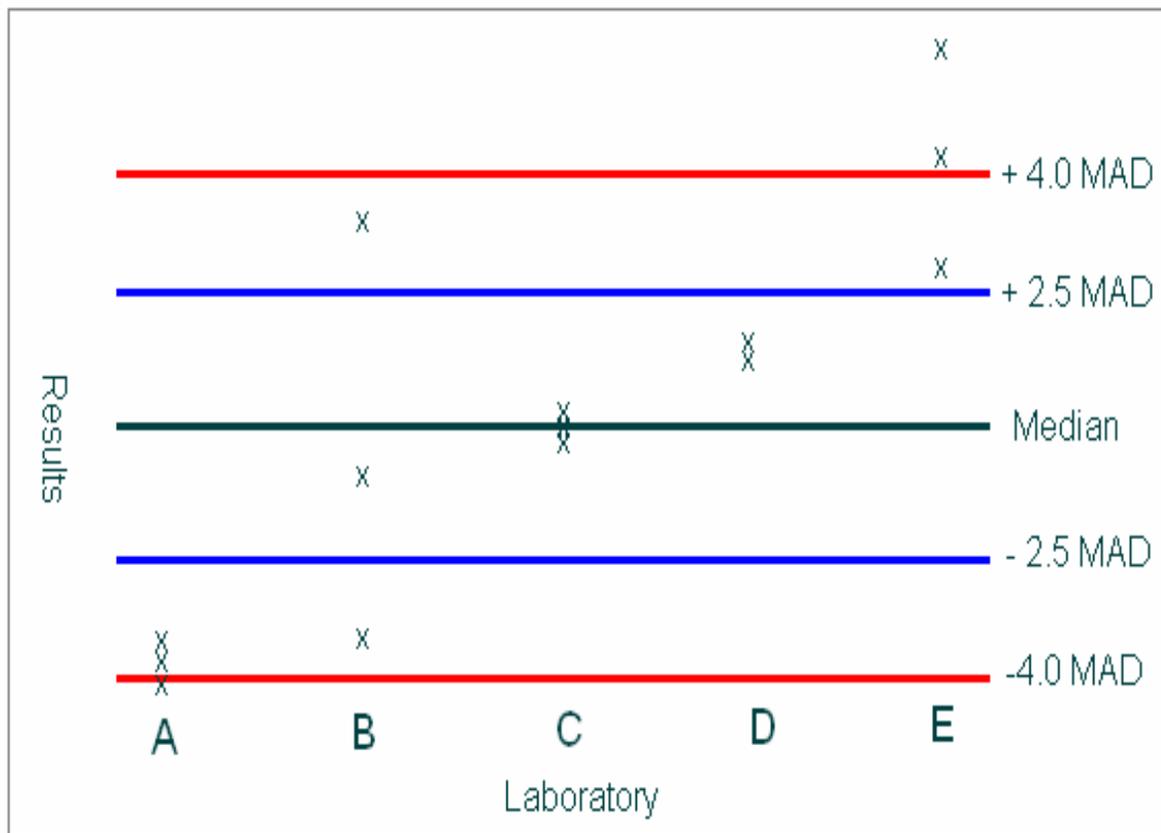
# Results



# Results

- To determine laboratory accuracy, the median and median absolute deviation (MAD) are determined for each test.
- Since each test is run in triplicate it is also possible to evaluate laboratory precision.
- A graph is developed showing the results from all labs for each of the tests.
- The graphs show how your lab compares with other labs participating in the program for both accuracy and precision. NOTE: Labs are not identified; you have to find your lab's data point.

# Graphs of results



- Lab A has good precision but poor accuracy.
- Lab B has good accuracy but poor precision.
- Labs C and D have good precision and accuracy.
- Lab E has poor precision and poor accuracy.

# Types of Tests

- Dry matter content
- Percent moisture
- Total Nitrogen (TKN or Combustion)
- Total Phosphorus
- Total Potassium
- Ammonium Nitrogen
- Water Extractable Phosphorus
- Electrical Conductivity
- Copper
- Zinc
- Sulfur

# Participation Requirements

- Voluntary Program
- Complete an application
- Pay the fee (\$400 for the 2006 program)
- Analyze three sets of three samples in triplicate (27 total per year.)

# Questions or Suggestions?

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