



### The Manure Analysis Proficiency Program: Trends in laboratory manure testing methods



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### Primary MAP goal

- Farmers rely on manure testing to determine crop nutrient requirements. The primary goal is to ensure farmers receive accurate manure testing results by using laboratories certified for manure testing.
- The MDA certifies laboratories for manure testing based on their performance in the MAP Program.
- Certification requires acceptable laboratory performance on both total phosphorus and on total nitrogen (either Total Kjeldahl Nitrogen or the Nitrogen by Combustion method).

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### Overview

The Manure Analysis Proficiency (MAP) Program, was established in the mid-1990s to assist analytical laboratories in the US Midwest verify the accuracy of laboratory manure analyses by the Minnesota Dept of Agriculture.

MAP methods are based on 1998 NCR-13 Publication: Recommended Methods of Manure Analysis, John Peters, University WI. Methods Manual:  
<http://www1.uwex.edu/ces/pubs/pdf/A3769.pdf>

Lab proficiency assessed based on an annual two single-blind exchanges of three manures samples representing dairy/beef, swine, poultry sources, each in triplicate.

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### MAP sample preparation

Manure sample preparation. Liquid manure samples are bulk homogenized, PT aliquots subsampled and frozen.

Solid manure samples are ground to minimum particle size, bulk blended and subsampled and frozen.

Manure proficiency samples homogeneity is evaluated based on total N and P analyses. Samples shipped frozen over night to laboratory participants.

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### MDA Proficiency data analysis

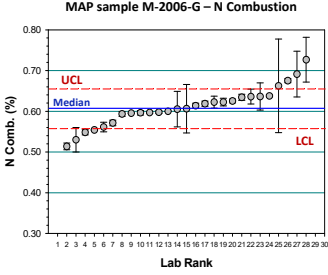
Lab proficiency is evaluated based on consensus of the inter-lab median and 95% confidence limits (CL) as calculated:

Median Absolute Deviation:  

$$MAD = \sum |x_i - x| / n$$

95% Confidence Limits (CL):  
 Median  $\pm$  (2.9 x MAD)

Individual lab method precision is assessed based on three replications (error bars at right).



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### MAP definitions

**Inter-Laboratory RMD:** Measure of relative variance of a method across multiple testing laboratories performing the analysis, as a calculated MAD/Median x 100.

**Intra-Laboratory RSD:** The median of the within laboratory of relative standard deviations for a method, as calculated by the median of all individual lab method RSD.

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### MAP reports

MAP reports provide a summary of method performance listing the median, 95% CL, intra-lab reproducibility, lab results, mean intra-lab repeatability and performance.

Lab 4012 results for sample M-04-C from 2004, for TKN at right was were flagged for low bias. Results for EC and TKN were flagged for poor precision.

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### MAP results - 2003

Proficiency results for a high solid vs liquid manure from 2003 are at right.

Inter-lab CL, as a percentage of the median, are generally lower for solid manures relative to liquid manure regardless of the parameter.

Method differences in N analysis were most significant.

Analysis	Sample M 2003-A		Sample M 2003-B	
	Median	95% CL	Median	95% CL
Total Solids (%)	74.0	2.1	1.54	0.26
TKN (%)	3.34	0.29	0.16	0.041
N - Comb (%)	3.42	0.35	0.17	0.055
P (%)	1.34	0.22	0.027	0.006
K (%)	1.33	0.15	0.093	0.017

Number of labs reporting: TS - 60, TKN - 48, N-Comb - 22, P - 59 and K - 59.

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### MAP N results 2003-2018

Inter-laboratory relative median deviations (RMD) results for nitrogen for 108 manure samples (TS 1.5 - 91%), 2003-2018, at right.

TKN inter-lab results show consistent RMD values < 7% for median N concentrations > 0.5%.

N-Comb results show consistent RMD values < 8% for manures > 0.8%, and RMD values 3-43% for lower concentrations.

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## MAP phosphorus

**MAP samples 2003-2018**

Inter-laboratory relative median deviations (RMD) results for total P for liquid (TS < 12%) and semi-solid/solid (TS > 12%) manures for 108 manure samples, 2003-2018, at right.

P inter-lab results show consistent RMD values ranging 5 – 18% for liquid manures with a mean of 10%. For solid and semi-solid manures the range was 4 – 11% with a mean of 6%.

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## Summary

- Results for N analysis show the TKN method consistently had the lowest inter-lab RMD values relative to N-Combustion. Factors:
  - TKN method uses larger sub sample size for analysis
  - Generally the N-combustion instrument has a higher MDL than TKN
- For liquid manures (TS < 12%) its recommended laboratories use the TKN method for N analysis.
- MAP results show continuing laboratory improvement inter-lab N analysis precision regardless of method, and differences in P RMD values between liquid and solid manures.
- Intra-laboratory RSD values had the poorest precision on samples with the lowest TS. Overall, across 108 PT samples, RSD values were < 4% across parameters evaluated.

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## Closing comments

### Acknowledgement

Thanks to Larry Gunderson, Supervisor Fertilizer Management Unit, Minnesota Department of Agriculture, St Paul, MN.

### MAP Methods Manual

The 2<sup>nd</sup> Edition of Recommended Methods of Manure Analysis has been published University of Minnesota Libraries Publishing and is available:  
<https://hdl.handle.net/11299/227650>

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