A National Laboratory Proficiency Testing Program for Manure Analysis EPA Grant #: CP-83036801-1

Manure Analysis Proficiency (MAP) Program

Final Report

December 2006

Jerry Floren Dr. Robert O. Miller Bruce Montgomery

TABLE OF CONTENTS

| A. ProposalB. Program objectivesC. Study time frame | |
|---|----|
| 8 5 | - |
| C Study time frame | 6 |
| | 6 |
| D. Highlights | 6 |
| II. Establishment of the advisory board | 8 |
| A. Advisory board members | 8 |
| B. Meeting times and locations | 10 |
| III. Develop a national manure testing proficiency program | 11 |
| A. Identification of laboratories | |
| B. Establish program requirements | 11 |
| C. Statistical standards | 12 |
| D. Equipment and supply costs | |
| E. Description of equipment | 14 |
| 1. Facility | |
| 2. Tools | |
| 3. Manure processing equipment | |
| F. Proficiency testing sample preparation | |
| 1. Proficiency sample logistics and numbering system | |
| 2. Equipment and methods used for sample preparation | |
| a) Solid manure preparation | |
| b) Filling solid manure sample bottles | |
| c) Liquid manure preparation | |
| d) Filling liquid manure sample bottles | |
| e) Slurry manure preparation | |
| 3. Labeling proficiency sample bottles | |
| 4. Shipping proficiency samples | |
| 5. MDA laboratory homogeneity evaluation | |
| IV. Training and technical assistance for the testing industry | |
| A. Project workshops | |
| 1. MAP Laboratory Analysis Workshop (April 24, 2003) | |
| 2. Seventeenth Soil-Plant Analyst's Workshop (February 24-25, 2004) | |
| 3. 2004 Laboratory Analysis Workshop for Manure Testing (June 8, 2004) | |
| 4. MAP Laboratory Analysis Workshop (August 24, 2004) | |
| 5. MAP Laboratory Analysis Workshop (September 12 – 13, 2004) | |
| 6. MAP Laboratory Analysis Workshop (March 16, 2005) | |
| 7. Nitrogen Recommendation Workshop for Laboratories (September 7, 20 | |
| 8. MAP Laboratory Analysis Workshop (May 10, 2006) | |
| B. Other professional workshops | |
| 1. Four-State Soil Testing Lab Conference | |
| American Society of Agronomy | |
| Mid Atlantic Soil Testing Workgroup | |
| | |
| Mid Atlantic Soil Testing Workgroup | |

| 6. International Symposium on Soil and Plant Analysis (ISSPA) | 50 |
|--|----|
| 7. Laboratory Analysis Workshop | 51 |
| 8. Laboratory Analysis Workshop | 52 |
| V. Quantify long-term improvements in manure analysis testing | 53 |
| A. Manure reference repository (library) | |
| B. Double blind study | |
| 1. Comparison of the single blind to the double blind | 55 |
| 2. Laboratory reports to their clients | 56 |
| 3. Analyses routinely performed by laboratories | 59 |
| C. Analysis variability of participating labs over time | 60 |
| D. Certified labs vs. non-certified labs | 61 |
| VI. Program sustainability | |
| A. Projected 2007 revenue and expenses for MAP and certification | 64 |
| B. Minnesota Department of Agriculture funding | 71 |
| VII. Future manure lab analysis needs and existing reference methods | 72 |
| A. Water Extractable Phosphorus (WEP) | 72 |
| B. Electrical Conductivity (EC) | 72 |
| C. Ammonium Nitrogen | |
| D. Updates for Recommended Methods of Manure Analysis | |
| 1. Sample preparation prior to laboratory analysis | 73 |
| 2. Published method for the Water Extractible Phosphorus (WEP) test | 73 |
| 3. Differences in macro versus micro Total Kjeldahl Nitrogen (TKN) | 73 |
| 4. Future tests to include in the MAP Program | 73 |
| 5. Laboratory customer reports and transcription errors | 73 |
| 6. Diagnosing laboratory errors and trouble shooting | 74 |
| E. Promotion of existing reference methods | 74 |
| VIII. Promote consistent, simplified recommendations for manure sampling | 76 |
| A. Workshops and presentations for manure lab clients | 76 |
| 1. 2005 Upper Midwest Manure Handling Expo | |
| 2. Open house Southern Research and Outreach Center | 79 |
| 3. Minnesota Water 2005 and Annual Water Resources Joint Conference | 79 |
| 4. Minnesota Water 2006 and Annual Water Resources Joint Conference | 79 |
| IX. Evaluate program impacts | 80 |
| A. Trends from the beginning to end of study | 80 |
| B. Assessment of analytical methods | 81 |
| 1. General | |
| 2. Accuracy and Precision | 81 |
| 3. Total Solids | |
| 4. Total Nitrogen (TKN or N-Combustion) | |
| 5. Potassium | 82 |
| 6. Phosphorus | 82 |
| 7. Ammonium Nitrogen | 83 |
| 8. Copper | 83 |
| 9. Sulfur | |
| 10. Zinc | |
| 11. Electrical Conductivity (EC) | 84 |

| | 12. Water Extractable Phosphorus | |
|------|--|-----|
| C | . Transcription errors | |
| X. | Appendix A – Certification of laboratories for manure analysis | |
| XI. | Appendix B – Advisory board meeting minutes | 88 |
| Α | . Indianapolis, IN (November 12, 2002) | 88 |
| B | . Denver, CO (November 4 and 5, 2003) | |
| C | . Newark, DE (September 13, 2004) | |
| XII. | Appendix C – Participating MAP Laboratories | |
| Α | . Participating MAP Laboratories in 2003 | |
| B | Participating MAP Laboratories in 2004 | 109 |
| C | Participating MAP Laboratories in 2005 | |
| D | Participating MAP Laboratories in 2006 | |

I. Introduction

This final report reflects the work completed with the project titled, *A National Laboratory Proficiency Testing Program for Manure Analysis*, EPA Grant #: CP-83036801-1. The program became known to the participants as the Manure Analysis Proficiency (MAP) Program, and the MAP acronym is used frequently in this report.

Numerous organizations have reported that proper manure crediting is the weakest link and a key component in nutrient management planning. Achieving uniform application rates, proper timing, and placement are additional manure handling challenges faced by the livestock industry.

Up until ten years ago the manure testing industry was chaotic. Unlike soil testing there were no standardized testing methods for labs to use. Additionally, methods and terminology for reporting back to livestock producers varied drastically. In 2003 the manual, *Recommended Methods of Manure Analysis*, was published. The manual had its roots from a joint meeting of regional soil testing workgroups in 1996 in Raleigh, North Carolina. Earlier that year the workgroups from NCR-13, SERA-6, and NEC-67 participated in a manure sample exchange that was discussed at the Raleigh meeting. This exchange and discussion sparked interest in developing a manure testing manual. The manual was published by the University of Wisconsin Extension and was a collaboration of the workgroups and the Minnesota Department of Agriculture. The manual is available on the Internet at: http://uwlab.soils.wisc.edu/pubs/A3769.pdf

This project represents the next step to improve laboratory manure analysis: a national manure proficiency program providing quality proficiency samples to nearly all laboratories in the United States and Canada that analyze manure. This program is named the Manure Analysis Proficiency Program, and it is the only program providing manure proficiency samples in North America.

The "Proposal" and "Program Objectives" are quoted directly from the grant proposal that initiated this project.

A. Proposal¹

Establish a national Laboratory Proficiency Testing Program for Manure Analysis through the expansion and modification of the Minnesota Department of Agriculture Manure Testing Laboratory Certification Program. This funding would build the framework for a national program under the umbrella of the North American Proficiency Testing (NAPT) program, purchase the required equipment to provide high quality

¹ Quoted from, *A National Laboratory Proficiency Testing Program for Manure Analysis*, a grant proposal submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, Bruce Montgomery, Dr. Robert Miller, and David Kral, page 1.

manure proficiency samples for 75-100 participating laboratories, resolve technical difficulties with laboratory manure analysis, build alliances with national stakeholders, and lay the foundation for facilitating technical transfer and outreach to laboratories. One important underlying goal is to design a long-range goal that will be as self-sufficient as possible.

B. Program objectives²

A national proficiency testing program would seek to ensure accurate and understandable manure test results for livestock producers. These objectives would be accomplished through: development of laboratory proficiency testing to ensure the accuracy of manure test results; identify future reference analysis methods; promote consistent recommendations for manure sampling; promote consistent reporting and interpretation of test results; and assist states, laboratories, and other key information providers in providing assistance to producers in the development and implementation of environmentally and economically sound nutrient management plans. Program oversight would be accomplished through establishment of an advisory board of stakeholders.

C. Study time frame

The original budget period was for two years from 09/01/2002 to 09/30/2004. The timeframe was extended at no additional cost to the Environmental Protection Agency (EPA) for four years through 09/30/2006. Participating laboratories received manure proficiency sample sets from 2003 through 2006 and 38 laboratories received an additional set of two manure samples in 2006 as a double blind study.

D. Highlights

The following are some highlights of this report:

- Thirty-six different manure proficiency samples were shipped during 12 exchanges from 2003 through 2006 to over 60 laboratories. (page)
- Laboratory enrollment decreased from a high of 84 in 2003 to the current level of 69 in 2006. However, the number of laboratories submitting results to the MAP Program actually increased from an average of 65.3 per quarter in 2003 to an average of 67.0 per quarter in 2006. (Appendix XII.)
- The 12 exchanges were "single blind" proficiency samples. "Single blind" samples are defined a samples identified to the labs as proficiency samples from the MAP Program. One "double blind" exchange was submitted to 38 randomly selected laboratories in 2007. "Double blind" samples are samples previously

² Ibid. Montgomery, B., etal. August 7, 2002.

used in proficiency testing but are shipped from a private partner in such a manner that the labs are unaware the samples originated from the MAP Program. These labs did not know the analytical concentrations of the samples. (page)

- For most analyses on liquid and solid manure, the majority laboratories provided satisfactory results. Total solids (or percent moisture) and total nitrogen by either the combustion method or the Kjeldahl method were precise. However, there is a concern for variability phosphorus analysis based on results from the double blind test. (page)
- It is feasible to prepare high quality liquid and solid manure proficiency samples; however, a technique to adequately prepare slurry manure proficiency samples was not found. (page)
- Data was analyzed using median and Median Absolute Deviation (MAD) values to reduce the effect from the outliers. Typically this data is not normally distributed, perhaps because of transcription errors. Confidence Limits were established using ± 2.5 MAD units from the median. For most analyses, 85% to 90% of laboratories are within 10% to 20% of the median except for dilute samples near the detection limits. (page)
- It is highly unlikely this program can continue to operate at the current level solely on the fees collected from laboratories. Increasing the fees to cover costs will likely result in a significant number of laboratories dropping out of the MAP Program and actually reduce the income generated from fees. Outside funding will be needed to continue running the program at current levels. (page)

II. Establishment of the advisory board

Tasks: ³ Identify stakeholders from public and private laboratories, testing industry organizations, state and federal governments, Extension, livestock commodity groups, custom manure applicators, agribusiness and environmental organizations. Establish Advisory Board of interested individual and select a chairperson. Conduct sufficient Advisory Board meetings prior to program implementation to establish program structure and plan activities. Conduct regular Advisory Board meetings thereafter to provide sufficient program oversight and planning for activities.

Note: The Advisory Board meeting minutes are in Appendix XI.

A. Advisory board members

Dr. Greg Binford University of Deleware Plant and Soil Science 165 Townsend Hall Newark, DE 19716 Phone: 302- 831-2146 Fax: E-mail: binfordg@udel.edu

Mr. Nat Dellavalle Dellavalle Laboratory, Inc. 1910 West McKinley Suite 110 Fresno, CA 93728-1298 Phone: 559-233-6129 Fax: 559-268-8174 E-mail: ndellavalle@dellavallelab.com

Mr. Jerry Floren Minnesota Department of Agriculture APPD-4 90 West Plato Boulevard Saint Paul, MN 55107-2094 Phone: 651-297-7082 Fax: 651-297-2271 E-mail: jerry.floren@state.mn.us

³ Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 3.

Dr. Robert Miller Soil and Crop Sciences Department Colorado State University Fort Collins, CO 80523 Phone: 970-217-2572 Fax: 970-416-5820 E-mail: rmiller@lamar.colostate.edu

Mr. Bruce Montgomery Minnesota Department of Agriculture APPD-2 90 West Plato Boulevard Saint Paul, MN 55107-2094 Phone: 651-297-7178 Fax: 651-296-7386 E-mail: bruce.montgomery@state.mn.us

Mr. John Peters Soil and Forage Analysis Laboratory Soil Science Department 8396 Yellowstone Drive Marshfield, WI 54449-8401 Phone: 715-387-2523 ext. 4 Fax: 715-387-1723 E-mail: jbpeter1@facstaff.wisc.edu

Mr. Keith Reid Ontario Ministry of Agriculture and Food Stratford Resource Centre 581 Huron Street Stratford, ON N5A 5T8 Phone: 519-271-9269 Fax: 519-273-5278 E-mail: keith.reid@omaf.gov.on.ca

Dr. Charles Shapiro University of Nebraska Haskell Agricultural Laboratory 57905 866 Road Concord, NE 68728-2828 Phone: 402-584-2803 Fax: 402-584-2859 E-mail: cshapiro@unl.edu

Dr. Ann Wolf Agricultural Analytical Services Lab 111 Tower Road University Park, PA 16802 Phone: 814-863-0841 Fax: 814-863-4540 E-mail: amw2@psu.edu

B. Meeting times and locations

The MAP Advisory Board met three times at the following locations on the dates indicated:

- Indianapolis, IN on November 12, 2002
- Denver, CO on November 4 and 5, 2003
- Newark, DE on September 13, 2004

III.Develop a national manure testing proficiency program

Tasks:⁴ Identify additional U.S. laboratories offering manure testing services and establish participation in the program. Develop and distribute program requirements to participating laboratories. Purchase equipment and establish methods needed for provision of homogeneous check samples to participating laboratories. Establish statistical standards for acceptable laboratory performance. Conduct proficiency testing (three rounds per year) based on laboratory performance of check sample analysis for routine test parameters. Assist laboratories in improving analytical methods to ensure acceptable performance. Make program information available in a variety of formats to promote use of manure testing services provided by participating laboratories.

A. Identification of laboratories

The following laboratories received a mailing and application to enroll in the MAP program in 2003:

- Laboratories currently enrolled in the NAPT soil and compost proficiency programs
- Laboratories currently certified by the Minnesota Department of Agriculture for manure analysis
- Laboratories identified by Web searches that indicated they analyzed manure

Approximately 200 laboratories were sent applications (most in the NAPT soil testing program) and 84 enrolled for 2003. However, only about 65 of the 84 actually sent in results. The number of labs sending in results has ranged from 65 to 67 from 2003 to 2006, and we feel this is the vast majority of laboratories that analyze significant numbers of manure samples.

B. Establish program requirements

At the first Advisory Board meeting (November 2002) we proposed evaluating the following tests in the MAP Program:

- Total Nitrogen
- Total phosphorus
- Total potassium
- Dry matter content
- Zinc

⁴ Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 3.

- Copper
- Electrical conductivity

Based on feedback from the Advisory Board, ammonium nitrogen was also included, and two nitrogen methods (total Kjeldahl nitrogen and nitrogen by combustion) would be evaluated separately. It was also proposed that the samples would be provided with triple replicates and the fee for the first year would be \$100 for laboratories in another NAPT proficiency program and \$200 for labs solely in the MAP Program.

C. Statistical standards

Based on lab results in NAPT soil testing program and the NAPT compost testing program, normally distributed data was not expected in the manure proficiency program. Results from each of our exchanges have indeed demonstrated the manure proficiency data is not normally distributed. and it seemed logical to handle the outliers in the manner used in the NAPT soil and compost proficiency programs.

Robust statistics allow the inclusion of all data by using the median instead of the average. Both the NAPT soil and compost proficiency programs use robust statistics by calculating the median instead of the average and the Median Absolute Deviation (MAD) instead of the standard deviation. Since laboratories were already familiar with the use of robust statistics using the median and MAD the Advisory Board decided to use the same statistics for the MAP Program.

A significant difference with the MAP Program compared to the NAPT soil and compost programs is that the MAP Program provides each sample in triple replicates. These triple replicates allow us to evaluate laboratories for precision in addition to accuracy.

D. Equipment and supply costs

The following table provides a listing of all equipment purchased during the life of this project. No equipment purchases have met the EPA definition for equipment of \$5,000 or more dollars.

Table 1: List of all purchase orders issued for the MAP Program exceeding \$500 sorted by date. Some of these are listed as "Equipment" by the state, but none meet the \$5,000 EPA definition for equipment. Cells highlighted in yellow are one time purchases. Turquoise cells are ongoing expenses.

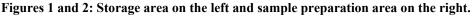
| Date | Vendor | Item | Amount |
|------------------------|-----------------------------|--|-------------------|
| 11/12/2002 | St. Cloud Restaurant Supply | Robot Coupe MP-600 Mixer | \$900.00 |
| 1/6/2003 | Geotech Environmental | Decaport Splitter | \$1,340.00 |
| 2/13/2003 | B&D Equipment | Robot Coupe VCM 60 quart (used) | \$2,500.00 |
| 3/24/2003 | VWR Scientific | Mettler Balance | \$1,071.60 |
| 3/24/2003 | VWR Scientific | 1 set digestion tubes | \$616.00 |
| 3/24/2003 | VWR Scientific | Various lab equipment | \$573.06 |
| 3/24/2003 | Foss North America | Rack for 20 digestion tubes | \$500.00 |
| 4/7/2003 | General Parts and Supply | Knife set for Robot Coupe VCM 60 qt | \$530.78 |
| 4/16/2003 | VWR Scientific | 4 cartons sample bottles & 1 cylinder | \$619.24 |
| 4/20/2003 | P J Distributing | Four, 24.9 cu ft freezers | \$1,856.00 |
| <mark>6/12/2003</mark> | Toolfetch | Cement mixer, Imer 350E | \$2,399.97 |
| 6/12/2003 | VWR Scientific | 6 cartons sample bottles | \$895.50 |
| 8/18/2003 | Central Lakes College | Install Electric | \$1,900.00 |
| <mark>9/15/2003</mark> | En Pointe Technologies | Projector, computer | \$2,215.36 |
| 10/17/2003 | Grovhac | Mixer | \$1,684.00 |
| 10/17/2003 | Grovhac | Mixer Stand | \$925.00 |
| 1/22/2004 | General Parts and Supply | Knife set for Robot Coupe VCM 60 qt | \$545.78 |
| 4/15/2004 | VWR Scientific | 8 cartons sample bottles | \$1,220.48 |
| 6/10/2004 | VWR Scientific | 4 cartons sample bottles | \$610.24 |
| 10/4/2004 | VWR Scientific | 9 cartons sample bottles | \$1,373.04 |
| 9/1/2005 | General Parts and Supply | Knife set for Robot Coupe VCM 60 qt | \$578.82 |
| 9/6/2005 | Fisher Scientific | 10 cartons sample bottles | \$1,290.10 |
| 7/31/2006 | VWR Scientific | 11 cartons sample bottles | \$1,949.61 |
| | | Total for all purchase orders greater than \$500.00 | \$28,094.58 |

E. Description of equipment

This section describes the tools, equipment, and various methods used during this project.

1. Facility

Preparation and storage of the manure proficiency samples is conducted at a shop rented from the Central Lakes Ag Center in Staples, Minnesota. This facility is leased for \$2,700 per year.







2. Tools

A variety of stands, trays, tables, and holders were built for this project. The following tools were helpful:

- Saws (power miter saw, circular saw, saber/jig saw, coping saw, hacksaw)
- Wrenches (adjustable end, pipe wrench, socket set, and combination wrenches)
- Cordless and variable speed electric drills and drill bits
- Screwdriver set
- Sander
- Clamps

Protective gear

- Hearing protector
- Steel toe boots
- Dust masks
- Eye protection
- Tyvek coveralls

- Rubber apron
- Kevlar gloves
- Rubber gloves

3. Manure processing equipment

The major pieces equipment essential for this project are described in this section:

- 60 quart Robot Coupe Vertical Cutter Mixer (VCM)
- ³/₄ horsepower Grovhac mixer, variable speed with stand
- 12.1 cubic foot Imer electric cement mixer
- Four chest freezers
- Dryer

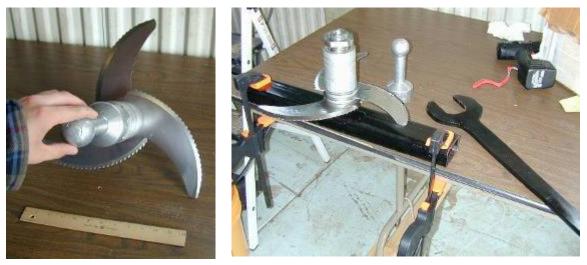
Figures 3 and 4: The Robot Coupe 60 Quart Vertical Cutter Mixer (VCM)



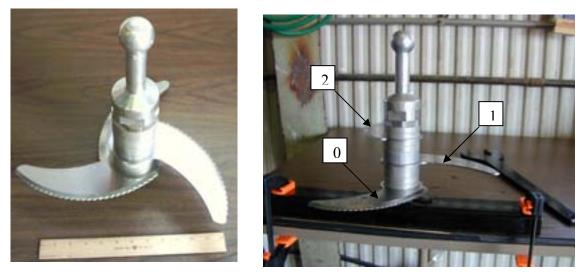


It is necessary to reduce the manure particle size, and the Robot Coupe 60 quart Vertical Cutter Mixer (VCM) handled this chore with ease on a variety of different manure types and consistencies. It also is useful to premix solid manure with water before adding it to the mixing barrel for final mixing with a prop mixer.

Figures 5 and 6: Serrated knife blades produced better results than smooth blades. The large wrench and the arbor holder were fabricated by a local blacksmith.



Figures 7 and 8: Spacers on the arbor allow the three blades to be placed at different depths. The numbers "0", "1", and "2" are stamped on the blades. The lowest blade is "0", the middle blade is "1", and the top blade is "2." Also note the blade bevel is up – the arbor assembly spins counter clockwise.



Figures 9 and 10: The Robot Coupe VCM was useful for reducing the particle size in dry manure and also for pre-mixing processed solid manure with water before mixing liquid manure with the Grovhac prop mixer.



Prop mixer and stand manufactured by Grovhac

Figure 11: A ³/₄ horsepower variable speed prop mixer prepared the liquid manure. Two mixing barrels were used, a cut down 50 gallon barrel and a 100 gallon barrel. Baffles were also added to the mixing barrels. The best method to prepare liquid manure was to add water to solid manure that had been processed in the Robot Coupe VCM to reduce particle size.



Figures 12 and 13: The 50 gallon mixing tank is on the left and the 100 gallon mixing tank is on the right. Aluminum baffles were added to aid mixing.



Figures 14 and 15: Solid manure is mixed in the Imer Workman 350E Electric cement mixer, a 12 cubic foot mixer. A wooden cover keeps the dust contained within the mixer.



Figures 16 and 17: A smaller mixer was used for some solid samples before the Imer mixer was purchased. This mixer was non-project equipment.





Dryer

Moist solid manure cannot be mixed in the cement mixer, so it is often necessary to dry the manure. Fortunately the space we rented had a dryer to use for this project.

Figures 18, 19, 20, and 21: Wooden trays with heavy duty screening on the bottom were built to hold the manure in the dryer.







Figures 22 and 23: The dryer has controls for heating and outside ventilation.





Figure 24: If a dryer is not available, manure can be dried outdoors on a tarp.



Freezers and wooden trays

The original intention had been to purchase a walk-in freezer. However, four chest freezers seem to work well. About 75 wooden trays were made were made in two sizes to hold and organize the samples.

Figures 25 and 26: The fourth chest freezer is near the back on the left. Bulk manure in four gallon pails will be processed in the Robot Coupe VCM before bottling or mixing with water for liquid manure.



Figures 27 and 28: Trays that fit snugly maximize the freezer capacity. Three smaller trays (30 bottles) fit over the freezer motor and compressor. Five larger trays (50 bottles) fit from top to bottom. A full freezer holds 1,090 sample bottles – more than enough for one exchange.



Figures 29 and 30: Wooden trays maximize the freezer space and make it easier to find specific sample sets.



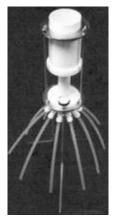


Figure 31 and 32: Robot Coupe MP600 Turbo hand mixer (better mixing results were obtained by using the Robot Coupe VCM).





Figure 33: Dekaport cone Teflon sample splitter (the hoses were too small and quickly clogged with manure solids)



F. Proficiency testing sample preparation

1. Proficiency sample logistics and numbering system

Each year from 2003 through 2006 participating laboratories received three rounds of manure proficiency samples. Each set consisted of three different types of manure with triple replicates (nine sample bottles per set). Each sample bottle contains about 200 ml of manure. Each year there are 27 sample bottles analyzed by each participating laboratory. One of the manure samples was also repeated for each of the three rounds giving another indication of precision.

Figure 34: Three rounds of samples. Samples A, E, and G are the same sample so results can be compared from round to round. Triple replicates (101, 102 and 103) allow evaluation for precision in addition to accuracy.



Figure 35: One set of Samples D, E, and F ready to ship to a participating laboratory for the second round of 2005. Each sample has three replicates labeled 101, 102, and 103. The bottles are 250 ml and contain about 200 ml of manure.



2. Equipment and methods used for sample preparation

a) Solid manure preparation

Figure 36: Thirty-two gallon garbage can of solid manure (turkey litter) before processing. This is enough for a single sample set (280 bottles).



The 32 gallon plastic trash cans with lids were useful for collecting manure in the field. They also fit in the chest freezer so the manure can be frozen before processing in the Robot Coupe VCM without transferring to a smaller container. Figure 37: Step 1 — Process manure in the Robot Coupe VCM to reduce particle size.



Reducing the particle size of solid manure is important in this proficiency program. It is acceptable to have some fibers such as hair, feathers, or straw up to two centimeters. However, the vast majority of particles should be in the range of one to three millimeters. Often the manure is processed several times in this machine. Typically it is processed "warm" from the field before placing in the freezer. Then it is processed again while frozen. If the particle size is still too large it will be returned to the freezer and processed again with the addition of dry ice. If the sample is too wet it can be dried and reprocessed after drying.

Figures 38 and 39: The finest particle size was obtained by processing frozen manure with dry ice. A six to eight pound block of dry ice is split in half and then chopped into finer pieces.





Figures 40 and 41: Three to four pounds of dry ice is added to the frozen manure in the VCM. The VCM is run for about 30 seconds on low speed, opened and contents stirred, and then run again for 30 seconds.





Figures 42 and 43: The dry ice makes for a cold mixture – the thermometer bottomed out at about -55 ° F after the manure was removed from the VCM.





Figures 44 and 45: Vinegar to lower the pH and reduce ammonium nitrogen losses can be added when processing manure either from the field or frozen without using dry ice. A garden sprayer was also used to add vinegar while mixing in the cement mixer. Vinegar can also be added when processing with the Robot Coupe.



Figure 46: Step 2 — After processing with dry ice, the contents are well mixed and most particles are less than three mm. This step needs to be repeated about five times to have enough manure to fill one sample set (280 bottles) for final mixing in a cement mixer.



Figure 47 Step 3 — After processing in the Robot Coupe VCM, the manure is mixed in the large Imer cement mixer.



Figures 48 and 49: A wooden cover on the cement mixer reduces dust.





b) Filling solid manure sample bottles

Figures 50 and 51: The fastest and most homogeneous samples (based on MDA lab homogeneity results) were produced by taking the sample directly from the cement mixer and using a large enough ladle to fill the sample bottle in one pass.





The cement mixer ran for four hours before starting to take the samples. The mixer would be stopped to fill one tray (50 bottles) and then started again for about ten minutes. During this time the caps would be placed on the bottles just filled and caps would be removed from the next 50 bottles to be filled.

Figure 52: For each manure sample set, 280 bottles were filled with about 200 ml of solid manure. This required 56 liters just to fill the sample bottles. Ideally about ½ the total mixed volume is used to fill the sample bottles, and ½ the volume remains in the mixer after the bottles are filled. That means there should be about 112 liters in the cement mixer before starting to fill the sample bottles.



Figures 53 and 54: Early solid sample sets were placed in plastic bags. This was much slower than using bottles, and one of the labs expressed concerns that there may be greater nutrient losses in bags than in bottles.





Figures 55 and 56: Early attempts involved transferring the solid manure from the cement mixer to a mortar mixing tray and filling the bottles from the tray. Filling the bottles directly from the cement mixer was quicker and produced more homogeneous samples based on the MDA lab homogeneity check.





Figures 57 and 58: Multiple passes with a smaller ladle (right) did not produce significantly more homogeneous samples and were much slower than making one pass with a larger ladle (left) based on the MDA lab homogeneity checks.





c) Liquid manure preparation

Liquid manure samples were prepared by mixing water with processed solid manure. The best method was to add enough water to the mixing barrel to cover the prop and turn on the mixer. While the mixer was running, solid manure processed with water in the Robot Coupe VCM was added to the mixing barrel. The principal difference in filling sample bottles with liquid manure compared to solid manure is that the mixer runs while filling liquid manure sample bottles.

Figure 59: A Grovhac ³/₄ horsepower, variable speed prop mixer, and a plastic barrel were the primary tools for mixing liquid manure.



Figures 60 and 61: Water and baffles are added to the mixing barrel (50 gallon barrel in picture). Solid manure that had been processed with water in the Robot Coupe VCM is added to the mixing barrel while the prop mixer is running.



Figures 62 and 63: Additional water and manure processed the Robot Coupe VCM are added to the mixing barrel while the mixer runs. The 100 gallon mixing barrel is shown in these pictures.



Figure 64: Various sized and shaped ladles were used to fill the sample bottles for both liquid and solid manure.



d) Filling liquid manure sample bottles

Several methods were evaluated to transfer the liquid contents from the mixing barrel to the sample bottles. A ladle and funnel produced the most homogeneous samples with the fewest problems.

Figures 65 and 66: A ladle and funnel proved to be the most efficient method for filling sample bottles with liquid manure. Cardboard and a plastic lid reduced manure drips on the bottles.



Figures 67 and 68: A siphon tube was the fastest and cleanest method for filling the sample bottles with liquid manure. However, solids can cause the tube to clog and the clogs may act as a filter and reduce the sample homogeneity. This method was attempted several times and finally abandoned in favor of using a ladle.



Figures 69 and 70: A dip tube was tried one time and quickly abandoned. It was messy, slow, and the sample homogeneity was not as good as other methods.





Figure 71: A peristaltic pump was used one time with even worse results than the dip tube. It clogged, spluttered, and was messy. The sample homogeneity was so poor the sample set was discarded. Fortunately, the pump was borrowed and not purchased.



e) Slurry manure preparation

An acceptable method to bottle slurry manure has not yet been found. The difficulty with slurry manure is that it quickly separates into liquid and solid portions after mixing ceases. We are experimenting with mixing liquid manure with high total solid content using the Grovhac prop mixer. Slurry manure samples require bottling while the contents are being mixed. Another type of mixer may be an option, such as a large bakery mixer or a horizontal ribbon or plough mixer. However, some type of opening will likely be needed to extrude the manure samples to avoid the danger of personnel getting caught in moving machinery.

Figures 72 and 73: Attempts to process solid manure with water in the Robot Coupe VCM to produce slurry type manure were not successful. This has the appearance of good slurry manure.





Figures 74 Figure 75: However, once the mixing stops the slurry separates into solid and liquid portions within minutes. This quick separation into liquid and semi-solid components makes it impossible to fill the 280 samples bottles with homogeneous samples when the manure has this consistency. A method needs to be found to fill the bottles while the manure is being mixed to produce samples of this consistency.



A cement mixer does not work on either slurry manure or solid manure with high moisture levels.

Figures 76 and 77: If the manure is too moist, balls from pea size to golf ball size form in the cement mixer, and the moist material also clumps to the drum sides and paddles. Obviously, sample bottles cannot be filled when the manure has this consistency. For solid samples this has to be reprocessed in the Robot Coupe VCM and dried. However, manure of this consistency can be used for the solid component in liquid manure samples.





3. Labeling proficiency sample bottles

Figures 78, 79, and 80: A jig was made to hold the sample bottle while attaching the label.



Figure 81: A simple hopper made from a couple of cardboard boxes used along with the labeling jig speeds up the labeling process.



Figures 82 and 83: Each sample set consists of five large trays (50 bottles/tray) and one small tray (30 bottles) for a total of 280 bottles. Each lab received three replicates for analysis and 20 bottles were analyzed to check homogeneity. That left about 50 bottles of each manure sample for the manure sample repository.



Figures 84 and 85: Each sample set is labeled individually and covered with a plastic bag and box before starting to label the next set. This reduces the chances of placing the wrong sample in a sample bottle or placing the wrong sample in a shipping carton.



Figure 86: Using an ID number for each sample bottle also helped reduce the chance of putting the wrong sample in an improperly labeled box or shipping a wrong sample to a laboratory.

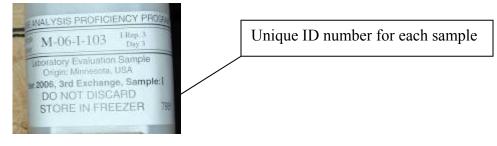


Figure 87: As each shipment was packed, the unique ID Number was recorded next to the sample name. During the study, no laboratory reported they received an incorrect sample.

| Alvey Lab Belleville IL | | | | 26006 |
|-------------------------|------------|------|------------|-------|
| M-05-G-101 7664 | M-05-H-101 | 4946 | M-05-I-101 | 5227 |
| M-05-G-102 4768 | M-05-H-102 | 5045 | M-05-I-102 | 5326 |
| M-05-G-103 4866 | M-05-H-103 | 5144 | M-05-I-103 | 5425 |

| AgSource Belm | ond Labs Be | elmond IA | | | 21006 |
|---------------|-------------|------------|------|------------|-------|
| M-05-G-101 | 4665 | M-05-H-101 | 4947 | M-05-I-101 | 5228 |
| M-05-G-102 | 4769 | M-05-H-102 | 5046 | M-05-I-102 | 5327 |
| M-05-G-103 | 4867 | M-05-H-103 | 5146 | M-05-I-103 | 5427 |

| Ray Ward | | | | | | 12 |
|----------------|-------------|------------|------|------------|-------|----|
| Ward Laborator | ies Kearney | NE | | | 15005 |] |
| M-05-G-101 | 4668 | M-05-H-101 | 4948 | M-05-I-101 | 577.9 | |
| 105-G-102 | 4770 | | -747 | M-05-I-1 | | |
| 103 | 17 | | | | | |

4. Shipping proficiency samples

All samples were shipped overnight by FedEx. There were 12 different shipments over the four years to over 60 USA labs. Only three USA labs had one of their sample sets delayed, and the delay was only one day. Two delays were caused by using an incorrect zip code. One delay was caused by the breakdown of a FedEx truck. Sample delays were more common to Canadian laboratories due to problems clearing Canadian Customs. Figures 88 and 89: Liquid samples are placed in a plastic bag. A Hobo temperature data logger is in the lower left corner of the package. The data logger was enclosed in several shipments to a few labs in the first years of the study. Packing material and a cover letter are added before closing the carton. A carton ready to ship – it will be returned to the freezer before pickup by FedEx.



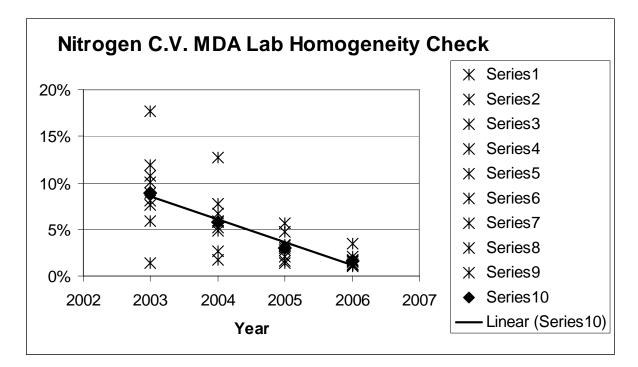


In the first MAP round shipped on July 22, 2003 the temperature data loggers included in shipments to Penn State University and Clemson University showed rapid warming during the first six hours of shipping. The transit time was about 22 hours. At the time of shipping the container temperature was -10 °F. After six hours the temperature for the Penn State container had increased 40 °F, and the Clemson temperature increased 62 °F. The Clemson container temperature was nearly 70 °F when it was opened at 9:34 AM (EDT) the following morning. The Penn State container temperature was about 60 °F when it was opened at 12:35 PM (EDT). In spite of the rapid warming, sample integrity and nutrient levels did not seem to be affected.

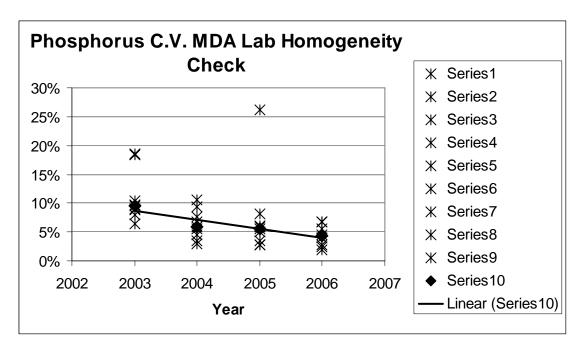
5. MDA laboratory homogeneity evaluation

Each sample set consists of 280 sample bottles. To check sample homogeneity, 20 sample bottles were randomly selected for each sample set after the sets had been bottled and placed in the freezer. These randomly selected sample bottles were analyzed for total nitrogen and total phosphorus by the Minnesota Department of Agriculture laboratory. The coefficient of variation (C.V.) was determined for each sample by dividing the standard deviation by the average and multiplying by 100. A few sets were also analyzed for moisture. However, the moisture C.V. values were so precise that moisture analysis was dropped.

The following charts have the C.V. values for results determined by the MDA laboratory. Twenty bottles were randomly selected using the unique bottle ID number by drawing numbers or by using the Microsoft Excel random number generator. Sample M-06-G (a repeating sample) was accidentally omitted. The total nitrogen and total phosphorus were determined by the laboratory and the C.V. values were determined for each of the nine samples (series 1 to 9) analyzed in a year. The linear trend line is for the median C.V. for each year.



The median C.V. for total nitrogen decreased from 8.9% in 2003 to 1.6% in 2006. Series 10 is the median; the other series (1 to 9) represent the nine samples collected each year.



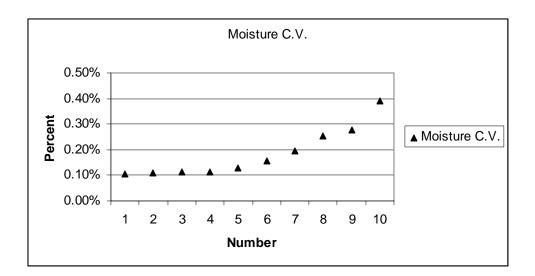
The median C.V. for total phosphorus decreased from 9.5% in 2003 to 4.3% in 2006. Series 10 is the median; the other series (1 to 9) represent the nine samples collected each year.

In 2004 and 2005 ten liquid samples were also analyzed by the MDA lab for percent moisture as a possible indicator for sample homogeneity. The range for moisture C.V. values was only 0.11 percent to 0.39 percent.

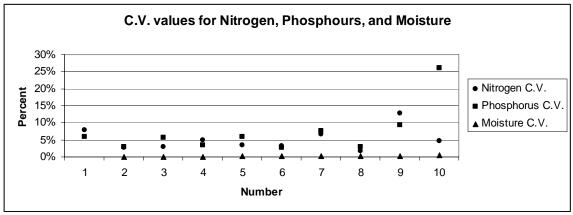
In 2004 and 2005 ten liquid samples were also analyzed for percent moisture along with total nitrogen and total phosphorus. The moisture C.V. values were so low that there was little value in determining moisture as an indication of sample homogeneity. For nitrogen or phosphorus a C.V. less than 5% is our goal. The highest C.V. we had for moisture was only 0.39%.

| Table 2: MDA Lab homogeneity check sample analysis showing very precise C.V. values for |
|---|
| moisture compared to nitrogen and phosphorus. |

| Number | SAMPLE | Nitrogen C.V. | Phosphorus C.V. | Moisture C.V. |
|--------|--------|---------------|-----------------|---------------|
| 1 | M-04-I | 7.80% | 5.91% | 0.11% |
| 2 | M-05-E | 2.73% | 2.85% | 0.11% |
| 3 | M-05-H | 3.00% | 5.62% | 0.11% |
| 4 | M-04-H | 4.84% | 3.47% | 0.11% |
| 5 | M-05-B | 3.40% | 5.93% | 0.13% |
| 6 | M-05-D | 3.19% | 2.82% | 0.15% |
| 7 | M-04-E | 6.71% | 7.60% | 0.20% |
| 8 | M-04-G | 1.76% | 3.00% | 0.25% |
| 9 | M-04-B | 12.73% | 9.34% | 0.28% |
| 10 | M-05-A | 4.72% | 26.13% | 0.39% |



The moisture C.V. is nearly negligible compared to the nitrogen and phosphorus C.V. values and is not a good predictor of sample homogeneity.



IV. Training and technical assistance for the testing industry

Tasks:⁵ Conduct seven workshops for the testing industry which will provide technical information on manure analysis procedures, analytical equipment, sample handling, reporting and quality assurance. Laboratory workshops would be coordinated with NAPT and other testing industry events.

A. Project workshops

The following workshops were either developed exclusively for the MAP Program, or significant contributions were made by MAP personnel, and their travel expenses were covered by EPA funds for the MAP Program. The audience for each of these workshops was laboratory staff from agricultural testing laboratories.

1. MAP Laboratory Analysis Workshop (April 24, 2003)

Washington State University Extension Research Center Prosser, WA: Attendance 14

Agenda not available, but this program was set up by Robert Miller specifically to meet the EPA requirements for laboratory workshops.

2. Seventeenth Soil-Plant Analyst's Workshop (February 24-25, 2004)

West Des Moines, Iowa

Two hours on February 25th were devoted to laboratory manure analysis presentations with travel expenses for Robert Miller covered by the EPA MAP funds.

February 24, 2004 - Moderator Jay Goos, North Dakota State University

| 1:00 PM | Introductory Comments – Darryl Warncke, Michigan State University |
|---------|--|
| 1:10 PM | Organic Matter vs. Organic Carbon: Methods of Analysis Maurice Watson, Ohio State University |
| 1:35 PM | Measuring C Sequestration with Routine Soil Sampling and Testing Larry Cihacek, North Dakota State University |
| 2:00 PM | National P Project: Implications for Soil and Manure Testing Peter Kleinman, Penn State |
| 2:30 PM | Relating Soil Testing to the P Index and CNMPs |

⁵ Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 4.

| | Jerry Lemunyon, NRCS, Fort Worth, Texas |
|----------------|--|
| 3:00 PM | Discussion |
| 3:10 PM | Break |
| 3:30 PM | Mehlich III Phosphorus Analysis: Colorimetric vs. ICP Antonio Mallarino, Iowa State University |
| 4:00 PM | Nutrient Uptake Requirements of Corn at Different Yield Levels Achim Dobermann, University of Nebraska, Lincoln |
| 4:25 PM | Particle Size Analysis: Pipette and Hydrometer Methods Ron Gelderman, South Dakota State University |
| 4:50 PM | Soil Testing and Fertilizer Use: What is the Relation? Paul Fixen, Phosphate and Potash Institute |
| 6:30 PM | Dinner followed by open discussion forum |
| February 25, 2 | 2004 |
| 8:00 AM | NAPT Program: What can we learn from the results? Robert Miller, Colorado State University |
| 8:30 AM | Minnesota Manure Sample Certification Program Jerry Floren, Minnesota Department of Agriculture |
| 9:00 AM | Manure Analyses: Reporting of Results John Peters, University of Wisconsin |
| 9:30 AM | NAPT Program: MAP, PAP, Accreditation Robert Miller, Colorado State University |
| 10:00 AM | Discussion |
| 10:15 AM | Break |
| 10:30 AM | Nitrogen Availability Indices |
| | Dan Walters, University of Nebraska, Lincoln |
| 11:00 AM | Soil N Test: Laboratory and Field Research Update John Sawyer, Iowa State University |
| 11:30 AM | Experience with Soil pH Buffers for Lime Recommendation Ray Ward, Ward Laboratories, Inc., Kearney, Nebraska |
| 11:50 AM | Discussion – Complete and turn in evaluation form |
| | |

3. 2004 Laboratory Analysis Workshop for Manure Testing (June 8, 2004)

Fayetteville, Arkansas Attendance: 11

Schedule:

| 7:45 – 8:15 A | M Registration |
|---------------|---|
| 8:15 AM | Welcome and Introduction |
| 8:30 AM | Quality of Laboratory Results and Proficiency Testing Robert O. Miller, Colorado State |
| 9:00 AM | The Manure Analysis Methods Manual John Peters, University of Wisconsin |
| 9:30 AM | MAP Program Sample Preparation Jerry Floren, Minnesota Department of Agriculture |
| 10:00 AM | Break |
| 10:15 AM | The Manure Analysis Proficiency Testing Program (MAP) Robert O. Miller, Colorado State |
| 10:45 AM | Laboratory Sampling of Manure Robert O. Miller, Colorado State |
| 11:15 AM | Laboratory Measurements and Uncertainty Robert O. Miller, Colorado State |
| 11:45 AM | Discussion |
| 12:00 PM | Lunch Break |
| 1:00 PM | Tour of the University of Arkansas Diagnostic Laboratory Ann Wolf, University of Arkansas |
| 2:15 PM | ICP Analysis: Matrix Modifiers and ICP operation Byron Vaughan, MDS Pharma Services, Lincoln, Nebraska |
| 3:00 PM | Break |
| 3:15 PM | Automated Soil Scooping Keith Hensley, Lignin Inc., Albuquerque, New Mexico |
| 3:45 PM | Laboratory Innovations from Around the World Robert O. Miller, Colorado State |
| 4:30 PM | Closing Remarks |
| 5:00 PM | Wrap up Session |

4. MAP Laboratory Analysis Workshop (August 24, 2004)

Minnesota Department of Transportation, St. Paul, Minnesota Department of Agriculture Attendance: 6

- 7:45 AM Registration
- 8:15 AM Welcome and Introduction

| 8:30 AM | Quality of Laboratory Results and Proficiency Testing Robert O. Miller, Colorado State |
|----------|--|
| 9:00 AM | The Manure Analysis Methods Manual John Peters, University of Wisconsin |
| 9:30 AM | MAP Program Sample Preparation Jerry Floren, Minnesota Department of Agriculture |
| 10:00 AM | Break |
| 10:15 AM | Results of the Manure Analysis Proficiency Testing Program, MAP Robert O. Miller, Colorado State |
| 10:45 AM | Laboratory Sampling of Manure Materials John Peters, University of Wisconsin |
| 11:15 AM | The Minnesota Laboratory Certification Program Jerry Floren, Minnesota Department of Agriculture |
| 11:45 AM | Questions and Answers |
| 11:55 AM | Lunch |
| 1:10 PM | An overview of soil buffer pH methods Byron Vaughan, MDS Harris Services, Lincoln, Nebraska |
| 2:00 PM | Measurement and Uncertainty Robert O. Miller, Colorado State |
| 3:00 PM | Break |
| 3:15 PM | Automated Soil Scooping for the Analytical Laboratory Keith Hensley, Lignin Inc., Albuquerque, New Mexico |
| 3:45 PM | Soil testing issues in the Upper Midwest – Open discussion Robert O. Miller, Colorado State |
| 4:00 PM | Closing Remarks |

5. MAP Laboratory Analysis Workshop (September 12 – 13, 2004)

Newark, Delaware Attendance: 22

MAP Laboratory Analysis Workshop

Newark Delaware September 12-13, 2004



Workshop Program

Sunday September 12, 2004

| 12:30 pm | Registration | 8 |
|----------|--|-----|
| 1:00 pm | Welcome and Introduction | |
| 1:10 pm | The Manure Analysis Methods Manual. John Peters, University of Wisconsin. Marshfield, WI. | 8: |
| 1:30 pm | MAP Program Sample Preparation. Jerry Floren, Minnesota Dept of Agriculture, Saint Paul, MN | 9: |
| 1:50 pm | Results of the Manure Analysis Proficiency Testing Program, MAP. Robert O. Miller, Colorado State Univ. Fort Collins, CO. | 9 |
| 2:20 pm | Laboratory Sampling of Manure Materials. John Peters University of Wisconsin. Marshfield, WI | 10 |
| 2:50 pm | Using Manure Analysis for Nutrient Recommendations. Don Horneck, Oregon State University, Hermiston, OR | 10 |
| 3:15 pm | Break | 30 |
| 3:30 pm | An Overview of Soil Buffer pH methods. Byron Vaughan, MDS Harris Laboratory, Lincoln, NE. | 1 |
| 4:00 pm | Calcium Hydroxide for Determining Lime Recommendations, David Kissel , University of Georgia, Athens, GA. | 1 |
| 4:45 pm | Discussion and Summary | |
| 5:00 pm | 1 st Day Session Break | R K |
| | | Th |

| 8:10 am | Laboratory Trouble Shooting Janice Kotuby-Amacher, Utah State University. Logan, UT |
|---------------------------|---|
| 8:40 am | Laboratory Quality - Evaluating Method Problems. Robert O. Miller, Colorado State University, Fort Collins, CO |
| 9:10 am | Analytical Method Detection Limits. Paul Vendrel University of Georgia Athens, GA |
| 9:40 am | Automated Soil Scooping. Keith Hensley, Lignin Inc. Albuquerque, NM |
| 10:10 am | Break |
| 10:20 am | Extractable Manure PO ₄ -P, Ann Wolf, Penn State University. State College, PA |
| 10:50 am | Virtual tour of Dellavalle Laboratory Nat Dellavalle, Fresno, CA. |
| 11:20 am | Implementing Laboratory Quality. Byron Vaughan, MDS Harris Laboratory, Lincoln, NE. |
| 11:45 am | Closing Remarks |
| Worksh | op Organization Committee |
| Robert Mill John Peter | and the second and second s |

This workshop is partially supported by funds from the US-EPA. Location arrangements by Karen Gartely, University of Delaware.

Evening (9/12/04) The Early Years of the Delmarva Poultry Industry

6. MAP Laboratory Analysis Workshop (March 16, 2005)

Monterey County Cooperative Extension Office Salinas, California Attendance: 15

| 8:00 AM | Registration |
|-------------|---|
| 8:30 AM | Welcome and Introduction |
| 8:35 AM | The Manure Analysis Methods Manual. John Peters, <i>University of Wisconsin</i> . Marshfield, WI. |
| 9:15 AM | Results of the Manure Analysis Proficiency Testing Program, MAP. Robert O. Miller, <i>Colorado State University</i> Fort Collins, CO. |
| 9:45 AM | Laboratory Sampling of Manure Materials John Peters University of Wisconsin. Marshfield, WI. |
| 10:15 AM | Break |
| 10:30 AM | Laboratory Trouble Shooting Janice Kotuby-Amacher, <i>Utah State University</i> . Logan, UT. |
| 11:00 AM | Variability of Soil and Plant Analyses. Donald Horneck, Oregon State University, Hermiston, OR. |
| 11:30 AM | Automated Soil Scooping. Keith Hensley, <i>Lignin Inc</i> . Albuquerque, NM. |
| 12:00 PM Li | unch Break (provided) |
| 1:00 PM | Soil pH Overview Donald Horneck, Oregon State University, Hermiston, OR. |
| 1:15 PM | An Overview of Soil Buffer pH methods. Byron Vaughan, MDS Harris Laboratory, Lincoln, NE. |
| 2:00 PM | Results of the California pH and Lime project. Robert O. Miller, <i>Colorado State Univ</i> . Fort Collins, CO. |
| 2:50 PM | Break |
| 3:10 PM | Implementing Laboratory Quality. Byron Vaughan, MDS Harris Laboratory, Lincoln, NE. |
| 3:40 PM | Results of the NAPT Performance Assessment Program. Robert Miller <i>Colorado State Univ</i> . Fort Collins, CO. |
| 4:15 PM | Closing Remarks Robert Miller John Peters, Byron Vaughan, Janice Kotuby-Amacher, Don Horneck, and Keith Hensley |

7. Nitrogen Recommendation Workshop for Laboratories (September 7, 2005)

St. Paul, Minnesota Attendance: 16

9:30 to 10:00 – Registration

10:00 to Noon – Developing a Regional Approach to N Rate Guidelines for Optimum Profitability, George Rehm, University of Minnesota

Noon to 1:00 – Lunch (on your own)

1:00 to 2:00 – Revisions to Minnesota Statute 18C.141 Soil and manure testing laboratory certification and Rule Revisions pertaining to this statute, Jerry Floren, Minnesota Department of Agriculture

8. MAP Laboratory Analysis Workshop (May 10, 2006)

Champaign, Illinois Attendance: 18

| 8:00 AM | Registration |
|----------|--|
| 8:20 AM | Welcome and Introduction |
| 8:30 AM | The Manure Analysis Methods Manual John Peters, University of Wisconsin |
| 9:00 AM | Results of the Manure Analysis Proficiency Testing Program MAP Robert O. Miller, Colorado State |
| 9:30 AM | Minnesota Department of Agriculture MAP Accreditation Jerry Floren, Minnesota Department of Agriculture |
| 10:00 AM | Break |
| 10:15 AM | Laboratory Sampling of Manure Materials John Peters, University of Wisconsin |
| 10:45 AM | MAP Questions and Answer Session |
| 11:00 AM | Introduction to pH Measurement Robert O. Miller, Colorado State |
| 11:15 AM | Modifications to SMP Buffer, the Sikora Buffer Frank Sikora, University of Kentucky |
| 12:00 PM | Lunch Break |
| 1:00 PM | Trouble Shooting Analytical Quality Robert O. Miller, Colorado State |
| 1:30 PM | Quality Control William Shakal, Sure-Tech Laboratories, Indianapolis, Indiana |

| 1:45 PM | Soil Scooping Techniques Robert O. Miller, Colorado State |
|--------------------|--|
| 2:15 PM | Automated LOI Keith Hensley, Lignin Inc., Albuquerque, New Mexico |
| 2:45 PM | Break |
| 3:00 PM | Quality and the Lab Client Don Horneck, Oregon State University |
| 3:30 PM 4:15 PM | Lab Quality, expectations of the client Closing Remarks |

B. Other professional workshops

Other workshops not developed specifically for the MAP Program, but where Robert Miller or Jerry Floren presented material related to manure analysis or the MAP Program. The primary audience at these workshops was agricultural laboratory staff or agricultural scientists and researchers.

1. Four-State Soil Testing Lab Conference

February 20-21, 2003 Dubuque, Iowa

On February 20, 2003 Jerry Floren gave a 20 minute presentation titled: *The establishment of a national manure testing proficiency program.*

On February 21, 2003 Jerry Floren participated in a four member panel discussion titled: *What is a certified lab in each State? How does your system work?*

2. American Society of Agronomy

November 2003 Denver, CO *Quality Control and Variability of Analytical Methods used for Manure and Compost Testing* Robert O. Miller, Colorado State University, Fort Collins, CO Jerry Floren, Minnesota Dept of Agriculture, St. Paul, MN Janice Kotuby-Amacher, USU-AL Director, Utah State University, Logan, UT Jan Jarman, Formerly of the Minnesota Dept of Agriculture, St. Paul, MN

3. Mid Atlantic Soil Testing Workgroup

Richmond, Virginia February 2003 Robert Miller gave a presentation on the Manure Analysis Proficiency (MAP) Program

4. Mid Atlantic Soil Testing Workgroup

Richmond, Virginia February 2004 Robert Miller gave a presentation on the Manure Analysis Proficiency (MAP) Program

5. Four-State Soil Testing Lab Conference

February 24-25, 2005 Dubuque, Iowa

On February 25, 2005 Jerry Floren gave the following, 30 minute presentation: *The national Manure Analysis Proficiency (MAP) program and certification for manure analysis* and also participated in a four member panel discussion titled: *Trends in manure analysis requirements, nutrient management planning and cost share programs.*

6. International Symposium on Soil and Plant Analysis (ISSPA)

January 31 through February 4, 2005 Cancun, Punta Cancun, Mexico Robert Miller presented the following paper: Successes of Proficiency Testing Programs for Manure and Compost Analyses in North America Robert O. Miller Soil and Crop Sciences Department Colorado State University Fort Collins, CO 80523 Phone: 970-686-5702 Fax: 970-491-0564 E-mail: rmiller@lamar.colostate.edu Janice Kotuby-Amacher USU-AL Utah State University Logan, UT 84332 E-mail: jkotuby@mendel.usu.edu Phone: 435-797-2217 Fax: 435-797-2117 Jerry Floren Minnesota Department of Agriculture APPD-4 90 West Plato Boulevard Saint Paul, MN 55107-2094 E-Mail: Jerry.Floren@state.mn.us Phone: 651-297-7082 Fax: 651) 297-2271

7. Laboratory Analysis Workshop

June 21, 2006 Guelph, ON OMAFRA Office - Attendance -- 14

Laboratory Analysis Workshop

Ontario Government Building 1 Stone Road West Guelph, Ontario

| Tuesda | y June 27, 2006 | | |
|----------|--|---------|--|
| 8:00 am | Registration | 1:00 pm | Analytical Quality and Uncertainty Robert O. Miller, Colorado State |
| 3:20 am | Welcome and Introduction | | Univ. Fort Collins, CO. |
| 8:30 am | Ontario Lab Accreditation. Keith Reid Ontario Ministry of Agriculture. | 2:00 pm | Implementing Quality Control in a soil testing lab - Byron Vaughan, MDS Harris Labs. Lincoln, NE. |
|):00 am | Introduction to pH Measurement. Robert O. Miller, Colorado State Univ. Fort Collins, CO. | 2:45 pm | Soil Scooping Techniques. Robert O. Miller, Colorado State Univ. Fort Collins, CO. |
| 9:30 am | Modifications to the SMP Buffer, the Sikora Buffer. Frank Sikora, University of Kentucky. | 3:00 pm | Break |
| 10:15 am | Break. | 3:15 pm | Automated LOI. Keith Hensley, Lignin Inc. Albuquerque, NM. |
| 10:30 am | Results of the Manure Analysis Proficiency Testing Program, MAP. Robert O. Miller, Colorado State Univ. Fort Collins, CO. | 3:45 pm | Analysis of Compost and Manure Samples: Inorganic and Organic N. Robert O. Miller, Colorado State Univ. Fort Collins, CO. |
| 11:00 am | MAP Question and Answer Session | 4:15 pm | Needs of the soil testing industry |
| 11:15 am | On-Line Matrix Modification for AAS and ICP Analysis. Byron Vaughan, MDS Harris Labs. Lincoln, NE. | 4:30 pm | Closing Remarks. Keith Reid Ontario Ministry of Agriculture. |
| | | Works | hop Organization Committee |
| 12:00 pm | Lunch Break (provided) | | t fee \$140/per person. Discount price ployee in attendance \$100. |

8. Laboratory Analysis Workshop

Washington State University Extension Research Center November 17, 2006 Prosser, WA -Attendance 12

Laboratory Analysis Workshop- PNW

WSU Ag Research and Extn Center

24106 N. Bunn Road

Prosser, Washington

Workshop Program

Friday November 17, 2006

8:00 am Registration

8:10 am Welcome and Introduction. Robert Stevens. WSU Prosser, WA

8:20 am Laboratory Proficiency - Soil, Manure and Compost **Robert O. Miller**, *Colorado State Univ*. Fort Collins, CO.

9:10 am Quality Assurance in an Agricultural Laboratory. **Steve McGeehan**, *University of Idaho, Moscow.*

9:40 am Automated Soil Organic Matter - LOI. **Keith Hensley**, *Lignin Inc*. Albuquerque, NM. 10:15 am Break

10:30 am Soil pH Measurement, Form and Function. **Robert O. Miller**, *Colorado State Univ.* Fort Collins, CO.

11:00 am Current ICP Optical Emission Capabilities **CurtisUrben**, *PerkinElmer*, WA. 12:00 pm Lunch Break (provided)

1:10 pm Soil and Plant Änalysis Variability. **Don Horneck**, Oregon State University, Hermiston, OR.

1:50 pm Future Directions in Soil Fertility and Soil Analysis. - **Robert O. Miller**, *Colorado State Univ.* Fort Collins, CO.

2:45 pm Break

3:00 pm Laboratory Client Needs and Expectations. **Jason Ellsworth**, *Wilbur-Ellis*. Pasco, WA.

3:40 pm Laboratory Client Relations, a Laboratory perspective. Lab Aron Quist, *Stanworth Consulting*. Blythe, CA.

4:15 pm Needs of the Lab Testing Industry. **Robert O. Miller**, *Colorado State Univ*. Fort Collins, CO.

4:30 pm Closing Remarks

Enrollment fee \$125/per person. Discount price for 2nd Employee in attendance \$90. Contact Robert Miller to enroll: rmiller@lamar.colostate.edu.

V. Quantify long-term improvements in manure analysis testing

Tasks: 6

• Determine the feasibility of long-term sample preservation. Pending findings, build a "library" of manure reference samples for future proficiency testing purposes;

• Develop and implement a plan to evaluate laboratory competency by the use of "double blind" sample exchanges. "Double blind" samples are samples previously used in proficiency testing but are shipped from a private partner in such a manner that the labs are unaware that the samples originated from the NAPT program. This will allow the program to examine bias characteristics under typical laboratory conditions;

Compare characteristics of analysis variability of labs currently active in the MN Certification program to non-certified laboratories; and
Compare characteristics of analysis variability of participating labs over time.

A. Manure reference repository (library)

Extra sample containers were filled for each of the 36 different manure sample sets prepared from 2003 through 2006, and these extra sample containers were collected to use as a reference repository. These samples have been stored in four chest freezers and one upright freezer at approximately 0 $^{\circ}$ F since they were initially collected.

Some of the extra samples have already been sent to MAP labs that are interested in trying new methods, need extra rerun sets for certification, or need reference samples. These samples could also be used by scientists and chemists developing new methods for manure analysis. There is no other source of manure that has been so well characterized by a variety of different labs.

B. Double blind study

The normal samples in this proficiency program are considered "single blind" samples. Laboratories know the sample origin is the proficiency program; however, they do not know the nutrient content of the samples. As part of this study, we were also interested if there would be a difference in results obtained from our standard, single blind, proficiency samples compared to samples a laboratory received from a client. These client samples are called "double blind" samples as the laboratories do not know the origin of the samples or the nutrient content. We wanted to learn if laboratories analyze normal production samples with the same rigor they use for proficiency samples.

⁶ Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 4.

The purpose of a double blind (DB) study is to determine if there is a difference in results obtained from a laboratory when they know the samples are from a proficiency program (single blind) compared to samples that appear to have been submitted from a client (double blind).

The DB samples were packed in 500 ml wide mouth bottles that producers commonly use to send manure samples to labs. These double blind sample bottles looked quite different from the normal proficiency sample bottles (250 ml narrow mouth bottles).

A cooperator was hired to pose as a client for the DB study. The DB cooperator allowed us to do the following:

- Use their telephone to contact labs to determine tests and prices
- Ship samples from their location
- Use their letterhead to prepare letters signed by their nutrient management specialist to accompany the samples
- Use their electronic logo to place on labels for the sample containers

In addition to assisting us with the double blind mechanics, the cooperator also completed the following tasks:

- Allowed their nutrient management specialist to answer questions from labs by phone or E-mail
- Collected the results from the laboratories
- Paid the laboratory invoices for the analysis fees and submitted an invoice to us for reimbursement

This study changed slightly from our original proposal. We proposed to conduct a double blind study near the project beginning and another double blind study near the project end to evaluate if there had been a change in laboratory performance from the beginning to end of the project. As the project developed, we had the following concerns with this proposal:

- It would be best to compare laboratory performance using identical manure samples; however, we could not be sure if the manure samples would degrade over time.
- Uncertainties if the same labs participate at both the beginning and end of the study

The double blind design was changed as follows:

- One DB study was conducted at the end of this project.
- Thirty-eight laboratories were selected at random for the double blind study.

- The double blind samples were duplicates of Sample M-06-E which had been sent out to the labs several months before the double blind samples were shipped.
- Each double blind lab received two identical samples; however, the labs did not know they were identical. This allowed us to analyze both accuracy and precision and compare it with accuracy and precision results obtained with the M-06-E exchange.

1. Comparison of the single blind to the double blind

The accuracy results for the DB samples on most of the tests were very close to the single blind (SB) results labs had for the same sample (M-06-E) analyzed a few months earlier.

The 2006 MAP program evaluated laboratory proficiency labs utilizing DB samples. Duplicates samples of MAP sample M-2006-E were shipped to 38 randomly selected labs of the 69 labs enrolled in the 2006 MAP Program. A comparison of SB and DB results indicate generally very good agreement in the median values across analyses for the two methods of submission. MAD values, however, were generally substantially higher for the DB evaluation. For moisture content, phosphorus, and zinc the MAD values were more than 2 times higher for the DB results over that of SB evaluation. For NH4-N, potassium, and copper the MAD values were comparable for both the SB and DB samples.

| Analysis | Single I | Blind | Double Blind | | |
|----------------------|----------|-------|--------------|-------|--|
| Analysis | Median | MAD | Median | MAD | |
| Total Solids (%) | 11.5 | 0.450 | 11.6 | 0.650 | |
| Moisture Content (%) | 88.6 | 0.480 | 88.4 | 0.800 | |
| NH4-N Total (mg/kg) | 1830 | 85.0 | 1800 | 74.0 | |
| Total Nitrogen (%) | 0.530 | 0.017 | 0.519 | 0.029 | |
| Total P (%) | 0.219 | 0.011 | 0.214 | 0.027 | |
| Total K (%) | 0.262 | 0.013 | 0.258 | 0.019 | |
| Total S (%) | 0.08 | 0.006 | 0.07 | 0.053 | |
| Total Zn (mg/kg) | 45.7 | 2.60 | 43.50 | 4.30 | |
| Total Cu (mg/kg) | 39.0 | 2.50 | 39.0 | 2.20 | |

 Table 3: The medians for most SB and DB results compared well. Generally the MAD values were larger for the DB results than for the SB results.

R*d* values compare the precision of lab results using the triple replicates in the SB exchange and double replicates in the DB exchange. R*d* is the coefficient of variation (C.V.) from individual labs replicates compared the overall C.V. calculated from all lab results. Comparison of intra-lab precision R*d*, indicate for total solids results were 1.41% and 3.0% for the single and double blind evaluation, respectively. R*d* values for nitrogen were 1.7% and 3.0%, phosphorus were 2.7% and 3.5%, potassium of 2.3% and 2.4%, and zinc 2.9% and 6.4% respectively for the single and double blind evaluations. It is worth noting that these differences may not be significant as the single blind samples were submitted in triplicate and double blind samples were submitted in duplicate.

A comparison of lab SB and DB data indicates near equivalent median values for the two methods of evaluation, but much greater confidence limits (CL) as noted by \pm value, for the DB method (see Table 4). For total solids the percent of labs within CL, the SB evaluation was nearly the same of DB, despite an increase in the confidence window from 9.7% to 14% of the median. Nitrogen also showed an equal percent of labs within CL, yet an increase in the confidence window of 8% to 14% of the median for the DB evaluation. The percent of within CL for phosphorus decreased, yet the confidence window increased from 12.5% of the median to 31.5% for the DB method. Lastly potassium indicated a drop of 16% of labs within CL, even though the confidence window increased from 12.4% of the median to 18.4% for the DB method.

These results indicate that DB evaluations have inherent greater inter-lab variations, especially for phosphorus and may result in a lower of percent of labs within confidence limits with DB samples compared to SB samples.

 Table 4: MAP 2006 comparison of single and double blind lab results showing Confidence Limits and percent labs passing (within CL).

| ANALYSIS | Singl | e Blind | Double Blind | | |
|------------------|------------|-------------------|--------------|-------------|--|
| ANALISIS | Value | e % within CL Val | | % within CL | |
| Total Solids (%) | 11.5 ±1.12 | 77.0% | 11.6 ±1.62 | 76.0% | |
| Nitrogen (%) | 0.53 ±0.04 | 72.0% | 0.52 ±0.07 | 74.2% | |
| Phosphorus (%) | 0.2 ±0.03 | 80.6% | 0.21 ±0.07 | 76.4% | |
| Potassium (%) | 0.26 ±0.03 | 85.0% | 0.26 ±0.05 | 69.0% | |

Results of the DB study indicate near equivalent median values for SB or DB evaluation methods, with larger MAD values for the DB method. For total solids the percent of labs within the confidence limit (CL) (based on 2.5 X MAD), the single blind evaluation was nearly the same of double blind, despite an increase in the confidence window from 9.7% to 14% of the median. Nitrogen also showed an equal percent of labs within CL for both evaluation methods, with an increase in the confidence window of 8% to 14% of the median for the double blind evaluation. The percent of labs within CL for phosphorus decreased, yet the double blind confidence window increased from 12.5% of the median to 31.5%. Clearly there is a significant concern of lab quality with CL of $\pm 31\%$ for phosphorus based on the double blind evaluation, even though the confidence window increased from 12.4% of the median to 18.4% for the double blind method. These results indicate that double blind evaluations have inherent greater inter-lab variations, especially for phosphorus and may also result in a lower of percent of labs within confidence limits.

2. Laboratory reports to their clients

This is the first time we have seen the actual reports the laboratories send to their clients, and some work with the labs is necessary to improve their customer reports. The following letter was mailed on December 19, 2006 to all MAP laboratories with our

observations on the client reports submitted by the 38 randomly selected laboratories in the DB study.

«Laboratory» «Title» «Full_Name» «Address_1» «Address_2» «PO_Box» «City», «State» «Zip Code»

December 19, 2006

Dear «Title» «Last_Name»:

This fall the Manure Analysis Proficiency (MAP) Program submitted double blind manure samples to 38 randomly selected laboratories that participated in the 2006 MAP Program. All 38 laboratories submitted results for the double blind study, and one laboratory submitted a corrected report.

These double blind samples consisted of two replicate samples that represented paired duplicates of sample M-06-E submitted to laboratories in the 2nd MAP round in 2006; however, the laboratories did not know that these samples originated from the MAP Program. There were two objectives to this double blind study.

- 1. How well did the laboratory results compare between the double blind study and sample M-06-E? (This data is still being analyzed.)
- 2. Could laboratories improve the reports they send to their clients? (Some general observations about laboratory client reports based on the reports we received from the 38 laboratories are given below.)

Positive observations about the reports

- Six labs had a contact person's name and/or a signature on the report.
- Two labs specified the method used for each analysis.
- One laboratory specified the detection limit for each analysis.
- Two laboratories specified using a density other than 8.33 pounds/gallon. If you determine the density to be different than 8.33 pounds/gallon, and you use the calculated density to calculate your results, the density should be specified on the report.

Observations to improve laboratory client reports

• One lab did not identify on their report forms if the results were for Pit 1 or Pit 2, and this made it impossible to match the results to the pit (except that the double blind samples were replicates).

- The cover letter specified the samples were "liquid manure." Two labs identified the samples as slurry manure, one as dry manure, and one as compost.
- The cover letter accompanying the double blind samples did not specify a livestock species. However, three labs identified the livestock as swine, one as dairy, and one as beef.
- Some labs use fonts as small as 8 points. Many farmers are over 50 with vision issues, and they may be looking at a fax or photocopy of your report. Use at least 12 point font size for your client reports.
- Phosphorus and Phosphate Potassium and Potash; a number of labs reported "Phosphorus as P_2O_5 " and "Potassium as K_2O ."
 - Phosphorus is P and Phosphate is P_2O_5
 - o Potassium is K and Potash is K₂O
- One laboratory reported P_2O_5 as Phosphorus Pentoxide and K_2O as Potassium Oxide. Your clients are much more familiar with phosphate and potash.
- One laboratory had Phosphorus as PO₂ instead of P₂O₅ and Potassium as KO₂ instead of K₂O.
- The labs used a variety of reporting units, and most used more than one unit. Since this was liquid manure, pounds per 1,000 gallons was the most appropriate and farmer friendly unit followed by pounds per acre inch. Pounds per ton were not appropriate. Twenty-six labs used pounds per 1,000 gallons; one lab used pounds per acre inch; six laboratories used pounds per ton without using either pounds per 1,000 gallons or pounds per acre inch.
- E.C. units are frequently used improperly. The correct units are either dS/m or mmhos/cm. Note: "mho" is ohm spelled backwards and a dimension (meter or centimeter) is also required. Many labs either misspelled "mmhos", or failed to include a dimension. Some examples are shown in the following table:

| Incorrect units used for E.C. | | | | | | |
|-------------------------------|--|--|--|--|--|--|
| mmhhos | | | | | | |
| mmhos (SS) | | | | | | |
| mmho / cm | | | | | | |
| mmohs/cm | | | | | | |
| μS | | | | | | |

- One laboratory had significant errors converting ppm to lbs/1,000 gallons.
- Does your laboratory use significant figures appropriately? Some examples of inappropriate use of significant figures were the following: 100,729.2 ppm soluble salts dry basis, 17,930.8 lbs/ac in of organic matter; lbs/1,000 gallons carried out to two or more decimal points.

- One lab gave the wrong formula for converting percent to pounds per 1,000 gallons: "as-is % X 0.834 = lbs. of element per 1,000 gallons." The correct factor to convert percent to "pounds per 1,000 gallons" is 83.3 not 0.834.
- One laboratory reported moisture as ppm instead of percent and then calculated the weight of moisture in 1,000 gallons of manure as 0.615 pounds.
- One laboratory transposed moisture and total solids. This is one of the more common errors in the MAP Program reports, but it was not expected to be a problem in the laboratory's customer reports.

Please review your client reports, and contact me if you have any questions.

On December 18, 2006 an E-mail was sent to all MAP labs with the subject, "Can you help the MAP Program?" The following is the message content:

We are in the process of writing our final report for this project to the program's sponsor, the Environmental Protection Agency. If you have thoughts on how the MAP program has assisted your laboratory, or suggestions for improvement, we would like to include your observations in our report. Please reply to this E-mail if you can give us any feedback on the MAP program.

We would really appreciate it if you could respond to this request.

Yours truly,

Jerry Floren

Phone: (651) 201-6642 Fax: (651) 201-6117 E-mail: jerry.floren@state.mn.us

3. Analyses routinely performed by laboratories

The following table shows the number and type of analyses performed by the 38 laboratories in the DB study. These are services provided to normal customers. Analyses available in the 2006 MAP Program are shaded. Organic nitrogen, calcium, magnesium, pH, and sodium are not currently in the MAP Program, but these tests were run by more labs than the following MAP tests: zinc, copper, and electrical conductivity. The MAP test Water Extractable Phosphorus (WEP) is experimental and labs were not expected to run it as part of their routine tests.

Table 5: Analyses in the yellow cells are currently in the MAP Program. The DB study demonstrated that a number of analyses not currently in the MAP Program are provided by some labs to their customers.

| Laboratories submit | ting results for the doubl | e blind study |
|-------------------------------|----------------------------|-----------------|
| Test | Laboratories | Percent of Labs |
| Total Nitrogen | 38 | 100.0% |
| Total Phosphorus or Phosphate | 38 | 100.0% |
| Total Potassium or Potash | 38 | 100.0% |
| Total Solids | 25 | 65.8% |
| Ammonium Nitrogen | 24 | 63.2% |
| Moisture | 23 | 60.5% |
| Sulfur | 11 | 28.9% |
| Organic Nitrogen | 10 | 26.3% |
| Calcium | 9 | 23.7% |
| Magnesium | 9 | 23.7% |
| pH | 9 | 23.7% |
| Sodium | 9 | 23.7% |
| Zinc | 8 | 21.1% |
| Copper | 7 | 18.4% |
| Iron | 7 | 18.4% |
| Manganese | 7 | 18.4% |
| Electrical Conductivity | 6 | 15.8% |
| Chloride | 4 | 10.5% |
| Soluble Salts | 4 | 10.5% |
| Boron | 3 | 7.9% |
| Carbon to Nitrogen Ratio | 3 | 7.9% |
| Organic Matter | 3 | 7.9% |
| Aluminum | 2 | 5.3% |
| Ash | 2 | 5.3% |
| Total Organic Carbon | 2 | 5.3% |
| Density | 1 | 2.6% |
| Lead | 1 | 2.6% |
| Molybdenum | 1 | 2.6% |
| Nickel | 1 | 2.6% |
| Salts | 1 | 2.6% |
| Sodium Absorption Ratio | 1 | 2.6% |
| Water Extractable Phosphorus | 0 | 0.0% |

C. Analysis variability of participating labs over time

Section *IX Evaluate program impacts (B.)* Assessment of analytical methods beginning on page 80 has a detailed discussion of analytical trends during the four years of this program.

D. Certified labs vs. non-certified labs

Since 1996 the Minnesota Department of Agriculture has certified laboratories for manure analysis. The following table compares the MAP performance of laboratories certified for manure analysis in 2006 with non-certified laboratories. The comparison is for the first round of 2006 (samples A, B, and C) and compares the following analyses:

- total solids
- total Kjeldahl nitrogen
- nitrogen by combustion
- total phosphorus
- total potassium

| Lab Type | Analysis and Sample | Number | A flags | P flags | Accuracy | Precision |
|---------------|--------------------------|--------|---------|---------|----------|-----------|
| Certified | Total Solids (A, B, & C) | 117 | 15 | 12 | 87.2% | 89.7% |
| Non-certified | Total Solids (A, B, & C) | 79 | 14 | 14 | 82.3% | 82.3% |
| Certified | TKN (A, B, & C) | 91 | 21 | 5 | 76.9% | 94.5% |
| Non-certified | TKN (A, B, & C) | 57 | 13 | 7 | 77.2% | 87.7% |
| Certified | N-Combustion (A, B, & C) | 59 | 9 | 1 | 84.7% | 98.3% |
| Non-certified | N-Combustion (A, B, & C) | 33 | 6 | 8 | 81.8% | 75.8% |
| Certified | Phosphorus (A, B, & C) | 120 | 10 | 4 | 91.7% | 96.7% |
| Non-Certified | Phosphorus (A, B, & C) | 82 | 19 | 14 | 76.8% | 82.9% |
| Certified | Potassium (A, B, & C) | 120 | 11 | 5 | 90.8% | 95.8% |
| Non-certified | Potassium (A, B, & C) | 82 | 13 | 11 | 84.1% | 86.6% |

Table 6: Comparison of results submitted by certified labs compared to results submitted from noncertified labs. These results were from the first exchange in 2006 for samples A, B, and C.

The column "Accuracy" gives the percentage of labs that were within the control limit of ± 2.5 MAD units from the median for accuracy. The column "Precision" gives the percent of labs with the coefficient of variation (C.V.) for their three replicates for each analysis lower than three times the overall C.V. Labs with results outside the control limit for accuracy are flagged with a "*L" (low) or a "*H" (high). Labs are flagged "*P" for poor precision. The percent of labs listed in the "Accuracy" and "Precision" columns is the percent of labs that were not flagged for accuracy or precision respectfully. Another way to think of this is the percent listed for accuracy or precision is the percent of labs that were not flagged with a "*L", "*H", or "*P" in the MAP reports for each exchange.

The bold numbers in these two columns are the labs that had the higher percentage of labs that were not flagged for accuracy or precision. The only analysis where the non-certified labs had a larger percentage was for TKN where the non-certified labs had 77.2% of passing labs compared to 76.9% of passing labs that were certified. The largest difference between certified and non-certified labs was for phosphorus.

Not all laboratories elect to become certified, and this may have been a better comparison if labs eligible for certification in 2006 (based on MAP performance in 2005) were compared with the labs that were not eligible. Certification requires the following:

• Have acceptable performance in the manure proficiency program.

- Complete an application for manure testing certification.
- Pay the fee for certification (\$100 if certified in the previous year, or \$200 in not certified in the previous year.

VI. Program sustainability

Tasks:⁷ The Advisory Board will develop a plan to insure the success of a long-term program that is functional after this funding period is completed. After equipment purchases, program establishment, and the number of participating labs stabilize, it will be possible to forecast future operating costs. The Board will evaluate various options such as adjusting annual participation fees, modifying services, and the need for external operating funds. The Board will then develop an appropriate strategy for securing funds.

With four years program experience it has become apparent the MAP Program, unlike soil proficiency testing programs, cannot be supported solely by fees paid by laboratories to participate in the program. There are several reasons why laboratory fees alone cannot support the MAP Program:

- The MAP Program is much more expensive to operate than a soil proficiency testing program. Manure samples require frozen storage and extra labor is required to prepare them. They also require more expensive, overnight shipping.
- Laboratories do not receive as much income for manure testing as they do for soil testing. For the 2007 growing season laboratories may analyze ten million soil samples, but only analyze about 30,000⁸.
- There are at least eight soil testing laboratories that will run over 200,000 soil samples for the 2007 growing season. Few, if any, labs will analyze more than 5,000 manure samples for 2007.
- Eighty-four laboratories signed up for the MAP Program in 2003, but only 65 submitted results. Sixty-nine laboratories signed up for the MAP Program in 2006, and 68 labs submitted results. It is unlikely there are many more labs than these that analyze significant numbers of manure samples. There are about 210 labs that analyze soil in North America.
- Each year the MAP fees increased by \$100 starting with \$100 in 2003 and ending with \$400 in 2006. Our Advisory Board felt that \$550 to \$600 would be the maximum we could charge without losing a significant numbers of labs. Sixty labs at \$600 per lab would generate \$36,000. An additional \$4,000 to \$5,000 could be generated by certifying labs for manure analysis. We feel this is near the top end of revenue that can be generated by direct fees collected from participating labs.

⁷ Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 5.

⁸ The numbers comparing soil and manure samples analyzed for the 2007 growing season are Dr. Robert Miller's best estimates based on conversations with laboratories in the United States and Canada.

A. Projected 2007 revenue and expenses for MAP and certification

The tables on the following pages give our best estimates for the projected costs and revenues to run the MAP Program and Manure Testing Certification Program in 2007. The following assumptions were made for 2007:

- The MAP enrollment fee would remain at \$400 for 2007
- The number of laboratories participating in 2007 would be the same as in 2006
- There would be no analysis of samples by the MDA lab to verify homogeneity (saving \$5,040)
- Any lab workshops have to meet all expenses by collecting fees
- There would be no major equipment purchases
- The tables break out fixed costs (independent of the number of participating laboratories) and variable costs (dependent on the number of participating laboratories)
- There would be three exchanges with three different manures in triplicate for each exchange (no change from previous years)

Unfortunately, there simply are not enough laboratories and not enough manure samples run to make this program self supporting solely from laboratory fees. In 2007, the Minnesota Department of Agriculture (MDA) tentatively plans to subsidize the program at a cost of nearly \$50,000. However, outside funding is needed if the program is to continue beyond 2007.

The advisory board thought the MAP fee could be increased to \$550 to \$600 without losing too many laboratories. However, even if the fee is raised to \$600 and 60 laboratories remain in the program only \$36,000 will be raised. Most of our costs are fixed. There are only minor savings in preparing samples for 40 laboratories compared to 70 laboratories.

We also looked at the extra expenses to provide this program to Canadian laboratories. Canadian labs require additional labor for sample preparation and shipping. Helping sample shipments clear Canadian Customs has also required additional labor. One Canadian lab has required significantly more help than other labs in the program. The Canadian labs pay for the sample shipping and also for an annual Export Permit, so they have additional fees not incurred by USA labs. There are extra costs associated with providing the MAP Program services to Canadian labs, but providing these services to the Canadian labs generates more income than dropping them from the program.

| REVENUE FROM USA AND CANADIAN LABS | 2006 | 2007 | 2006 Fee | 2007 Fee | Total 2006 | Total 2007 | Change |
|---|--|------|----------|----------|-------------|-------------|--------|
| USA Laboratories in MAP: | 61 | 61 | \$400.00 | \$400.00 | \$24,400.00 | \$24,400.00 | \$0.00 |
| Canadian Laboratories in MAP: | 8 | 8 | \$400.00 | \$400.00 | \$3,200.00 | \$3,200.00 | \$0.00 |
| TOTAL LABORATORIES IN MAP: | 69 | 69 | \$400.00 | \$400.00 | \$27,600.00 | \$27,600.00 | \$0.00 |
| USA Certified Manure Testing Labs: | 40 | 40 | \$100.00 | \$100.00 | \$4,000.00 | \$4,000.00 | \$0.00 |
| Canadian Certified Manure Testing Labs: | 5 | 5 | \$100.00 | \$100.00 | \$500.00 | \$500.00 | \$0.00 |
| TOTAL CERTIFIED MANURE TESTING LABS: | 45 | 45 | \$100.00 | \$100.00 | \$4,500.00 | \$4,500.00 | \$0.00 |
| TOTAL REVEN | TOTAL REVENUE FOR MAP AND CERTIFICATION: \$32,100.00 \$32,100.00 | | | | | | |

Table 7: Revenue from labs for the MAP Program and manure testing certification in 2006 compared with projected revenue in 2007 if there is no change in the fees charged to labs. If fees are increased for 2007, the number of labs participating in MAP is expected to decline.

| FIXED PROGRAM COSTS (independent on number of labs) | Cost/Unit | Factor 2006 | Factor 2007 | Current 2006 | Proposed 2007 | Change |
|--|-------------|----------------|----------------|--------------|------------------|-------------|
| | | | | | | - |
| J. Floren Hourly Labor plus Benefits | \$37.13 | 1,253 | 835 | \$46,513.96 | \$31,001.88 | \$15,512.08 |
| Annual Staples rent | \$5,400.00 | 1 | 0.5 | \$5,400.00 | \$2,700.00 | -\$2,700.00 |
| Annual MDA Office Space Rent for J. Floren* | \$12,000.00 | 72.5% | 48.3% | \$8,700.00 | \$5,798.61 | -\$2,901.39 |
| Annual cost for Robot Coupe replacement knives | \$600.00 | 1 | 1 | \$600.00 | \$600.00 | \$0.00 |
| Annual equipment repair fund | \$600.00 | 0 | 1 | \$0.00 | \$600.00 | \$600.00 |
| Workshop expenses over lab fees to attend workshop | \$2,000.00 | 1 | 0 | \$2,000.00 | \$0.00 | -\$2,000.00 |
| Fee for Dr. Robert Miller | \$24,875.00 | 1 | 86.43% | \$24,875.00 | \$21,500.00 | -\$3,375.00 |
| Travel Expenses for Shipping Samples | \$1,116.00 | 1 | 86.7% | \$1,116.00 | \$967.50 | -\$148.50 |
| Travel Expenses for Preparing Samples | \$1,464.00 | 1 | 74.6% | \$1,464.00 | \$1,092.00 | -\$372.00 |
| Cost for MDA Lab to Analyze Samples for N and P | \$28.00 | 170 | 0 | \$4,760.00 | \$0.00 | -\$4,760.00 |
| | | | | | | - |
| Total Direct Fixed Costs: | | | | \$95,428.96 | \$64,259.99 | \$31,168.97 |
| Total Indirect Fixed Costs: | | 16.0% | 16.0% | \$15,268.63 | \$10,281.60 | -\$4,987.03 |
| | | | | | | - |
| TOTAL FIXED COSTS: | | | | \$110,697.59 | \$74,541.59 | \$36,156.00 |
| *Office space includes adjustment for vacation & holidays. | | | | | | |

Table 8: The majority of the program expenses are fixed. These costs remain the same regardless of whether there are 20 or 70 laboratories participating in the programs. The savings in 2007 are primarily labor as we become more efficient at sample preparation and fewer reports need to be prepared.

Table 9: A few costs are reduced if fewer labs participate. These variable costs include reduced postage and shipping costs and fewer sample bottles and shipping boxes needed.

| VARIABLE PROGRAM COSTS (dependent on number of labs) | Cost/Unit | Factor 2006 | Factor 2007 | Current 2006 | Proposed 2007 | Change |
|--|-----------|----------------|----------------|-----------------|------------------|-----------|
| FedEx for Shipping Three MAP Exchanges (USA Only) | \$18.31 | 183 | 183 | \$3,350.73 | \$3,350.73 | \$0.00 |
| Postage for USA Labs | \$3.73 | 61 | 61 | \$227.53 | \$227.53 | \$0.00 |
| Postage for Canadian Labs | \$5.41 | 8 | 8 | \$43.28 | \$43.28 | \$0.00 |
| Sample Bottles | \$0.71 | 2520 | 2340 | \$1,786.91 | \$1,659.27 | -\$127.64 |
| Sample Boxes | \$0.35 | 207 | 207 | \$72.45 | \$72.45 | \$0.00 |
| Total Direct Variable Costs: | | | | \$5,480.90 | \$5,353.26 | -\$127.64 |
| Total Indirect Variable Costs: | | 16.0% | 16.0% | \$876.94 | \$856.52 | -\$20.42 |
| | TOTAL | VARIABLE | COSTS: | \$6,357.84 | \$6,209.78 | -\$148.06 |

Table 10: Comparison of the fixed and variable costs from the above tables.

| TOTAL OF FIXED AND VARIABLE COSTS | Current 2006 | Proposed 2007 | Change |
|-----------------------------------|--------------|------------------|-------------|
| | | | - |
| Total Fixed Costs | \$110,697.59 | \$74,541.59 | \$36,156.00 |
| Total Variable Costs | \$6,357.84 | \$6,209.78 | -\$148.06 |
| | | | - |
| TOTAL FIXED AND VARIABLE COSTS: | \$117,055.43 | \$80,751.38 | \$36,304.06 |

| COST OF COMBINED PROGRAMS (MAP and Manure Laboratory Testing Certification) | Current 2006 | Proposed 2007 | Change |
|---|---------------------------|---------------------------|--------------------------|
| | | | - |
| Total fixed and variable costs: | \$117,055.43 | \$80,751.38 | \$36,304.06 |
| Income from laboratory fees: | \$32,100.00 | \$32,100.00 | \$0.00 |
| PROGRAM SHORTFALL REVENUE MINUS COSTS: | <mark>-\$84,955.43</mark> | <mark>-\$48,651.38</mark> | <mark>\$36,304.06</mark> |
| Costs above fees for each laboratory participating in the programs: | -\$1,231.24 | -\$705.09 | \$526.15 |

Table 11: Combining the costs and revenues shows there is an estimated shortfall of \$48,651.38 for the 2007 program.

It is more expensive to service Canadian labs in the MAP Program. The increased costs include increased labor for the following: record keeping, sample preparation, issues with Canadian Customs, and requests for special assistance. The following three tables estimate the increased costs for the Canadian laboratories.

| DIFFERENCES IN VARIABLE EXPENSES BETWEEN USA LABS AND CANADIAN LABS | | | | | | |
|---|-------|-------------|--------------|--------------|---------------|--------|
| Variable Expenses vs. Revenue Applicable to USA Labs | Hours | Hours/lab | Unit Cost | Current 2006 | Proposed 2007 | Change |
| MAP Revenue | | | | \$24,400.00 | \$24,400.00 | \$0.00 |
| Manure Lab Certification Revenue | | | | \$4,000.00 | \$4,000.00 | \$0.00 |
| Total USA Revenue from MAP and Manure Certification | | | | \$28,400.00 | \$28,400.00 | \$0.00 |
| Prepare Shipments | 123 | 2.02 | \$37.13 | \$5,297.42 | \$5,297.42 | \$0.00 |
| Problem Labs | 40 | 0.66 | \$37.13 | \$1,722.74 | \$1,722.74 | \$0.00 |
| FedEx Shipping | | | \$18.31 | \$3,886.85 | \$3,886.85 | \$0.00 |
| Postage | | | \$3.73 | \$263.93 | \$263.93 | \$0.00 |
| Total USA Labs Revenue minus Variable Expenses: | | \$17,229.06 | \$17,229.06 | \$0.00 | | |
| Per Lab USA Revenue minus Variable Expenses: | | \$282.44 | \$282.44 | \$0.00 | | |

Table 12: Variable expenses vs. revenue for USA labs.

Table 13: Variable expenses vs. revenue for Canadian labs.

| Variable Expenses vs. Revenue Applicable to Canadian Labs | Hours | Hours/lab | Unit Cost | Current 2006 | Proposed 2007 | Change |
|---|-------|-----------|--------------|-----------------|------------------|--------|
| MAP Revenue | | | | \$3,200.00 | \$3,200.00 | \$0.00 |
| Manure Lab Certification Revenue | | | | \$500.00 | \$500.00 | \$0.00 |
| Total Canadian Revenue from MAP and Manure Certification | | | | \$3,700.00 | \$3,700.00 | \$0.00 |
| Prepare Shipments | 33 | 4.13 | \$37.13 | \$1,421.26 | \$1,421.26 | \$0.00 |
| Problem Labs | 20 | 2.50 | \$37.13 | \$861.37 | \$861.37 | \$0.00 |
| Clear Customs | 15 | 1.88 | \$37.13 | \$646.03 | \$646.03 | \$0.00 |
| Postage | | | \$5.41 | \$50.20 | \$50.20 | \$0.00 |
| FedEx Shipping | | | \$0.00 | \$0.00 | \$0.00 | \$0.00 |
| Total Canadian Labs Revenue minus Variable Expenses: | | \$721.14 | \$721.14 | \$0.00 | | |
| Per Lab Canadian Revenue minus Variable Expenses: | | | \$90.14 | \$90.14 | \$0.00 | |

| | Current | Proposed | |
|---|----------|----------|--------|
| Variable Expenses Differences: USA Labs minus Canadian Labs | 2006 | 2007 | Change |
| Per Lab USA Revenue minus Variable USA Expenses: | \$282.44 | \$282.44 | \$0.00 |
| Per Lab Canadian Revenue minus Variable Canadian Expenses: | \$90.14 | \$90.14 | \$0.00 |
| DIFFERENCE USA Labs minus Canadian Labs: | \$192.30 | \$192.30 | \$0.00 |

B. Minnesota Department of Agriculture funding

The immediate future of this program is unclear. MDA will have to make substantial changes to make the program self sustaining over the next year.

VII. Future manure lab analysis needs and existing reference methods

Tasks:⁹ Work with the testing industry and certifying agencies such as U.S. EPA, the Association of Official Analytical Chemists (AOAC) and the Soil and Plant Analysis Council to designate new reference methods and identify where new methods are needed. Assist laboratories in adopting the use of reference methods. The Board will provide leadership in finding research funds for continued methodology development.

A. Water Extractable Phosphorus (WEP)

The WEP test was added in 2006 based on input from our Advisory Board and participating laboratories. The method is not in the manual, and it may be too soon to add it to the manual until we have more data.

B. Electrical Conductivity (EC)

Even after four years laboratories are still struggling with EC. We don't have enough experience yet to recommend a specific method, and there does not seem to be a method universally accepted by the labs.

C. Ammonium Nitrogen

Ammonium nitrogen results were quite spread out the first two years, but this may have been an issue with a number of labs using the wrong unit to report their results. In 2005 NH₄-N was sub-divided into the following methods of determination: Ion selective electrode (ISE), spectrophotometric (Spec), and distillation (Dist). Results since 2005 indicate a slight low bias by the Spec method relative to the other two, which are nearly identical across sample types.

D. Updates for Recommended Methods of Manure Analysis

⁹ Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 5.

The manual, *Recommended Methods of Manure Analysis* was first published in 2003. The following topics could be addressed in the next manual update depending on future funding:

1. Sample preparation prior to laboratory analysis

One significant challenge facing laboratories is how they select a representative subsample for analysis from the ½ to 1 liter bottle provided by their customers. Should the manure be dried, pulverized, shaken and poured, or sampled with a dip tube? If drying is necessary, are some nutrients lost? We should start asking these questions and see if labs that dry manure before analysis are losing significant quantities of nutrients. There was a consistent low bias by four reporting labs for sample M-2006-G, possibly associated with drying of the samples and volatilization loss of NH₄-N nitrogen.¹⁰

2. Published method for the Water Extractible Phosphorus (WEP) test

The WEP test was added to the MAP Program in 2006. This is still an experimental test, and it is not listed in the *Recommended Methods of Manure Analysis*. As we get more experience with this test, its methods should be published in a revised manual.

3. Differences in macro versus micro Total Kjeldahl Nitrogen (TKN)

The results for TKN are quite precise. However, we started asking labs to specify if they are digesting a macro (over 1 gram sample) or micro (less than 1 gram sample) for TKN in the 3rd round of 2006. Additional results from different manure sources are required before we will know if there is a difference between using micro or macro digestion for TKN.

4. Future tests to include in the MAP Program

Calcium, magnesium, pH, sodium, iron, and manganese were analyzed more frequently in the double blind study than some of the other tests currently in the MAP Program. Laboratories should be contacted and asked if there is a regional need to include these tests in the MAP Program.

5. Laboratory customer reports and transcription errors

¹⁰ MAP Program Proficiency Report 3rd Exchange 2006, Dr. Robert Miller, October 10, 2006, page 2.

Other sections of this report have described problems with reports laboratories provide to clients and also with typical transcription errors found in the MAP exchange reports. These problems should be included in a revised methods manual.

6. Diagnosing laboratory errors and trouble shooting

The manure manual should have a section on correcting analysis problems discovered by participating in the MAP Program. Instructions and examples on using problem analysis tools, such as fishbone diagrams, would give laboratories a systematic method to correct specific analysis problems.

E. Promotion of existing reference methods

The manual, *Recommended Methods of Manure Analysis*, was first published in 2003 – the first year of the MAP Program. This is the first and only methods manual for manure analysis in North America.

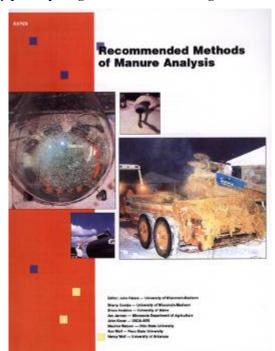


Figure 90: Each laboratory participating in the 2003 MAP Program received a copy.

The manual is also available on the Web at these URLs: http://uwlab.soils.wisc.edu/pubs/A3769.pdf

or

http://www.sera17.ext.vt.edu/Documents/Recommended_Methods_Manure_Analysis.pdf

Laboratories participating in the MAP Program are encouraged to use methods from this manual. The Wisconsin URL to download the manual is printed on the cover letter accompanying the samples and also on the spreadsheet used by laboratories to report their results. The editor, John Peters, gave presentations about the new manual at six of the regional workshops sponsored by the MAP Program.

VIII. Promote consistent, simplified recommendations for manure sampling

Tasks: ¹¹ Work with NRCS and other state/federal agencies in the development of manure sampling guidance materials using information from State Extension Services and previously conducted research on variability in test results due to sampling. Develop material in a variety of formats that can be used to help producers adopt easy to use sampling practices that are site-specific and that result in representative manure samples.

A. Workshops and presentations for manure lab clients

1. 2005 Upper Midwest Manure Handling Expo

Manure Handling for the 21st Century

August 11, 2005

Southern Research and Outreach Center, University of Minnesota, Waseca, Minnesota Jerry Floren served on the planning committee and had a demonstration on *Collecting Representative Manure Samples for Laboratory Analysis*

Audience: Approximately 700 (primarily commercial manure applicators and large livestock producers).



Figure 91

¹¹ Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 5.



Figure 92



Figure 93



Figure 94



Figure 95



Figure 96

2. Open house Southern Research and Outreach Center

University of Minnesota, Waseca, Minnesota September 15, 2005 Display by Jerry Floren: *Getting a representative manure sample*

3. Minnesota Water 2005 and Annual Water Resources Joint Conference

October 25-26, 2005 Brooklyn Center, Minnesota Jerry Floren displayed the following poster: *Methods and results from the first two years of the Manure Analysis Proficiency Program* Audience: 500+ primarily government staff and researchers.

4. Minnesota Water 2006 and Annual Water Resources Joint Conference

October 24-25, 2006 Brooklyn Center, Minnesota Jerry Floren and Robert O. Miller displayed the following poster: *Certification of Laboratories for Manure Analysis* Audience: 500+ primarily government staff and researchers.

IX. Evaluate program impacts

Tasks: ¹² Assess improvements in laboratory performance of manure analysis through statistical evaluation of check sample results. Assess laboratory use of reference methods, reporting recommendations and educational material through laboratory surveys. Assess increases in the use of laboratory manure analysis through laboratory tracking of manure samples. Assess changes in producer behavior, use of recommendations and practice adoption through a variety of measurement tools.

A. Trends from the beginning to end of study

MAP analytical methods were based on those listed in *Recommended Methods of Manure Analysis* edited by John Peters (http://cecommerce.uwex.edu/pdfs/A3769.PDF) and include: total solids, moisture content, electrical conductivity (EC), NH₄-N, total Kjeldahl nitrogen (TKN), combustion nitrogen, phosphorus, potassium, sulfur, zinc and copper. NH₄-N was separated into three methods reflecting the analytical method of determination: electrode, spectrophotmetric and distillation methods. Water Extractable Phosphorus (WEP), an experimental method not included in the manual, was added to the MAP Program in 2006.

Data analysis of each sample included: the number of results; minimum value, maximum value; median value; median absolute deviation (MAD); overall reproducibility (Rd); individual reported lab values; repeatability (Rp) of lab value; and mean lab value reported. Median and MAD were used to analyze the data as a majority of the data sets evaluated in the MAP and other lab proficiency programs (CAP, NAPT, USGA-PT) indicate these data sets are non-normally distributed and skewed. Lab proficiency <u>Confidence Limits</u> were based on the median ± 2.5 X the MAD and bias flagged as *L or *H. Lab precision was based on Rp values exceeding three (3) times the Rd, and flagged with a *P adjacent to the Rp results.

¹² Quoted directly from: A National Laboratory Proficiency Testing Program for Manure Analysis, a Grant Proposal Submitted to the U.S. Environmental Protection Agency, Revised August 7, 2002, page 6.

B. Assessment of analytical methods

1. General

Since 2003 the MAP program has completed 12 exchanges testing 36 manure samples, evaluating the performance of 84 analysis laboratories. Lab proficiency generally improved over the four years with total solids and moisture showing significant improvement. Generally labs fell into three classes: those proficient analyzing all manure types (i.e. liquid, semi-solid and solid types); those proficient with liquid samples but not solids; and those proficient at solid samples but not liquids. Greatest lab improvements were for labs un-accustomed to analyzing liquid samples. Proficiency improvement was noted for nitrogen and potassium, but to a lesser degree. Phosphorus showed only small improvement in inter-lab quality. Sulfur, zinc, and copper analyses generally showed only small improvement inter-lab proficiency. Significant improvements in NH4-N analyses only occurred after sub-dividing the analysis into three dominant methods. In 2006 Water Extractable Phosphorus (WEP) was added to the MAP program, and although only 12 labs analyzed WEP there has been improvement during 2006. Lastly there was generally little improvement in EC as there appears to be no accepted defined method used by the testing labs. Efforts to standardize have not reduced the amount of inter-lab variation for the EC method.

2. Accuracy and Precision

Overall 90% of the labs obtained results within 10% of the median value for total solids, with the exception of liquid samples containing less than 5% TS, when the value was within 20% of the median. Results for nitrogen indicate that 85% of the labs obtained within 10% of the median value, with the exception of liquid samples containing less than 0.3% nitrogen, when the value was within 50% of the median. Results for phosphorus indicate 90% of the labs obtained results within 20% of the median value, with the exception for liquid samples containing less than 0.04% phosphorus, when the value was 35% of the median. Lastly results for potassium indicate 90% of the labs obtained results within 15% of the median, with the exception of liquid samples containing less than 0.2% K, when the value was 25% of the median.

Results for the MAP program from 2003 through 2006, based on lab precision (RSD values) indicate that for the TKN method there is a loss of precision for nitrogen contents less than 0.3% N, for the combustion N method contents less than at 0.6% N. For total phosphorus at this concentration was 0.05% P. For concentrations below these levels RSD values increase exponentially, as the each of the methods approach detection limits.

Overall laboratory performance, based on precision and proficiency indicates significant improvement. Across the three exchanges precision scores in 2003 were 92% and proficiency 84.8%. In 2005 these values averaged were 92.1% and 85.8%, respectively. In 2006 these values improved to 92.8% and 86.6%. For 2006 precision scores average 92.5% and proficiency scores averaged 89.3% across exchanges. Overall, there was 4.5% improvement in lab proficiency scores over the four years.

3. Total Solids

An average of 66 labs provided total solid results. Manure total solids for samples in the MAP program since 2003 have ranged from 1.5% to 83.0%, with MAD values averaging 1.0% to 1.5% of the median for the solid samples and 10% to 14% for the liquid samples. Reproducibility (R*d* intra-lab precision) average ranges from 0.3% to 0.9% for the solid samples and from 4% to 6% for the liquid samples. Moisture content was added in the 2006 MAP program, to reinforce with the labs the difference between total solids and moisture content, as this was an issue for four to eight labs annually. Inter-lab and intra-lab precision for moisture approximated results found for total solids.

4. Total Nitrogen (TKN or N-Combustion)

Nitrogen (TKN or N-Combustion) An average of 47 labs provided TKN results. Manure TKN for samples in the MAP program since 2003 have ranged from 0.039% to 3.74%, with inter-lab MAD values averaging from 2.8% to 6.0% of the median for the solid samples and from 7.0% to 23% for the liquid samples. Reproducibility (R*d* intra-lab precision) averages 2.0% for the solid samples and 3.2% to 6.5% for the liquid samples. An average of 30 labs provided N Combustion results potassium results. Results for N by combustion ranged from 0.039 - 3.92%, with inter-lab MAD values averaging 3.5 - 5.1% of the median for the solid samples and 10 - 15% for the liquid samples. Reproducibility (R*d* intra-lab precision) averages 1.8% for the solid samples and 4.0% to 6.2% for the liquid samples. There was continued improvement in R*d* values for both TKN and N combustion from 2003 through 2006.

5. Potassium

An average of 66 labs provided potassium results. Manure potassium for samples in the MAP program since 2003 have ranged from 0.038% to 1.40%, with inter-lab MAD values averaging 4.8% - 7.4% of the median for the solid samples and 7.0% - 8.5% for the liquid samples. Reproducibility (R*d* intra-lab precision) averages 2.1% for the solid samples and 3.0% for the liquid samples. There was continued improvement in R*d* values for potassium from 2003 through 2006 reducing from 3.0% to 2.0%.

6. Phosphorus

An average of 66 labs provided phosphorus results. Manure phosphorus for samples in the MAP program since 2003 have ranged from 0.014% to 1.34%, with inter-lab MAD values averaging 5.3% to 10.2% of the median for the solid samples and 7.4% to 18% for the liquid samples. Reproducibility (R*d* intra-lab precision) ranged from 1.2% to 3.5% for the solid samples and from 2.2% - 4.2% for the liquid samples. There was continued improvement in R*d* values for phosphorus in 2006 over that of 2003 and 2004 exchanges.

7. Ammonium Nitrogen

An average of 55 labs provided NH₄-N results. In 2005 NH₄-N was sub-divided into three well defined methods of determination: Ion selective electrode (ISE), spectrophotometric (Spec) and distillation (Dist). Manure NH₄-N for samples in the MAP program since 2003 have ranged from 42 mg kg⁻¹ to 12,600 mg kg⁻¹, with inter-lab MAD values averaging 8.5% to 18.0% of the median for the solid samples and 7.6% to 45% for the liquid samples. Reproducibility (Rd intra-lab precision) averages 2.5% to 5.1%. Results since 2005 indicate a slight low bias by the Spec method relative to the other two, which are nearly identical across sample types. Reproducibility (Rd intra-lab precision) indicates the ISE method averages 2.0%, Spec method averages 2.5% and Dist method averages 1.4%. There was continued improvement in Rd values for both TKN and N combustion from 2005 through 2006.

8. Copper

Copper had an average of 55 labs provided results. Manure copper results since 2003 have ranged from 0.37 mg kg⁻¹ to 279 mg kg⁻¹, with inter-lab MAD values averaging 4.6% to 9.1% of the median for the solid samples and 8.6% to 37% for the liquid samples. Reproducibility (R*d* intra-lab precision) ranged from 1.6% to 3.2% for the solid samples and from 2.2% to 10.9% for the liquid samples. There was continued improvement in R*d* values for copper from 2003 through 2006.

9. Sulfur

Sulfur was added to the MAP program in 2004, with an average of 36 labs providing results. Manure sulfur for samples in the MAP program since 2004 have ranged from 0.010% to 0.48%, with inter-lab MAD values averaging 7.5% to 12.0% of the median for the solid samples and 7.6% to 20% for the liquid samples. Reproducibility (R*d* intra-lab precision) ranged from 1.8% to 3.3% for the solid samples and 3.6% to 6.2% for the liquid samples. There was continued improvement in R*d* values for sulfur in 2006 over that of the 2003 and 2004 exchanges.

10. Zinc

An average of 54 labs provided results for zinc. Manure zinc for samples in the MAP program since 2003 have ranged from 1.5 mg kg⁻¹ to 323 mg kg⁻¹, with inter-lab MAD values averaging 8.0% of the median for the solid samples and 14% for the liquid samples. Reproducibility (R*d* intra-lab precision) ranged from 2.0% to 3.2% for the solid samples and 3.4% to 11.0% for the liquid samples. There was continued improvement in R*d* values for zinc from 2003 through 2006.

11. Electrical Conductivity (EC)

Electrical conductivity was completed by an average of 37 labs. EC was defined as 1:1 and 1:2 methods in 2006. Manure EC for samples in the MAP program since 2003 have ranged from 4.8 dS/m to 16.1 dS/m, with inter-lab MAD values averaging 7.1% to 43% of the median. EC was highly correlated with NH4-N content. Reproducibility (R*d* intra-lab precision) ranged from 1.5% - 6.9%. There was some improvement in EC in 2006; however, it continues to have the highest inter-lab variation of all tests in the MAP program.

12. Water Extractable Phosphorus

WEP was added to the MAP program in 2006, with an average of 12 labs providing results. Manure WEP for samples in the MAP program have ranged from 2700 mg kg⁻¹ to 10,060 mg kg⁻¹, with inter-lab MAD values averaging 18.5% to 23.0% of the median. Inter-lab variation was high as this is a new method performed by only 18% of the labs participating in the MAP program. Reproducibility (R*d* intra-lab precision) ranged from 2.2% to 3.7%.

C. Transcription errors

Transcription errors plagued laboratories throughout this study. It is challenging to enter data on forms that are different than the forms the laboratory normally uses. This is compounded when the units normally used by a specific laboratory are different than the units required for the MAP report. Fortunately, most laboratories did not repeat the same transcription errors in subsequent reports. The following are some of the most common transcription errors:

- Using the wrong units
- Reporting percent moisture instead of total solids
- Misplacing the decimal point
- Confusing samples
- Reporting on the replicates instead of the samples
- Submitting the wrong report (for example, submitting Round 1 for 2005 instead of Round 1 for 2006).
- Entering the data on the wrong cell of the report form
- Digit transposition (for example, 501 reported as 105).
- Calculation errors

X. Appendix A – Certification of laboratories for manure analysis

This purpose of the MAP project was to develop a proficiency program for manure testing laboratories. Hopefully, laboratories participating in proficiency programs have a higher level of performance than non-participating laboratories. However, a proficiency program does not in itself provide an accurate gauge of a laboratory's ability to perform specific analyses.

Extra steps are required to evaluate laboratory performance. In 1996 the Minnesota Department of Agriculture began certifying laboratories for manure testing, and this is the only manure testing certification program in North America. Certification evaluates each laboratory's performance on different tests in the MAP Program, and laboratories with acceptable performance are invited to become certified for manure testing.

Approximately 80 percent of labs participating in the MAP Program have acceptable levels of performance to meet the certification requirements. For certification, each laboratory is given a score for each test. The score depends on the number of samples attempted (0 to 9), number of flags for accuracy, and the number of flags for precision.

Laboratory results from the proficiency program are used to assign scores to laboratories. Median and Median Absolute Deviation (MAD) values are calculated for each analysis on the nine different manure samples exchanged during the year. Laboratories with results exceeding -4.0 MAD units from the median are flagged "**L" (low). Results greater than +4.0 MAD units from the median are flagged "**H" (high). Laboratories are flagged "**L" or "*H" for results greater than ± 2.5 MAD units from the median but less than ± 4.0 MAD units from the median. These are considered "accuracy" flags and five points are deducted for each accuracy flag a laboratory receives for a particular analysis.

In addition to losing points for accuracy, labs may also lose points for poor precision. The mean, standard deviation and Coefficient of Variation (C.V.) are calculated for each of the three replicates (101, 102, and 103) for a particular manure and analysis. The median C.V. is calculated and labs with C.V. values for their three replicates exceeding three times the overall median C.V. for all laboratories are flagged for poor precision with a "*P" and three points are deducted.

Scoring system objectives:

- To be eligible for certification labs must send in results for at least five of the nine samples.
- At least 80% of eligible labs will pass a particular analysis for endorsement. If the data is tight, more than 80% of the labs may have passing scores.

- Each analysis is worth eight points. Five points are deducted for results that exceeded ± 2.5 MAD units from the median. These were marked with the following: *L, *H, **L, or **H.
- Three points were deducted for low precision where the C.V. of the three replicates exceeded three times the overall median C.V. of all the labs.
- For certification, labs must have passing scores for both **nitrogen and phosphorus**; other tests are certified individually.

| Number of Samples Analyzed | Maximum Score | Automatic Passing Score |
|-------------------------------|---------------|----------------------------|
| 9 | 72 | 52 |
| 8 | 64 | 49 |
| 7 | 56 | 46 |
| 6 | 48 | 43 |
| 5 | 40 | 40 |
| 4 or less | 32 or less | Not eligible |

Note: In some instances it was not possible to reach the 80 percent objective of labs passing a particular test using the table above. In those instances, enough labs with scores slightly below the minimum passing score were given passing scores to make the final percent of labs passing at least 80 percent. Additional labs were added in 2006 (for 2007 certification) to meet the 80% level for Copper, NH_4 -N, Potassium, and Zinc.

Certification results from 2006 for certification in 2007. Additional laboratories were given passing scores for NH4-N, Phosphorus, Potassium, Total Kjeldahl Nitrogen, and Zinc in order to have at least 80% of laboratories receive passing scores for those

| Analysis | Raw Percent Passing Labs | Additional labs given passing scores | Final percent of labs passing |
|----------------|-----------------------------|--------------------------------------|----------------------------------|
| Copper | 77.2% | 2 | 80.7% |
| EC | 85.3% | 0 | 85.3% |
| N (Combustion) | 86.2% | 0 | 86.2% |
| NH4-N | 79.6% | 1 | 81.5% |
| Phosphorus | 81.5% | 4* | 89.1% |
| Potassium | 75.4% | 3 | 80.0% |
| Sulfur | 80.5% | 0 | 80.5% |
| T.K.N. | 83.0% | 1* | 85.1% |
| Total Solid | 85.3% | 0 | 85.3% |
| Zinc | 77.8% | 2 | 81.5% |

analyses

*In order to have about 80 percent of the labs passing both nitrogen and phosphorus, the percent of labs passing phosphorus and nitrogen by TKN was increased.

While this is the only manure certification program in North America, it should be kept in mind that it evolved from the Minnesota Department of Agriculture's certification

program of 14 Midwestern labs in 1996. Now typically 40 to 50 labs are certified throughout the United States and in Canada.

To truly make this a national certification program, the following work is necessary:

- Set up an advisory panel of stakeholders
- Conduct meetings around the country to determine regional differences and needs
- Determine the tests needed in various regions
- Rewrite MDA's rule for soil and manure testing certification to reflect the stakeholders' requirements in various regions

XI. Appendix B – Advisory board meeting minutes

A. Indianapolis, IN (November 12, 2002)

Manure Proficiency Testing Advisory Board Meeting One Minutes Westin Hotel, Indianapolis, IN

November 12, 2002

Attending:

Dr. Greg Binford University of Deleware

Mr. Nat Dellavalle Dellavalle Laboratory, Inc. Fresno, CA 93728-1298

Mr. Jerry Floren Minnesota Department of Agriculture

Dr. Robert Miller Colorado State University

Mr. Bruce Montgomery Minnesota Department of Agriculture

Ms. Roberta Parry EPA East, Office of Waters

Mr. John Peters Univesity of Wisconsin

Mr. Keith Reid Ontario Ministry of Agriculture and Food

Dr. Charles Shapiro University of Nebraska

Dr. Ann Wolf Agricultural Analytical Services Lab Agenda:

1:30 PM to 1:40 PM Introductions

1:40 PM to 2:00 PM Bruce Montgomery – Overview of the EPA Grant Agreement for the Manure Proficiency Testing Program

2:00 PM to 2:20 PM Bob Miller-Benefits of Proficiency Testing and Performance Indicators

2:20 PM to 2:30 PM Jerry Floren – Logistics and Timelines for Converting a State to a National Program

2:20 PM to 2:40 PM Bob Miller – Overview of the Educational and Technical Support Component

2:40 PM to 2:50 PM John Peters/Ann Wolf – Update on Standard Methods and Laboratory Methods Manual

2:50 PM to 3:00 PM Discussion on challenges ahead and schedule next meeting

Bruce Montgomery Overview of the EPA Grant Agreement for the Manure Proficiency Testing Program

Handouts:

- A National Laboratory Proficiency Testing Program for Manure Analysis, a grant proposal submitted to the U.S. Environmental Protection Agency (Revised August 7, 2002)
- Develop and Implement National Manure Proficiency Testing Program
- Manure Proficiency Testing Program Advisory Board Meeting (on back of above)

Our unit in the Minnesota Department of Agriculture (MDA) runs several programs to reduce NPS pollution.

Farm Nutrient Management Assessment Program (FANMAP). An in depth set of interviews with 700 farmers to learn about their nutrient management practices. FANMAP has implications for manure management. When farmers just use commercial fertilizer their fertilizer rates are close to University of Minnesota recommendations. However, in more complex cropping systems proper credit for nutrients from manure or legumes is seldom given. The animal numbers are not the problem – rather it is manure management that causes problems. One problem is that many farmers do not understand the reports or the reporting units.

The Minnesota Pollution Control Agency enforces feedlot regulations in Minnesota. MDA received pass through funds for two Extension staff and funds to develop a Manure Analysis Certification Program. The manure certification program began in 1996 with 14 labs participating. In 2002, 50 labs participated and 43 labs received certification. Beginning in October of 2002, the Environmental Protection Agency provided MDA with \$176,000 to convert our manure certification program into a "National Laboratory Proficiency Testing Program for Manure Analysis." The Soil Science Society of America will administer the program; this includes collecting fees from participating labs and disbursing the fees to other participants. Bob Miller of the North American Proficiency Testing (NAPT) Program will develop the proficiency aspects, conduct seven regional workshops for lab staff, and promote the program through brochures, mailings, and a Web site. MDA will provide the proficiency test samples.

The advisory board will provide oversight to the program. A key challenge is to make the program self sufficient after the initial, two year funding from EPA is completed. We need to determine how much the labs can pay.

Review of Work Plan (on handout)

- 1. Establishment of the Advisory Board
- 2. Develop and Implement National Manure Proficiency Testing Program
- 3. Provide Training and Technical Assistance Activities
- 4. Develop Strategies and Techniques to Quantify Long-term Improvements
- 5. Develop a Long-term Strategy for Program Sustainability
- 6. Provide Recommendations on Future lab Analysis Needs
- 7. Promote use of Consistent Simplified Recommendations
- 8. Assess and Evaluate Program Impacts and Final EPA Report

A key task is to find a way to make this a long-term, sustainable program.

Bob Miller

Benefits of Proficiency Testing and Performance Indicators

Bruce Montgomery and I have a long history. About six years ago we looked at soil proficiency testing and launched the North American Proficiency Testing (NAPT) Program. We provided soil, water, and plant proficiency testing samples. NAPT has 166 labs – 130 USA, 25 Canadian, and other labs in Chile, Mexico, and Guatemala. Labs receive five soil samples, three plant samples, and three water samples. NAPT tracks 92 separate analysis methods. Labs should only submit analysis results for the tests they normally run. We need at least eight labs to submit data for a particular test/method to provide statistically valid results.

NAPT uses the median and Median Absolute Deviation (MAD) units to analyze lab results. The MAD is similar to standard deviation except is based on the median rather than the mean. Results outside the 90% confidence level are flagged. The labs receive four sets of five soils throughout the year (one set per quarter). One of the five soils is repeated throughout the course of the four exchanges. Iowa, Minnesota, Nebraska, Missouri, Indiana, and Ontario evaluate lab performance based on the lab's NAPT results, and I provide NAPT data to them. I only know one other statistical program that might be better than MAD, but it is quite involved. For the manure proficiency testing program we plan to use two types of tests, single blind and double blind. With the single blind test the labs would receive nine samples of manure, three times a year. The nine samples would only be three different manure samples so the labs would actually be running three samples in triplicate. This will allow us to determine not only the labs accuracy, but also their precision. The single blind samples would only be provided to labs that wanted to participate in the program and paid a fee to participate. For the two years of EPA funding we expect the fee to be about \$200 per year. This is a single blind test because the labs will know these are proficiency test samples; however, they will not know the nutrient content of the samples.

In the double blind test labs will receive unmarked samples. We will try to make these look like they were submitted by a farmer or crop consultant. Two double blind tests will be conducted. We will send anonymous manure samples to all labs we can find that analyze manure. This will be done near the beginning of the program and near the end. The double blind test allows several possibilities for further evaluation. How well do labs perform on ordinary manure samples? What reports do they provide farmers? Do the labs that participate in the proficiency testing program perform better than non-participating labs? Has lab performance improved during the course of the program?

I also provide proficiency testing for labs that analyze materials used in golf course construction. A single golf green costs about \$50,000 to construct; the last thing the golf course wants is a golfer to get muddy shoes on the green. In order to assure quick draining, greens are constructed with a sand base. The particle size distribution of this base depends on the climate and rainfall in a region. Therefore, a particularly important test for labs working with the golf course industry is the ability to properly determine particle size distribution. For golf courses the most important specifications have been developed over the past 40 years and have been in place during the last 15 years.

The two handouts show the types of reports received by the labs involved in the golf course proficiency testing (PT) program. Similar reports could be prepared for the manure PT program. I propose that labs have the opportunity to test for the following in the manure PT program:

- Total Nitrogen
- Total phosphorus
- ➢ Total potassium
- Dry matter content
- > Zinc
- ➢ Copper
- Electrical conductivity

In addition to proficiency testing program, we will also have to opportunity to evaluate the analysis methods used by labs and the reports that labs send to their clients. Are some methods better than others? Can we find acceptable methods that use more environmentally friendly reagents? If labs make recommendations, are their recs based on land grant recommendations? Are the reporting units farmer-friendly?

Jerry Floren

Logistics and Timelines for Converting a State to a National Program Three handouts:

- List of 50 labs that paid fees and sent in applications for MDA's Manure Certification Program in 2002.
- List of 43 labs that met MDA's certification requirements in 2002 and were certified for manure analysis.
- Possible equipment for the Manure Proficiency Testing Program

NOTE: I was unable to load a CD of my original presentation onto the laptop, so some additional pictures are shown that were not available for my presentation.

As Bruce Montgomery mentioned, MDA's manure certification program has grown from 14 labs in 1996 to 50 labs applying, and 43 becoming certified in 2002. For certification, each lab must meet the following requirements:

- Fill out an application, pay an initial registration fee of \$100, and pay an annual fee of \$100.
- Analyze eight manure samples in duplicate for dry matter content, total nitrogen, total phosphorus, and total potassium. Labs may also test for ammonium nitrogen as an optional endorsement for certification.
- Provide methods used for analysis.
- Provide client reports.
- > Have at least 80% of test results within ± 4.0 MAD units of the median.

We are now preparing to convert our existing certification program to a national proficiency testing program. A major challenge will be providing adequate amounts of check samples that are uniform from sample to sample within the batch. Fortunately, the EPA grant allows us to purchase equipment that will make it easier to prepare suitable volumes of homogenous check samples and speed up the bottling process. The following equipment is what we are considering for this program:

Patterson Kelly twin shell blender with intensifier

Cost: \$40,000 new and \$20,000 used

Recommended by Bob Miller and used by him to mix compost. The machines are available in a variety of sizes so that a single batch can provide enough manure for several rounds of check samples. A disadvantage of this machine is that you cannot load sample jars while it is running, the machine has to be stopped to remove material.

Bob's comments: Plan on purchasing a power washer for clean up. The V shell rotates about 25 times a minute and inside the V shell is a bar with tines. The bar spins much more rapidly than the shell and is effective in reducing particle size. If the machine runs for an hour, you get 1,500 splitting cycles. Compost particle size is about 1 mm after using this machine.

Robot Coupe 60 quart Tilting Vertical Mixer (R60TS)

Cost: \$6,500 for a new 25 quart, or \$18,750 for a new 60 quart. A used Hobart 40 quart vertical mixer is another possibility.

The MDA lab has a 25 quart Robot Coupe. Our lab supervisor says we should be able to reduce solid manure to pepper size particles when using these machines along with dry manure and dry ice. They can also be used for liquid and slurry manure. Unfortunately, you cannot sample during the mixing process, and even the 60 quart machine is not large enough to mix a single batch at one time. Therefore, these are prep machines, and further mixing would be required using other equipment.

There are a number of advantages to using manure with a particle size of pepper. It could be used as-is for a sample of solid manure. It could be mixed with liquid manure that had been decanted or filtered to produce manufactured manure ranging in consistency to a heavy paste to a dilute liquid with low dry matter content.

Robot Coupe MP-600 hand mixer (\$900).

This is similar to the high shear rotor-stator mixers. The MP-600 has an 80 gallon capacity. It is used in the restaurant business to prepare sauces and to prep cooked beans to prepare Mexican refried beans.

It has a cutter that should reduce particle size while it mixes. It has many of the same advantages as the high shear rotor-stator mixer, but it is not as heavy duty. However, it operates on standard 110 volt and is light enough to be used in the field (with a generator). This is the first piece of equipment we will order.

Peristaltic pump (about \$2,500)

A pump similar to this would allow us to bottle liquid samples while they are being mixed with either the Robot Coupe MP-600 mixer, a rotor-stator mixer, or a prop mixer. The volume to pump can be set and the unit can run a purge-pump-purge cycle before each sample. This would replace the ladle and greatly speed up liquid bottling. It should also result in more uniform samples than using a ladle.

Bob Miller Overview of the Educational and Technical Support Component

Another component of the program is the seven regional workshops that will be conducted throughout the country. I will conduct three workshops in 2003 and four workshops in 2004. These will be one day workshops for lab staff on quality control, monitoring, new equipment/methods, Kjeldahl digestion, and sampling on the lab bench. The focus is on lab technique for analytical technicians. Most will have three to five years experience. The labs buy into the program much more readily when they have a chance to meet with me and participate in workshops. Tentative plans are for workshops in the following locations; however, this could change if there is an opportunity to piggy back onto another lab meeting:

John Peters and Ann Wolf Update on Standard Methods and Laboratory Methods Manual

END OF MINUTES FROM THE FIRST MAP ADVISORY BOARD MEETING

B. Denver, CO (November 4 and 5, 2003)

Manure Proficiency Testing Advisory Board Meeting Two Minutes

November 4 and 5, 2003 Denver, CO Jerry Floren

These meetings were held during the annual meeting of the Soil Science Society of America in Denver, CO. Because all the participants were not able to meet at the same time, two meetings were held. The first meeting was the evening of November 4, 2003 and the following members attended: Nat Dellavalle, Jerry Floren, Jerry Lemunyon, Bob Miller, John Peters, and Keith Reid. The following members attended the morning of November 5, 2003: Nat Dellavalle, Jerry Floren, Jerry Lemunyon, Keith Reid, and Ann Wolf.

Meeting Agenda

Bob Miller - First Exchange Results and Their Significance

Jerry Floren - Equipment and Methods Used to Produce the Proficiency Samples

Group Discussion/Brainstorming How can the MAP Program become self-sustaining? Who benefits from the program, and are they likely to provide financial support? How much can labs afford to pay for proficiency samples? How much does sample preparation cost?

Schedule a dedicated meeting; we have funds to provide for travel expenses. Who should attend? Should we start using conference calls?

Bob Miller – First Exchange Results and Their Significance

Sixty of 84 labs enrolled in the program submitted results for the first exchange. Results from the nine Canadian labs enrolled in the program were delayed due to problems importing manure through Canadian Customs and no Canadian lab results are included in the report.

Because each manure sample was analyzed in triplicate, there are up to 180 data points. This provides a nice data set. Another advantage of using triplicate samples is that laboratory precision can also be evaluated in addition to accuracy. Values were flagged with either a *H (high) or a *L (low) if the accuracy was outside ± 2.5 MAD units. Values were flagged with a *P if the Rp value exceeded three times the Rd value, and this denotes a lack of precision relative to the overall industry.

The tightest data set was for total solids, especially the solid turkey litter (M-03-A-101, 102, and 103).

Results for total nitrogen were also quite tight.

Ammonium nitrogen was highly variable. Eleven labs reported units in percent instead of ppm, but even correcting for that left more variability than we want. (Note by Jerry Floren: Two of the manure samples that will be shipped in round 2 were sprayed with distilled white vinegar to lower the pH. Also, the solid samples for round 2 were placed in sample bottles instead of the plastic bags used in round 1.)

Since the analysis for phosphorus, potassium, zinc, and copper uses the same digest, it was surprising to see the P results were about twice as variable as the results from the other analytes. Bob will check to see if the method (ICAP or colorimetric) influenced the results.

Electrical conductivity was quite variable, especially on the dry turkey litter where values ranged from 0.1 to 23. Bob will check to see if there is a regional difference. This test is mainly run only in the west. Bob will also check to see if there is a correlation between the variability in the ammonium nitrogen and the EC.

It is clear additional work is needed on ammonium nitrogen and electrical conductivity. The third exchange will have an artificial manure sample to help us get a better handle on these issues.

Jerry Floren – Equipment and Methods Used to Produce the Proficiency Samples

A Power Point presentation was shown with photographs of the equipment used to prepare the proficiency samples. Contact Jerry Floren if you would like a copy of the Power Point slides. The following are text descriptions of the procedures used for preparing the samples:

Solid Manure (turkey litter, dried cow patties, composted cow manure)

Process frozen manure in a 60 quart Robot Coupe Vertical Chopping Mixer (VCM). This machine has three serrated blades that revolve at around 3,600 RPM. It has been used primarily to process dryer manure, usually frozen and with dry ice. However, good results have also been obtained just by processing frozen samples without dry ice. When processing with dry ice, about three pounds of dry ice are added to 2.5 gallons of frozen, dry manure. This is processed for 20 to 30 seconds. It may also work for mixing liquid to slurry manure. However, it has only been used for drier manure up to this point.

After processing in the VCM, the manure is sifted through two sets of ¹/₄ inch hardware cloth. The sifted manure is then mixed in a cement mixer. We have two cement mixers.

The smaller is about three cubic feet and the larger is about 12 cubic feet. Sifted manure is mixed for 15 to 25 minutes in the cement mixer before placing it in the sample containers.

After mixing in a cement mixer the samples are transferred to the sample bottles using either three passes with a 1/3 cup measuring cup, or four passes with a 1/4 cup measuring cup. Lower C.V. values were obtained with four passes; however, there were significant coarse fragments (crushed rock/sand) in the three pass samples. This grit did not dissolve in the digest and may have contributed to their higher C.V. values.

Preparation of Liquid Manure

The liquid manure was prepared using a 1/20 horsepower mixer with a prop having a diameter less than three inches. Since then a larger mixer (3/4 HP with a 10 inch prop) has been purchased.

Turkey litter that had been processed in the VCM, sifted through the ¹/₄" hardware cloth, and sifted several times through a flour sifter was used to prepare the solid portion of the liquid manure. The liquid portion was a mixture of tap water and liquid manure run several times through a screened funnel.

The first set of samples was filled using a peristaltic pump. These samples had unacceptable C.V. values and were discarded. A second set of samples was prepared by draining directly through a tube. These also were discarded because of unacceptable C.V. values.

The last two sets were prepared by making use of a racking barrel. Liquid manure was placed in the racking barrel for 8 to 12 hours to allow the larger, denser solids to settle. Liquid was decanted from the racking barrel by a screened tube about six inches above the barrel base into a five-gallon bucket. The contents in the five gallon bucket were vigorously mixed with a Robot Coupe MP600 hand mixer. After this initial mixing the contents were place in a larger mixing barrel with three baffles and a 1/20th HP mixer. Four passes from the mixing barrel were made with a ladle to fill the sample bottles.

We have now obtained a much larger mixer for liquid manure. It has a ten-inch prop and a ³/₄ horsepower motor. It also has a variable speed controller.

Preparation of Slurry and Semi-solid manure

An unsuccessful attempt was made to prepare one set of high moisture, solid manure. The source was macerated solid manure from the University of Minnesota manure handling facility in St. Paul. Cow and swine manure at the U of M are commingled. Then the solid and liquid portions are mechanically separated. The solid portion runs through a macerator.

The macerated manure was frozen and processed with dry ice in the VCM. The particle size and homogeneity appeared excellent. However, when this was mixed in the cement mixer it balled up in pea size to golf ball size clumps. Probably another type of mixer is needed for solid manure with high moisture content. Nat Dellavalle suggested looking at mixers in the food industry.

Group Discussion/Brainstorming – How Can the MAP Program Become Self-Sustaining?

Who benefits from the program, and are they likely to provide support?

All meat producers benefit from accurate manure analysis. The pork producers are the group most likely to financially support the MAP program. Dairy and turkey growers may also be interested.

Crop producers, especially the corn growers, may provide support. Other than corn growers, are there other crop producers that use large volumes of manure?

Would the larger meat packing firms provide support?

How much can labs afford to pay for proficiency samples?

Laboratories benefit from the MAP Program and realize the \$100 fee is highly subsidized. Ann Wolf thought a realistic range for labs is around \$500 to \$550. The third sample set will have a survey enclosed estimating the costs to prepare the samples and asking the labs to respond with how much they could afford to pay in order to continue the MAP program.

Some ways to reduce the program costs were discussed.

Provide single samples instead of triplicates. Or, provide one or two samples in triplicate and the remaining as single samples. This would save on preparation, materials, shipping, and reduce the amount of freezer space.

Eliminate the double blinds.

Explore the possibility of using two day shipping instead of overnight.

Could the number of samples analyzed by the MDA lab as a homogeneity check be reduced? (Currently MDA analyzes 20 from each manure set for total nitrogen and total phosphorus.)

Look for another product that could be prepared with the existing equipment to offset the program costs. Perhaps a dried/ground reference sample could be developed to sell to labs in addition to the MAP samples. Jerry Floren will look into NIST protocols for preparing these samples.

Would there be a market for wine bedding samples or sewage sludge?

Could the program be expanded into a certification program, and charge labs an extra fee for certification?

How much does it cost to prepare proficiency samples?

The cost to prepare the samples was discussed. After the meeting, Jerry Floren refined his estimates and developed a new spreadsheet with estimated costs to prepare the MAP samples. These estimates are solely for the sample preparation costs expected to be incurred by the Minnesota Department of Agriculture and do not include any costs from the North American Proficiency Testing (NAPT) Program or from the Soil Science Society of America (SSSA).

There are variable and fixed costs incurred with sample preparation. Variable costs are linked to the following: Do any sample sets have to be discarded because of poor homogeneity? How many labs are participating? Are samples prepared in triplicate, duplicate, or single? Method of shipping and number of samples shipped? Cost for sample bottles (triplicate, duplicate, or single) Salary and travel costs relating to triplicate, duplicate, or single samples

The following costs are fixed, or they are a minor component of the overall cost: Rent for preparation space Equipment repair and maintenance Equipment depreciation Consumable supplies such as boxes, tape, paper etc. Insurance Phone and postage

The cost for double blind samples is also classified as a fixed cost and it is expected to be about \$150 per lab if 75 labs participate. Double blinds cannot be shipped to the Canadian labs because all manure shipments into Canada require an Import Permit that identifies the importer.

For the variable costs the following three scenarios were developed:

Scenario 1: Exactly like we are doing now. Nine different sets of manure in triplicate with shipments spring, summer, and fall. In addition, two sample sets had to be discarded because of poor homogeneity.

Scenario 2: Exactly like we are doing now except that no sets are discarded because of homogeneity issues.

Scenario 3: No sets are discarded, sets are collected as single sample bottles instead of in triplicates, and the MDA lab only analyzes 10 samples per set instead of 20 for the homogeneity check. Shipping is still by overnight delivery, but the cost is reduced because of the lower weight.

END OF MINUTES FOR THE SECOND MAP ADVISORY BOARD MEETING

C. Newark, DE (September 13, 2004) Manure Proficiency Testing Advisory Board Meeting Three Minutes September 13, 2004 Newark, DE

Advisory Board Member Present: Greg Binford, Nat Dellavalle, Jerry Floren, Jerry Lemunyon, Mike Hall, Bob Miller, John Peters, Keith Reid, Ann Wolf Joining by Phone: Bruce Montgomery, Charles Shapiro

Agenda

- MAP budget and expenses for check sample preparation and data analysis.
- Suggestions for making the MAP program self-sustaining.
- Developing a national certification program for manure analysis using the MAP samples.
- Should both precision and accuracy be used to evaluate lab performance?
- Should median and MAD be used instead of mean and standard deviation with outliers removed?
- Should labs be evaluated on a curve, or within agronomic rates?
- Should there be a provision for reruns? Or, could the best two of three rounds be used in place of reruns?
- Should labs be penalized for not meeting the deadline for reporting results?
- Should there be a "Double Blind" component? If so, how many double blinds are necessary?
- Methods and issues related to the available phosphorus tests.

I. MAP Budget and expenses for check sample preparation and data analysis and suggestions for making the program self-sustaining

Jerry Floren distributed a spreadsheet with the estimated costs for preparing the check samples as they are now being distributed (three shipments, three times a year, in triplicate, or 27 samples analyzed by each lab.) The projected cost for the Minnesota Department of Agriculture (MDA) to prepare and ship these samples is about \$41,100. The projected cost for Bob Miller of the North American Proficiency Testing (NAPT) Program to handle the data analysis and communicate with the labs is about \$1,000 per month. The cost for the Soil Science Society of America (SSSA) to promote the program and collect the fees is not known; however, Bob Miller shared that SSSA may raise its overhead fee to 25 percent.

We estimate that about the maximum that could be raised by the labs is in the \$22,500 to \$25,000 range. If the price to participate is raised to \$450 to \$500 we would expect about 50 labs to remain in the program. That means there is a significant shortfall between what the labs can afford to pay and what it costs to run the program.

These are the reasons why we feel that \$25,000 is near the upper end of what can be raised by fees paid by labs to participate.

- Fewer manure samples are run in labs than soil samples. There are probably only three to four labs that analyze 6,000 manure samples in a year. The majority probably only analyze about 600 samples in a year. The total of manure samples analyzed by all labs is probably around 25,000. A survey will be sent to the labs to get a better estimate for these numbers.
- There are about 60 labs that returned results for the first round of 2004. We hope that at least 50 of these will stay with the program if the price is increased to \$500.

Several other proficiency programs charge around \$500 to provide their samples. It should be kept in mind that the labs are also running 27 samples for the program. If an average cost for manure analysis is \$40, this represents an additional cost of \$1,080 for the labs to participate.

Several ways to reduce the costs for preparing the samples were discussed. These included the following:

- Have two exchanges instead of three. Perhaps having two shipments of four different manure samples instead of three shipments of three samples.
- Have only single samples instead of triplicates and do not have the labs run triplicates.
- Ship single samples instead of triplicates, but have the labs "double dip" or "triple dip" on different days to get the replicate data.
- Have a less expensive lab than MDA analyze the samples for homogeneity before shipping. (Note: at dinner John Peters said Wisconsin analyzes for percent moisture and total nitrogen for \$10 and \$19 for a full analysis. MDA charges \$28 for total N and total P and an additional \$12 for percent solids.)
- Use two day shipping instead of overnight.
- At dinner the possibility of shipping all the samples early in the year was discussed. Labs could be given a reduced rate (say \$450 instead of \$500, or free shipping) if they agreed to receive all their samples at one time and store them in their freezers until needed. This would save shipping costs, labor, travel expenses, and freezer space.

Even if very significant cost saving were made, there still is too large a gap between estimated expenses and projected fees collected from labs to totally support the program. Outside funding is necessary. The program is working well as it is now, and cutting components should be a last resort. We should at least ask the Environmental Protection Agency if they would consider contributing the shortfall amount. Perhaps this would be more palatable if the program was ramped up to include certification.

To obtain donations from outside sources they first should be identified and promotional material should be prepared to make our case. Some interested parties are the following:

- Livestock producers
- Meat processors
- Laboratory equipment manufacturers and vendors
- Corn growers
- State Associations of Agriculture

Bruce Montgomery stated that while the MAP Program is well known to labs that provide manure analysis, it is not well known to other audiences. We need to start preparing material and promoting the program to a wider audience. Jerry Floren will produce some promotional material and see if some popular farm journals can write an article about the program. Some of the items that should be explained in these efforts are the following:

- What is the range of results?
- What is it worth to have more accurate results?
- What happens if the results are erroneous?
- What is the confidence level of results for different tests?
- Why is it so difficult to prepare check samples from manure?

There was some discussion about the types of promotional material that could be produced. Some type of brochure or handout would be useful. A Web page could have letters of support from labs on how the program has helped them solve problems. Jerry Lemunyon said NRCS could provide a letter describing why they feel the MAP Program and more accurate manure analysis is important. The State Associations of Agriculture could also be asked to provide letters of support. A Power Point presentation on how the samples are prepared could also be on the Web.

II. Developing a national certification program for manure analysis using the MAP samples

Should both precision and accuracy be used to evaluate lab performance?

Bob Miller feels that precision (measured by using triplicates) is a very useful measure of a lab's performance and should be included in the certification. Ann Wolf feels that they do enough QA checks to monitor their precision and that replicates in a proficiency program require a lot more effort for very little additional information. Bob showed a lab that had a median value for its replicates near the median of all the labs, but had very poor precision. Ann and Jerry Floren would like to see how that lab performed on other tests. Were all of its tests near the median with a large range between the lower and upper value. It seems more likely that if a lab is struggling with precision, they will also be struggling with accuracy.

Ann had another concern about using precision (triplicates) for certification. Will labs report results on proficiency samples used for certification if there is a large spread in their results?

Should median and MAD be used instead of mean and standard deviation with outliers removed?

The MAP data is skewed. For this reason a method to handle outliers needs to be used. Everyone seemed to feel that the NAPT use of median and MAD is the logical way to handle the statistics for the MAP Program.

Should labs be evaluated on a curve, or within agronomic rates?

If we say that all labs must have at least 80 percent of their results within ± 2.5 MAD units of the median, we are saying that about 20 percent of the labs will fail. For total nitrogen the data is so tight that we could fail labs that actually are providing good numbers. If the data is so tight that the range from high to low does not make a significant difference in the recommendation given to the farmer, it does not seem fair to penalize a lab. There may be some tests where 100 percent of labs pass.

Should there be a provision for reruns? Or, could the best two of three rounds be used in place of reruns?

There seemed to be agreement that even the best of labs could have a bad reporting period, and some type of rerun option should be available. There is a provision for reruns in Minnesota's rule for soil testing labs. However, with NAPT replacing MDA as the provider for soil samples the rerun provision does not work. The labs see the results before MDA gets them. Instead, MDA now allows labs to drop their lowest quarter soil test results if their score is below the required 80 percent.

Another way to handle this would be to provide a separate rerun set for labs not meeting the requirements. There would be an extra fee for this set.

Should labs be penalized for not meeting the deadline for reporting results?

Everyone agreed that labs being certified needed to submit their results by the deadline. Thirty days was enough time to report manure test results.

Should there be a "Double Blind" component? If so, how many double blinds are necessary?

The cost of a double blind study, and the difficulty of getting double blind samples into some labs, makes this questionable. It will probably only happen if EPA or another government agency provides the funding.

III. Ann Wolf briefly described the Water Extractable Phosphorus test. It should be available within a year. A soluble P test will probably be added to the MAP Program.

END OF MINUTES FROM THE THIRD ADVISORY BOARD MEETING

XII. Appendix C – Participating MAP Laboratories

From 2003 through 2006 the number of laboratories that submitted applications for the MAP Program and paid the annual fee decreased from 84 laboratories in 2003 to 69 laboratories in 2006. This decrease was likely due to the MAP fee increasing from \$100.00 per year in 2003 to \$400.00 in 2006. However, the number of laboratories that submitted results (actively participating in the MAP Program) increased slightly during this time period from 65 laboratories in 2003 to 67 laboratories in 2006. Fifty-seven labs have been in the program for all four years and 11 labs have been in the program for 11 years.

| Year | Round | Number of Labs Enrolled in MAP | Number of Labs Submitting Results | Average Number of MAP Labs Submitting Results | Annual Fee |
|------|-------|-----------------------------------|--------------------------------------|--|------------|
| 2003 | 1 | 84 | 60 | | |
| 2003 | 2 | 84 | 66 | 65.3 | \$100.00 |
| 2003 | 3 | 84 | 70 | | |
| 2004 | 1 | 71 | 61 | | |
| 2004 | 2 | 75 | 66 | 63.7 | \$200.00 |
| 2004 | 3 | 76 | 64 | | |
| 2005 | 1 | 72 | 68 | | |
| 2005 | 2 | 73 | 67 | 66.3 | \$300.00 |
| 2005 | 3 | 73 | 64 | | |
| 2006 | 1 | 69 | 68 | | |
| 2006 | 2 | 69 | 68 | 67.0 | \$400.00 |
| 2006 | 3 | 69 | 65 | | |

| Number of Years Enrolled in MAP Program | Number of Laboratories |
|---|------------------------|
| 4 years | 57 |
| 3 years | 11 |
| 2 years | 5 |
| 1 year | 20 |

Tables on the following pages list the laboratories enrolled in the MAP Program from the years 2003 through 2006.

A. Participating MAP Laboratories in 2003

| Lab Name | Address 1 | City | State | Zip | Country |
|--------------------------------------|---------------------------------------|-----------------|-------|-------|---------|
| A&E Labs, Inc. | 79960 550th Ave. | Jackson | MN | 56143 | USA |
| A&L Analytical Lab | 2790 Whitten Rd. | Memphis | TN | 38133 | USA |
| A&L Great Lakes Labs | 3505 Conestoga Dr. | Fort Wayne | IN | 46808 | USA |
| A&L Plains Ag. Labs | 302 34th St. | Lubbock | ТХ | 79410 | USA |
| A&L Western Agri Labs | 1311 Woodland Ave., #1 | Modesto | CA | 95351 | USA |
| Ag & Environmental Testing Lab | 105 Carrigan Dr., 219 Hills Bldg. | Burlington | VT | 05405 | USA |
| Ag Analytical Services Lab | Tower Road | University Park | PA | 16802 | USA |
| Ag Resource Consulting, Inc. | 131 5th St. | Albany | MN | 56307 | USA |
| Agri Analysis, Inc. | PO Box 483 | Leola | PA | 17540 | USA |
| Agri-Check, Inc. | 323 6th St. | Umatilla | OR | 97882 | USA |
| Agricultural Diagnostic Laboratory | 1366 W. Altheimer Dr. | Fayettevile | AR | 72704 | USA |
| Agricultural Service Lab | 171 Old Cherry Rd. | Clemson | SC | 29634 | USA |
| Agri-King, Inc. | 18246 Waller Rd. | Fulton | IL | 61252 | USA |
| AgSource Cooperative | 106 N Cecil St. | Bonduel | WI | 54107 | USA |
| AgSource-Belmond Labs | 1245 Hwy. 69 N | Belmond | IA | 50421 | USA |
| AGVISE Laboratories | 902 13th St N | Benson | MN | 56215 | USA |
| Alvey Laboratory, Inc. | 1511 E Main St. | Belleville | IL | 62222 | USA |
| Analytical Lab, Univ. of Maine | 5722 Deering Hall | Orono | ME | 04469 | USA |
| AV Labs, Inc. | 64 N Broadway Ave. | Othello | WA | 99344 | USA |
| Best-Test Analytical Services | 3211 Citation Road NE | Moses Lake | WA | 98827 | USA |
| Brookside Laboratories, Inc. | 308 S Main St. | New Knoxville | OH | 45871 | USA |
| Cascade Analytical, Inc. | 3014 GS Center Rd. | Wenatchee | WA | 98801 | USA |
| Corn Belt Seed Testing | 1955-500th St SW | KaLona | IA | 52247 | USA |
| CSU Soil, Water, & Plant Testing Lab | c/o Central Receiving 200 W. Lake St. | Fort Collins | CO | 80523 | USA |
| Custom Laboratory, Inc. | 204 C St. | Golden City | MO | 64748 | USA |
| Dairyland Laboratories | 217 E Main | Arcadia | WI | 54612 | USA |

| Lab Name | Address 1 | City | State | Zip | Country |
|---------------------------------|------------------------------------|-----------------|-------|-------|---------|
| DANR Analytical Laboratory | One Shields Ave. | Davis | CA | 95616 | USA |
| Dellavalle Laboratory, Inc. | 1910 W. McKinley Ave. | Fresno | CA | 93728 | USA |
| Denele Agrilink Laboratories | 1232 South Ave. | Turlock | CA | 95380 | USA |
| Eco Agri Labs, Inc. | 3009 E Hwy. 12 | Willmar | MN | 56201 | USA |
| Edglo Labs, Inc. | 2121 E Washington Blvd. | Fort Wayne | IN | 46803 | USA |
| Fruit Growers Laboratory | 853 Corporation St. | Santa Paula | CA | 93060 | USA |
| IAS Laboratories | 2515 E University Dr. | Phoenix | AZ | 85034 | USA |
| International Ag Labs, Inc. | 800 W Lake Ave. | Fairmont | MN | 56031 | USA |
| Iowa Testing Laboratories, Inc. | 1101 North Iowa Street | Eagle Grove | IA | 50533 | USA |
| LGI Labs | 1532 DeWitt St. | Ellsworth | IA | 50075 | USA |
| Litchfield Analytical Services | 535 Marshall St. | Litchfield | MI | 49252 | USA |
| Midwest Laboratories | 13611 B St. | Omaha | NE | 68144 | USA |
| Monarch Laboratory | 563 E Lindo Ave. | Chico | CA | 95926 | USA |
| MVTL | 1126 N Front St. | New Ulm | MN | 56073 | USA |
| MVTL Laboratories, Inc. | 35 W Lincoln Way | Nevada | IA | 50201 | USA |
| NDSU Soil Testing | Box 5575, Waldron Hall #103 | Fargo | ND | 58105 | USA |
| Olsen's Laboratory | 210 E First St. | McCook | NE | 69001 | USA |
| Olson Biochemistry Labs | Box 2170, South Dakota State Univ. | Brookings | SD | 57007 | USA |
| Platte Valley Laboratories | 914 Hwy. 30 | Gibbon | NE | 68840 | USA |
| Rock River Lab, Inc. | N8741 River Rd. | Watertown | WI | 53094 | USA |
| SDK Laboratories | 1000 Corey Rd. | Hutchinson | KS | 67504 | USA |
| Servi-Tech Laboratories | 1816 E Wyatt Earp | Dodge City | KS | 67801 | USA |
| Servi-Tech Labs | 1602 Parkwest Dr. | Hastings | NE | 68902 | USA |
| Soil & Forage Analysis Lab | 8396 Yellowstone Dr. | Marshfield | WI | 54449 | USA |
| Soil Control Lab | 42 Hangar Way | Watsonville | CA | 95076 | USA |
| Soil Search LLC | 42125 S Morton Rd. | Kennewick | WA | 99337 | USA |
| Soil Testing Lab | MSU, Land Resrouces & Env. Sci. | Bozeman | MT | 59715 | USA |
| Soil, Plant & Water Lab | 2400 College Station Rd. | Athens | GA | 30602 | USA |
| SoilTest Farm Consultants | 2925 Driggs Dr. | Moses Lake | WA | 98837 | USA |
| Spectrum Analytic | 1087 Jamison Rd. | Washington C.H. | ОН | 43160 | USA |
| Stearns DHIA Laboratories | 825 12th St. S | Sauk Centre | MN | 56378 | USA |

| Stukenholtz Laboratory | PO Box 353 | Twin Falls | ID | 83303 | USA |
|--|------------------------------------|----------------|----|---------|--------|
| Sunland Analytical Lab, Inc. | 11353 Pyrites Way #4 | Rancho Cordova | CA | 95670 | USA |
| Sure-Tech Laboratories | 2435 Kentucky Ave. | Indianapolis | IN | 46221 | USA |
| SWAT Lab, Gerald Thomas Hall, Rm. 269 | Box 30003, MSC 3Q | Las Cruces | NM | 88003 | USA |
| UK Lexington Soils Lab | 103 Regulatory Science Bldg. | Lexington | KY | 40546 | USA |
| Univ. of Deleware | 531 S. College Ave. | Newark | DE | 19717 | USA |
| Univ. of Maryland, Soil Testing Lab NRSL | Rm. 0225 H.J. Patterson Hall | College Park | MD | 20742 | USA |
| Univ. of Wyoming Soil Test Lab | 16th & Gibbon | Laramie | WY | 82071 | USA |
| USU Analytical Lab | 166 Ag Science Bldg. | Logan | UT | 84322 | USA |
| Valley Tech Agricultural Lab | 2120 S "K" St. | Tulare | CA | 93274 | USA |
| Ward Laboratories, Inc. | 4007 Cherry Ave. | Kearney | NE | 68848 | USA |
| Waters Agricultural Laboratories, Inc. | 257 Newton Hwy. | Camilla | GA | 31730 | USA |
| Waters Agricultural Laboratories, Inc. | 2101 Calhoun Rd. | Owensboro | KY | 42301 | USA |
| West Virginia Dept. of Agriculture | HC 85, Box 302 | Moorefield | WV | 26836 | USA |
| Western Laboratories | 211 Hwy. 95 | Parma | ID | 83660 | USA |
| Woods End Research Laboratory | 20 Old Rome Rd. | Mt. Vernon | ME | 04352 | USA |
| A&L Canada Laboratories | 2136 Jetstream Rd. | London | ON | N5V 3P5 | CANADA |
| Accutest Laboratories, Inc. | 8-146 Colonnade Rd. | Ottawa | ON | K2E 7Y1 | CANADA |
| Agri-Food Laboratories | 1-503 Imperial Rd North | Guelph | ON | N1H6T9 | CANADA |
| Enviro-Test Laboratories | 124 Veterinary Road | Saskatoon | SK | S7N 5E3 | CANADA |
| Lab IRDA | 2700 Einsten Complexe Scientifique | Ste Foy | QC | G1P3W8 | CANADA |
| Norwest Labs-Lethbridge | 3131 1st Avenue South | Lethbridge | AB | T1J 4H1 | CANADA |
| Norwest Labs-Winnipeg | 1357 Dugald Road | Winnipeg | MB | R2J 0H3 | CANADA |
| Soil & Feed Laboratory | 440 University Ave. | Charlottetown | PE | C1A 7N3 | CANADA |
| Soil & Nutrient Laboratory | 95 Stone Road West | Guelph | ON | N1H 8J7 | CANADA |
| Stratford Agri Analysis | 1131 Erie St. | Stratford | ON | N5A 6S4 | CANADA |

| Lab Name | Address | City | State | Zip | Country | Phone |
|--------------------------------------|-----------------------------------|-----------------|-------|------------|---------|--------------|
| A&L Analytical | 2790 Whitten Road | Memphis | TN | 38133 | USA | 901-213-2400 |
| A&L Canada Labs | 2136 Jetstream Road | London | ON | N5U3P5 | Canada | 519-457-2575 |
| A&L Eastern Labs | 7621 Whitepine Road | Richmond | VA | 23237 | USA | 804-743-9401 |
| A&L Great Lakes Labs | 3505 Conestoga Drive | Fort Wayne | IN | 46808 | USA | 260-483-4759 |
| A&L Western Agrilabs | 1311 Woodland Ave. #1 | Modesto | CA | 95351 | USA | 209-529-4080 |
| Accutest Labs | 8-146 Colonnade Road | Nepean | ON | K2E7Y1 | Canada | 613-727-5692 |
| Ag Analytical Service Lab | Tower Road | University Park | PA | 16802 | USA | 814-863-0841 |
| Ag Resource Consulting | PO Box 667; 131 5th Street | Albany | MN | 56307-0667 | USA | 320-845-6321 |
| Agri Analysis Inc | 280 Newport Road; PO Box 483 | Leola | PA | 17540 | USA | 717-656-9326 |
| Agri-Check, Inc. | 323 6th St., PO Box 1350 | Umatilla | OR | 97882 | USA | 541-922-4894 |
| Agricultural Services Lab | 171 Old Cherry Road | Clemson | SC | 29634 | USA | 864-656-2300 |
| Agriculture Diagnostic Lab | 1366 W. Altheimer Drive | Fayetteville | AR | 72704 | USA | 479-575-3908 |
| AgriFood Labs | 1-503 Imperial Road North | Guelph | ON | N1H6T9 | Canada | 519-837-1600 |
| Agri-King, Inc | 18246 Waller Road | Fulton | IL | 61252 | USA | 815-589-2525 |
| Agronomic & Environmental Labs Inc | 79960 550th Ave | Jackson | MN | 56143 | USA | 507-847-4767 |
| AgSource Belmond Labs | 1245 Hwy 69 | Belmond | IA | 50421 | USA | 641-444-3384 |
| AgSource Cooperative | 106 N. Cecil Street; PO Box 7 | Bonduel | WI | 54107 | USA | 715-758-2178 |
| Agvise Labs | 902 13th Street North; PO Box 187 | Benson | MN | 56215 | USA | 320-843-4109 |
| Alvey Lab | 1511 E. Main Street, PO Box 175 | Belleville | IL | 62222 | USA | 618-233-0445 |
| Analytical Lab | 5722 Deering Hall | Orono | ME | 04469 | USA | 207-581-2945 |
| Auburn University Soil Testing Lab | 961 S. Donahu Dr. | Auburn | AI | 36849 | USA | 334-844-3961 |
| Best Test Analytical Services | 3394 Bell Road NE Ste B | Moses Lake | WA | 98837 | USA | 509-766-7701 |
| Brookside Labs | 308 South Main Street | New Knoxville | ОН | 45871 | USA | 419-753-2448 |
| ChemRight Laboratories, Inc. | 117 N. Main St. | Maquoketa | IA | 52060 | USA | 563-652-4226 |
| Cornbelt Feed Lab | 1955 500th Street SW | Kalona | IA | 52247 | USA | 319-683-2201 |
| CSU Soil, Water, & Plant Testing Lab | 200 W. Lake Street | Fort Collins | СО | 80523 | USA | 970-491-5061 |
| Custom Lab, Inc | 204 C Street | Golden City | MO | 64748 | USA | 417-537-8337 |
| Dairyland Labs | 217 E Main Street | Arcadia | WI | 54612 | USA | 608-323-2123 |
| Dairyland Labs | 217 E Main Street | Arcadia | WI | 54612 | USA | 608-323-2123 |
| DANR Analytical Labs | 1 Shields Ave | Davis | CA | 95616 | USA | 530-752-0147 |

B. Participating MAP Laboratories in 2004

| Delaware Dept of Agriculture | 2320 South DuPont Hwy | Dover | DE | 19901 | USA | 302-698-4527 |
|--------------------------------------|---------------------------------------|----------------|----|------------|--------|--------------|
| Dellaville Labs | 1910 W. McKinley Ave Ste 110 | Fresno | CA | 93728 | USA | 559-233-6129 |
| IAS Laboratories | 2515 E. University Dr. | Phoenix | AZ | 85034 | USA | 602-273-7248 |
| International Ag Labs | 800 W. Lake Ave; PO Box 788 | Fairmont | MN | 56031 | USA | 507-235-6909 |
| Iowa Testing Laboratories | 1101 North Iowa Ave | Eagle Grove | IA | 50533 | USA | 515-448-4741 |
| IRDA | 2700 Einstein Complexe Scientifique | Ste-Foy | QC | G1P3W8 | Canada | 418-644-6821 |
| LG, Inc | 1532 DeWitt Street | Ellsworth | IA | 50075 | USA | 515-836-4444 |
| Litchfield Analytical Services | 535 N. Marshall Street | Litchfield | MI | 49252 | USA | 517-542-2915 |
| Midwest Labs | 12611 B Street | Omaha | NE | 68144 | USA | 402-334-7770 |
| MVTL Labs | 35 West Lincoln Way | Nevada | IA | 50201 | USA | 515-382-5486 |
| North Dakota State Univ | Waldron Hall #103; PO Box 5575 | Fargo | ND | 58105 | USA | 701-231-9589 |
| Norwest Labs | 3131 1st Ave South | Lethbridge | AB | T1J4H1 | Canada | 403-329-9266 |
| Norwest Labs | 1357 Dugald Road | Winnepeg | MB | R2J0H3 | Canada | 204-982-8630 |
| Olsen's Lab | 210 E. 1st St; PO Box 370 | McCook | NE | 69001 | USA | 308-345-3670 |
| Olson Biochemistry Labs SDSU | 1029 N. Campus Drive; Box 2170 | Brookings | SD | 57007 | USA | 605-688-6171 |
| PEI Analytical Labs | 440 University Ave; PO Box 1600 | Charlottetown | PE | C1A7N3 | Canada | 902-368-5622 |
| Platte Valley Labs | 914 Hwy 30; PO Box 807 | Gibbon | NE | 68840 | USA | 308-468-5975 |
| Rock River Lab, Inc | 710 Commerce Drive; PO Box 169 | Watertown | WI | 53094 | USA | 920-261-0446 |
| ServiTech Labs | 1816 E. Wyatt Earp Blvd; PO Box 1397 | Dodge City | KS | 67801 | USA | 620-227-7123 |
| ServiTech Labs | 1602 Parkwest Drive | Hastings | NE | 68901 | USA | 402-463-3522 |
| Soil & Nutrient Laboratory | 95 Stone Road West | Guelph | ON | N1H8J7 | Canada | 579-767-6226 |
| Soil, Plant, & Water Lab | 2400 College Station Road | Athens | GA | 30602-9105 | USA | 706-542-5350 |
| SoilTest Farm Consultants | 2925 Briggs Drive | Moses Lake | WA | 98837 | USA | 509-765-1622 |
| Spectrum Analytic | 1087 Jamison Road; Box 639 | Washington | ОН | 43160 | USA | 740-335-1562 |
| Stearns DHIA Labs | 825 12th St. S; PO Box 227 | Sauk Centre | MN | 56378-0227 | USA | 320-352-2028 |
| Sunland Analytical | 11353 Pyrites Way #4 | Rancho Cordova | CA | 95670 | USA | 916-852-8557 |
| SWAT Lab NMSU | 2990 Knox Street | Las Cruces | NM | 88003 | USA | 505-646-4422 |
| SWFAL Oklahoma State | 048 Ag Hall | Stillwater | ΤХ | 74078 | USA | 405-744-7771 |
| Univ of Delaware | 153 Townsend Hall; 531 S. College Ave | Newark | DE | 19717 | USA | 302-831-1385 |
| Univ of Kentucky Soils Lab Lexington | 103 Regulatory Services Bldg | Lexington | KY | 40546 | USA | 859-257-2785 |
| USU Analytical Labs | 166 Ag Science Bldg | Logan | UT | 84322-4830 | USA | 435-797-2217 |
| UVM Ag & Environmental Testing Lab | 105 Carrigan Drive | Burlington | VT | 05405 | USA | 802-656-3030 |
| UW Soil & Forage Lab | 8396 Yellowstone Drive | Marshfield | WI | 54449 | USA | 715-387-2523 |
| Valley Tech Ag Lab | 2120 South K Street | Tulare | CA | 93277 | USA | 559-688-5684 |
| Ward Laboratories | 4007 Cherry Ave; PO Box 788 | Kearney | NE | 68848 | USA | 308-234-2418 |

| Waters Agricultural Lab | 257 Newton Hwy; PO Box 382 | Camilla | GA | 31730 | USA | 229-336-7216 |
|-------------------------|------------------------------|------------|----|-------|-----|--------------|
| Waters Agricultural Lab | 2101 Calhoun Road | Owensboro | KY | 42301 | USA | 270-685-4039 |
| Western Laboratories | 211 Hwy 95, PO Box 1020 | Parma | ID | 83660 | USA | 208-722-6564 |
| Woods End Research Labs | 20 Old Rome Road; PO Box 297 | Mt. Vernon | ME | 04352 | USA | 207-293-2457 |
| WV Dept of Agriculture | 60B Industrial Park Road | Moorefield | WV | 26836 | USA | 304-538-2397 |

C. Participating MAP Laboratories in 2005

| Lab Name | Contact | Address_1 | Address_2 | City | State | Zipcode | Country |
|---|---------------------------|------------------------------------|-----------------------------|--------------|-------|----------------|---------|
| | | | | | | | |
| SoilTest Farm Consultants | BRENT THYSSEN | 2925 DRIGGS DR | | MOSES LAKE | WA | 98837 | USA |
| Kuo Testing Labs, Inc. | EUGENE KUO | 337 S 1st AVE | | OTHELLO | WA | 99344 | USA |
| Best-Test Analytical Services | Stephen Jones | 3394 Bell Road NE | | Moses Lake | WA | 98837 | USA |
| AV Labs Inc. | ALMA L BARAJAS | 64 N BROADWAY AVE | | OTHELLO | WA | 99344 | USA |
| Agri-Check, Inc. | DARA RUSSELL | 323 6TH STREET | PO BOX 1350 | UMATILLA | OR | 97882 | USA |
| A&L Western Agri Labs | JITENDRA LAL | 1311 WOODLAND AVE | SUITE #1 | MODESTO | СА | 95351 | USA |
| DANR Analytical Labs | DIRK M HOLSTEGE | HOAGLAND ANNEX, ATTN: GARY CHAN | UNIVERSITY OF CALIFORNIA | DAVIS | СА | 95616 | USA |
| Dellavalle Labs | PEGGY MILLER | 1910 W MCKINLEY AVE | SUITE 110 | FRESNO | СА | 93728 | USA |
| VALLEY TECH AG LAB | SAM MODESITT | 2120 SOUTH "K" ST | | TULARE | CA | 93274 | USA |
| Stukenholtz Laboratory | PAUL STUKENHOLTZ | 2924 ADDISON AVE E | PO BOX 353 | TWIN FALLS | ID | 83301 | USA |
| Western Laboratories | CATHY BINGHAM | 211 HWY 95 | PO BOX 1020 | PARMA | ID | 83660 | USA |
| USU Analytical Labs | JANICE KOTUBY- AMACHER | UTAH STATE UNIVERSITY | AG SCIENCE RM 166 | LOGAN | UT | 84322- 4830 | USA |
| IAS Laboratories | Sheri McLane | 2515 E University Dr. | | Phoenix | AZ | 85034 | USA |
| CSU Soil, Water, & Plant Testing Lab | JAMES R SELF | A319 NESB | 200 W LAKE ST | FORT COLLINS | СО | 80523- 1120 | USA |

| SWAT Lab NMSU | F.W. Boyle, Jr. | 2990 Knox St. | | Las Cruces | NM | 88003 | USA |
|------------------------------|-------------------|----------------------------|---------------------|-------------|----|-------|-----|
| North Dakota State Univ | LARRY SWENSON | PO BOX 5575 | WALDRON HALL 103 | FARGO | ND | 58105 | USA |
| Olson Biochemistry Labs SDSU | NANCY THIEX | OLSON BIOCHEMISTRY LABS | BOX 2170 | BROOKINGS | SD | 57007 | USA |
| Midwest Labs | JEROME J KING | 13611 B ST | | OMAHA | NE | 68144 | USA |
| ServiTech Labs | MICHAEL PERRY | 1602 PARK WEST DR | | HASTINGS | NE | 68901 | USA |
| Ward Laboratories | RAY WARD | 4007 CHERRY AVE | PO BOX 788 | KEARNEY | NE | 68848 | USA |
| Olsen's Lab | KEVIN GROOMS | 210 E FIRST ST | PO BOX 370 | МССООК | NE | 69001 | USA |
| Platte Valley Labs | STUART PESEK | 914 HIGHWAY 30 | PO BOX 807 | GIBBON | NE | 68840 | USA |
| ServiTech Labs | SEAN JENKINS | 1816 E WYATT EARP BLVD | PO BOX 1397 | DODGE CITY | KS | 67801 | USA |
| OK State Soil Testing | TRAVIS HANKS | 048 AG HALL | | STILLWATER | OK | 74078 | USA |
| Servi-Tech Laboratories | STEVE HARROLD | 6921 S BELL | | AMARILLO | TX | 79109 | USA |
| Agvise Labs | CINDY EVENSON | 902 13TH ST N | PO BOX 187 | BENSON | MN | 56215 | USA |
| Ag Resource Consulting | JOANNE PROM | 131 5TH ST | PO BOX 667 | ALBANY | MN | 56307 | USA |
| MVTL Labs | Mary Ann Baumgart | 1126 N. Front St. | | New Ulm | MN | 56073 | USA |
| International Ag Labs | PAT FLEMING | 800 W LAKE AVE | PO BOX 788 | FAIRMONT | MN | 56031 | USA |
| A&E Labs | RON SINN | 79960 550TH AVE | | JACKSON | MN | 56143 | USA |
| Stearns DHIA Labs | SAMANTHA ADAMS | 825 12TH ST S | PO BOX 227 | SAUK CENTRE | MN | 56378 | USA |
| Rock River Lab, Inc | TWILAH KULOW | 710 COMMERCE DR | PO BOX 169 | WATERTOWN | WI | 53094 | USA |
| AgSource Cooperative | STEVE PETERSON | 106 N CECIL ST | PO BOX 7 | BONDUEL | Wi | 54107 | USA |

| Dairyland Laboratories | DAVE TAYSOM | 217 EAST MAIN ST | | ARCADIA | WI | 54612 | USA |
|--|-----------------|----------------------------|-----------------------------------|--------------------------|----|----------------|-----|
| UW Soil & Forage Lab | TINA SEEGER | UNIVERSITY OF WISCONSIN | 8396 YELLOWSTONE DR | MARSHFIELD | WI | 54449 | USA |
| MVTL Labs | TERESA C SJULIN | 35 W LINCOLN WAY | PO BOX 440 | NEVADA | IA | 50201 | USA |
| Iowa Testing Laboratories, Inc. | Jack W. Henry | 1101 North Iowa Street | PO Box 188 | Eagle Grove | IA | 50533 | USA |
| AgSource Belmond Labs | RANDY LAW | 1245 HYW 69 | | BELMOND | IA | 50421- 7554 | USA |
| Custom Lab, Inc | MONTY DADE | 204 C STREET | BOX 391 | GOLDEN CITY | МО | 64748 | USA |
| Agriculture Diagnostic Lab | NANCY WOLF | UNIV OF ARKANSAS | 1366 W ALTHEIMER DR | FAYETTEVILLE | AR | 72704 | USA |
| Litchfield Analytical Services | STAN FORCE | 535 N MARSHALL ST | PO BOX 457 | LITCHFIELD | MI | 49252 | USA |
| Alvey Lab | RANDY ALVEY | 1511 E MAIN ST | PO BOX 175 | BELLEVILLE | IL | 62222 | USA |
| Agri-King, Inc | JEFF HORST | 18246 WALLER ROAD | | FULTON | IL | 61252 | USA |
| A&L Great Lakes Labs | LOIS K PARKER | 3505 CONESTOGA DR | | FORT WAYNE | IN | 46808 | USA |
| Sure-Tech Laboratories | Bill Shakal | 2435 Kentucky Ave. | Bldg. 9 | Indianapolis | IN | 46221 | USA |
| Brookside Labs | GREG MEYER | 308 S MAIN ST | | NEW KNOXVILLE | ОН | 45871 | USA |
| Spectrum Analytic | VERNON PABST | 1087 JAMISON RD | PO BOX 639 | WASHINGTON COURTHOUSE | ОН | 43160 | USA |
| Univ of Kentucky Soils Lab Lexington | FRANK SIKORA | UNIV OF KENTUCKY | 103 REGULATORY SERVICE BLDG | LEXINGTON | KY | 40546 | USA |
| Waters Agricultural Lab | RHONDA WERNER | 2101 OLD CALHOUN RD | | OWENSBORO | KY | 42301 | USA |
| A&L Analytical | SCOTT MCKEE | 2790 WHITTEN RD | | MEMPHIS | TN | 38133 | USA |
| Auburn University Soil Testing Laboratory | Hamilton Bryant | 961 South Donahue Drive | | Auburn University | AL | 36849- 5411 | USA |

| | | | UNIVERSITY OF | | | | |
|---|---------------------------------|--------------------------------------|--------------------------|-----------------|----|----------------|--------|
| Analytical Lab | BRUCE HOSKINS | 5722 DEERING HALL | MAINE | ORONO | ME | 04469 | USA |
| Woods End Research Lab | WILLIAM BRINTON | PO BOX 297 | | MT VERNON | ME | 04352 | USA |
| UVM Ag & Environmental Testing Lab | ELIZABETH CARR / DONALD ROSS | 219 HILLS BUILDING | 105 CARRIGAN DRIVE | BURLINGTON | VT | 05405 | USA |
| Dairy One | MICHAEL J REUTER | 730 WARREN ROAD | | ITHACA | NY | 14850 | USA |
| Ag Analytical Service Lab | ANN M WOLF | THE PENNSYLVANIA STATE UNIVERSITY | TOWER RD | UNIVERSITY PARK | РА | 16802 | USA |
| Agri Analysis Inc | Tim Hoerner | 280 Newport Road | PO Box 483 | Leola | PA | 17540 | USA |
| Univ of Delaware | Karen Gartley | 153 Townsend Hall | 531 S. College Ave | Newark | DE | 19717 | USA |
| Delaware Dept of Agriculture | Teresa A. Crenshaw | 2320 South DuPont Hwy | | Dover | DE | 19901 | USA |
| Cumberland Valley Analytical Services | SHARON WEAVER | 14515 INDUSTRY DRIVE | | HAGERSTOWN | MD | 21783 | USA |
| A&L Eastern Agricultural Labs. Inc. | PAUL CHU | 7621 WHITEPINE RD | | RICHMOND | VA | 23237 | USA |
| WV Dept of Agriculture | MATTHEW SITES | 60B INDUSTIRAL PARK ROAD | | MOOREFIELD | WV | 26836 | USA |
| Agricultural Services Lab | KATHY MOORE | 171 OLD CHERRY RD | | CLEMSON | SC | 29634 | USA |
| Agricultural and Environmental Services Laboratories | DAVID E KISSEL | 2400 COLLEGE STATION RD | UNIVERSITY OF GEORGIA | ATHENS | GA | 30602- 9105 | USA |
| Waters Agricultural Lab | KEITH DOMINEY | 257 NEWTON HWY | PO BOX 382 | CAMILLA | GA | 31730 | USA |
| AgriFood Labs | PAPKEN BEDIRIAN | 1-503 IMPERIAL RD N | | GUELPH | ON | N1H 6T9 | Canada |
| Soil & Nutrient Laboratory | NICK SCHRIER | UNIV OF GUELPH | 95 STONE ROAD WEST | GUELPH | ON | N1H 8J7 | Canada |
| A&L Canada Labs | NIGEL STEADMAN | 2136 JETSTREAM RD | | LONDON | ON | N5V 3P5 | Canada |

| Stratford Agri Analysis | JAMES BRIMNER | 1131 ERIE ST | BOX 760 | STRATFORD | ON | N5A 6W1 | Canada |
|-------------------------|------------------------|---------------------------------------|---------------|---------------|-----|---------|--------|
| PEI Analytical Labs | MARLENE MCNEILL | 440 UNIVERSITY AVE | PEI DEP OF AG | CHARLOTTETOWN | PEI | C1A 4N6 | Canada |
| Norwest Labs | MONIQUE CHAPMAN | 3131- 1ST AVENUE SOUTH | | LETHBRIDGE | AB | T1J 4H1 | Canada |
| Lab IRDA | PIERRE AUDESSE | 2700 Einsten Complexe Scientifique | | Ste Foy | QC | G1P3W8 | Canada |
| | CHEDVL ANN | | | | | | |
| Norwest Labs | CHERYL-ANN SHURVELL | 1357 DUGALD ROAD | | WINNIPEG | MB | R2J 0H3 | Canada |

D. Participating MAP Laboratories in 2006

| Laboratory | Address 1 | Address 2 | PO Box | City | State | Zip Code |
|--|-------------------------|------------------------------|-------------|--------------------|-------|----------------|
| A & L Analytical | 2790 Whitten Road | | | Memphis | TN | 38133 |
| A & L Canada Labs | 2136 Jetstream Road | | | London | ON | N5V 3P5 |
| A & L Eastern Agricultural Labs. | | | | | | |
| Inc. | 7621 Whitepine Road | | | Richmond | VA | 23237 |
| A & L Great Lakes Labs | 3505 Conestoga Drive | | | Fort Wayne | IN | 46808 |
| A & L Western Agri Labs | 1311 Woodland Avenue | Suite 1 | | Modesto | CA | 95351 |
| Ag Resource Consulting, Inc. | 131 5th Street | | PO Box 667 | Albany | MN | 56307- 0667 |
| Agri Analysis Inc | 280 Newport Road | | PO Box 483 | Leola | PA | 17540 |
| Agri-Check, Inc. | 323 6th St. | | PO Box 1350 | Umatilla | OR | 97882 |
| Agricultural Analytical Service Laboratory | Tower Road | | | University Park | PA | 16802 |
| Agricultural & Environmental Testing Lab | 219 Hills Building | 105 Carrigan Drive | | Burlington | VT | 05405-0082 |
| Agricultural and Environmental Services Laboratories | University of Georgia | 2400 College Station Road | | Athens | GA | 30602-9105 |
| Agricultural Service Laboratory | 171 Old Cherry Road | | | Clemson | SC | 29634 |
| Agriculture Diagnostic Lab | 1366 W. Altheimer Drive | | | Fayetteville | AR | 72704 |
| AgriFood Labs | 503 Imperial Road North | Unit #1 | | Guelph | ON | N1H 6T9 |
| Agri-King, Inc | 18246 Waller Road | | | Fulton | IL | 61252 |
| AgSource Belmond Labs | 1245 Hwy 69 | | | Belmond | IA | 50421 |
| AgSource Cooperative | 106 N. Cecil Street | | PO Box 7 | Bonduel | WI | 54107 |
| Agvise Laboratories Inc. | 902 13th Street North | | PO Box 187 | Benson | MN | 56215 |
| Alvey Laboratory Inc. | 1511 E. Main Street | | PO Box 175 | Belleville | IL | 62221 |
| Analytical Lab | 5722 Deering Hall | University of Maine | | Orono | ME | 04469 |

| Auburn University Soil Testing Laboratory | ALFA Building, 961 South Donahue Drive | Auburn University | | Auburn | AL | 36849-5411 |
|---|--|-------------------------|-------------|------------------|----|------------|
| Best-Test Analytical Services | 3394 Bell Road NE | | | Moses Lake | WA | 98837 |
| Brookside Laboratories, Inc. | 308 South Main Street | | PO Box 456 | New Knoxville | ОН | 45871 |
| CSU Soil, Water, and Plant Testing Lab | 200 W. Lake Street | Room A319 NESB | | Fort Collins | со | 80523-1120 |
| Custom Laboratory, Inc | 204 C Street | | | Golden City | MO | 64748 |
| Dairy One | 730 Warren Road | | | Ithaca | NY | 14850 |
| Dairy Tech Labs | 805 Rohrerstown Road | | | Lancaster | PA | 17601 |
| Dairyland Laboratories | 217 E Main | | | Arcadia | WI | 54612 |
| DANR Analytical Labs | 1 Shields Ave | 207 Hoagland Hall | | Davis | СА | 95616 |
| Delaware Dept of Agriculture | 2320 South DuPont Hwy | | | Dover | DE | 19901 |
| Dellavalle Labs | 1910 W. McKinley Avenue | Suite 110 | | Fresno | CA | 93728 |
| IAS Laboratories | 2515 E University Dr. | | | Phoenix | AZ | 85034 |
| International Ag Labs | 800 W Lake Ave. | | | Fairmont | MN | 56031 |
| Iowa Testing Laboratories, Inc. | 1101 North Iowa Street | | PO Box 188 | Eagle Grove | IA | 50533 |
| Lab IRDA | 2700 Einsten Complexe Scientifique | | | Ste Foy | QC | G1P 3W8 |
| Litchfield Analytical Services | 535 N. Marshall Street | | PO Box 457 | Litchfield | MI | 49252 |
| Magic Valley Labs, Inc. | 210 Addison Avenue | | | Twin Falls | ID | 83301 |
| Midwest Labs | 13611 B Street | | | Omaha | NE | 68144 |
| MVTL Laboratories, Inc. | 1126 N. Front St. | | PO Box 249 | New Ulm | MN | 56073 |
| MVTL Labs | 35 West Lincoln Way | | | Nevada | IA | 50201 |
| North Dakota State University | Soil Testing Laboratory | Waldron Hall #103 | PO Box 5575 | Fargo | ND | 58105 |
| Norwest Labs - Bodycote | 3131 1st Ave South | | | Lethbridge | AB | T1J 4H1 |
| Norwest Labs/Bodycote | 1357 Dugald Road | | | Winnipeg | MB | R2J 0H3 |
| Olsen's Agricultural Lab | 210 East First Street | | PO Box 370 | McCook | NE | 69001 |
| PEI Analytical Laboratories | 440 University Avenue | | PO Box 1600 | Charlottetown | PE | C1A 7N3 |
| Platte Valley Labs | 914 Hwy 30 | | PO Box 807 | Gibbon | NE | 68840 |
| Rock River Lab, Inc | 710 Commerce Drive | | PO Box 169 | Watertown | WI | 53094-0169 |

| Servi-Tech Laboratories | 6921 S Bell | | | Amarillo | ΤX | 79109 |
|---|------------------------------|----------------------------|-------------|---------------------------|----|----------------|
| Servi-Tech Laboratories | 1602 Parkwest Drive | | PO Box 169 | Hastings | NE | 68902 |
| ServiTech Labs | 1816 E. Wyatt Earp Blvd | | PO Box 139 | Dodge City | KS | 67801 |
| Soil & Nutrient Laboratory | 95 Stone Road West | | | Guelph | ON | N1H 8J7 |
| Soil, Water and Forage Analytical Laboratory | Oklahoma State University | 045 Ag Hall | | Stillwater | ОК | 74078 |
| SoilTest Farm Consultants | 2925 Driggs Dr. | | | Moses Lake | WA | 98837 |
| South Dakota State University | Olson Biochemistry Labs | 1029 N. Campus Drive | Box 2170 | Brookings | SD | 57007 |
| Spectrum Analytic, Inc. | 1087 Jamison Road | | PO Box 639 | Washington Court House | ОН | 43160 |
| Stearns DHIA Labs | 825 12th St. South | | PO Box 227 | Sauk Centre | MN | 56378- 0227 |
| Stratford Agri Analysis | c/o Daco Laboratories Ltd. | 1131 Erie St. | Box 760 | Stratford | ON | N5A 6S4 |
| Stukenholtz Laboratory | 2924 Addison Ave East | | PO Box 353 | Twin Falls | ID | 83301-0353 |
| Sure-Tech Laboratories | 2435 Kentucky Ave. | Bldg. 9 | | Indianapolis | IN | 46221 |
| Univ of Delaware | 153 Townsend Hall | 531 S. College Ave | | Newark | DE | 19717 |
| Univ of Kentucky Soils Lab Lexington | 103 Regulatory Services Bldg | | | Lexington | KY | 40546 |
| USU Analytical Labs | 166 Ag Science Bldg | | | Logan | UT | 84322-4830 |
| UW Soil & Forage Lab | 8396 Yellowstone Drive | | | Marshfield | WI | 54449 |
| Ward Laboratories | 4007 Cherry Ave | | PO Box 788 | Kearney | NE | 68848 |
| Waters Agricultural Lab | 2101 Calhoun Road | | | Owensboro | KY | 42301 |
| Waters Agricultural Lab | 257 Newton Hwy | | PO Box 382 | Camilla | GA | 31730 |
| Western Laboratories | 211 Hwy. 95 | | PO Box 1020 | Parma | ID | 83660 |
| Woods End Research Laboratory | 290 Belgrade Road | | PO Box 297 | Mt. Vernon | ME | 04352 |
| WV Dept of Agriculture | 60B Industrial Park Road | | | Moorefield | WV | 26836 |