A Western Evaluation of Soil Testing Laboratory Performance

By Robert O. Miller

Researchers have reported on the variability in soil testing lab results by submitting a split sample to several different labs. Some of this variation can be explained by the use of different extraction procedures, but some is apparently due to lab error. Proficiency testing and performance verification are becoming increasingly important because of heightened nutrient management accountability, government programs, and environmental litigation.

With expanding utilization of nutrient management plans (NMPs) there is an increasing emphasis on soil sampling and testing. Variation in soil testing laboratory results have been documented through the submission of duplicate, blended samples to laboratories in the west (Davis et al., 1999; Lorbeer et al., 1999; Koenig, 2003). Although in specific instances this variation can be attributed to differences in extraction methodologies, variation among labs using identical procedures does exist (Miller and Kotuby-Amacher, 1997).

In 1998, as a membership activity of the Soil Science Society of America (SSSA), the North American Proficiency Testing (NAPT) Program was developed as a tool to assist soil testing laboratories across North America with the quality of their analysis. Guidelines for the NAPT program were developed by groups familiar with standardizing methods for soil and plant analysis and developing recommendations within the U.S. and Canada including: Regional Soil and Plant Analysis Workgroups; state/provincial Departments of Agriculture; the Soil and Plant Analysis Council, SSSA; the Canadian Society of Soil Science; and private and public soil and plant analysis laboratories. Participation in the program is voluntary. Annually, NAPT participating labs receive soil, plant, and water samples on a quarterly basis, and they subsequently provide a report on their soil testing proficiency. The NAPT program provides an opportunity for laboratories to under-go self improvement and make modifications to correct analysis problems. The NAPT Program offers to work with labs to resolve any problems and also offers workshops to improve the quality and precision of testing lab results.

The voluntary nature of the NAPT Program means that not all labs participate. In addition, submitted samples are analyzed by the labs as single-blind samples, in that the participating lab knows the samples are for proficiency testing and only the soil test value is unknown. Although many labs participate, not all provide results, and of the labs that do provide results, some have analysis values falling outside acceptable ranges.

Proficiency testing and performance assessment of soil testing labs is becoming even more important.

In Utah, it was reported that paired soil samples submitted by livestock producers to two laboratories gave contrasting results (Koenig, 2003). A survey of consultants and labs in the western region indicated that some form of laboratory certification program was needed for NMPs. In July of 2003, representatives of the USDA Natural Resources Conservation Service (NRCS) from the western U.S. and the NAPT Oversight Committee reviewed issues involving lab quality. It was agreed that a double-blind evaluation of the labs using standard reference soils would be the most cost effective means of assessing the performance of soil testing laboratories. Double-blind in this instance means soils of known values would be submitted by surrogate clients, such that the lab would not know the analytical value or the real source of the soils being tested. This program, the NAPT Performance Assessment Program (PAP), was endorsed by the NRCS in 2004. It was implemented as a pilot program for labs in the western U.S. which provide soil testing for NRCS-approved NMPs.

Participating labs in the 2004 PAP pilot were required to: 1) enroll in the NAPT program; 2) provide quarterly analysis of pH_{water}, electrical conductivity (EC), nitrate-nitrogen (NO_3-N), phosphorus (P), potassium (K), and soil organic matter (SOM) results; 3) provide soil method information on all client reports; 4) agree to a double-blind evaluation of their analytical performance; and 5) agree to a code of ethics. The program was based on seven soil analyses:

- pH, saturated paste or 1:1 (soil:water ratio)
- EC, saturated paste or 1:1 (soil:water ratio)
- NO_3-N, cadmium (Cd) reduction, ISE or CTA
- Ammonium (NH_4-N), all methods
- Phosphate (PO_4-P), Olsen (1:20) colorimetric
- K, ammonium acetate or Olsen extractable
- Organic matter, Walkely Black or Loss on Ignition (LOI)

Table 1. Chemical properties of soils utilized in the 2004 NAPT-PAP Program.

Soil	pH, 1:1 H_2O	NO_3-N, Cd red	P, Olsen	K (Am. acetate)	SOM (LOI)
		mg/kg[1]	mg/kg	mg/kg	%
2003-108	5.50	189	69	294	3.30
2003-119	6.43	7.3	45	122	1.20
2003-120	6.00	42.5	30	1,130	2.25
2004-102	8.52	45.0	115	482	1.89
2004-104	7.90	61.0	166	435	3.20

[1]mg/kg is equivalent to parts per million (ppm)

Five soil samples were selected from the NAPT 2003 and 2004 program archives for use in the PAP (**Table 1**). One soil was duplicated in the program to evaluate laboratory reproducibility on double-blind samples. Soils were prepared and aggregated to resemble "real world" lab samples. Surrogate lab clients were contacted and engaged in shipping and submitting samples to the participating lab.

Twenty-one labs enrolled in the 2004 pilot PAP program from the states of Washington, Oregon, California, Idaho, Utah, Montana, Wyoming, Colorado, North Dakota, Nebraska, Kansas, and Tennessee. Results were obtained from 20 labs. A review of the reports provided indicated that only two of the participating labs had provided method information, and in specific cases the unit information was not provided. Laboratory analytical performance was evaluated based on the median and median absolute deviation (MAD) of the double-blind database, using 90% (2.5xMAD) confidence interval for the 20 labs providing data.

A comparison of NAPT and PAP median and MAD values and those of the 20 PAP labs is shown in **Table 2** for soil 2003-120. A majority of the analyses in PAP had MAD values significantly higher than those observed in the NAPT program. This was potentially associated with additional variability introduced as a result of additional sample handling within the participating lab (drying and grinding). However, as the increased MAD values were strongly

Table 2. A comparison of NAPT and PAP median and MAD values for soil 2003-120.

Soil 2003-120 Platner	NAPT statistics Median	NAPT statistics MAD	PAP statistics Median	PAP statistics MAD
pH, 1:1 H_2O	6.10	0.10	6.20	0.20
EC, 1:1 H_2O, dS/m	0.44	0.10	1.1	0.51
NO_3-N, mg/kg	42	5	51	8
NH_4-N, mg/kg	1	1	7	3
P – Olsen, mg/kg	30	3.0	38.3	3.4
K -Am. acetate, mg/kg	1,100	94	992	175
SOM, %	1.68	0.20	2.00	0.20

associated with P and K analyses, lab analytical bias was likely a factor.

Performance of individual labs was based on a weighting of individual analyses (pH 5%; EC, 5%; NO_3-N, 30%; PO_4-P, 40%; K, 10%; and SOM, 5%) to determine the total proficiency score. Successful labs were those that met an overall proficiency score of 80%. This proficiency value was selected based on standard scores utilized in the Iowa and Minnesota soil lab registration programs.

A comparison of results for NAPT soil ID 2003-120 is listed in **Table 3**. Overall, 11 of 12 labs provided pH (1:1 H_2O) results within 0.25 units (2.5 x MAD) of the median. Labs #7, #8, #12, #14, #15, #16, and #17 provided pH by the saturated paste method. For NO_3-N, 5 of 18 reporting labs were flagged as exceeding the 90% confidence interval.

For Olsen P, 6 of the 17 reporting labs had results exceeding the 90% confidence interval of 38.0 ± 8.7 mg/kg. It is important to note that the majority of soil samples used in these evaluations had

Table 3. Results for soil 2003-120 from the PAP program.

2003-120 Platner	PAP values Median	PAP values MAD	Lab 1	2	3	4	5	6	7	8	9	10
pH, 1:1 H_2O	6.20	0.16	6.61*	6.00	6.40	6.10	5.80	6.20		6.10		6.40
EC, 1:1 H_2O, dS/m	1.1	0.41	0.2	1.2	1.8*		1.1			0.9		
NO_3-N, mg/kg	51	8.5	35	41	29*		48	167 *	51	59	51	51
NH_4-N, mg/kg	7	3	11	4								
P – Olsen, mg/kg	38	3.5	26*	35	23*	32	74*		41		34	39
K -Am. acetate, mg/kg	992	175	825	990	670		1,260	1,343	1,020	1,280	1,175	930
SOM, %	2.00	0.20	3.50*	1.80	1.62		2.3	2.00	1.58	2.00	1.60	2.10

	Median	MAD	11	12	13	14	15	16	17	18	19	20
pH, 1:1 H_2O	6.20	0.16	6.50							6.00	6.00	6.4
EC, 1:1 H_2O, dS/m	1.1	0.41	0.4	68					1.2	0.6		1.9
NO_3-N, mg/kg	51	8.5	210*		43	68	185*	49	64	44	61	29*
NH_4-N, mg/kg	7	3	10						7	4		
P – Olsen, mg/kg	38	3.5	43	200*	38	200 *	37	14.6 *	39	38		46
K -Am. acetate, mg/kg	992	175	971	332*	1,249	332*	400*	907	860	994	1,040	
SOM, %	2.00	0.20	2.20	3.10*	2.10	3.1*		1.80	1.93	1.80	2.00	1.95

* Lab values exceeding warning limit (2.5 x MAD) of PAP median.

Table 4. Comparison of PAP median and MAD duplicate soil submissions.

	2003-108 Thorndike - 1		2003-108 Thorndike - 2	
Analyses	Median	MAD	Median	MAD
pH, 1:1 H_2O	5.80	0.15	5.60	0.10
EC 1:1 H_2O, dS/m	3.13	1.86	2.22	1.24
NO_3-N, mg/kg	203	36	216	27
NH_4-N, mg/kg	32	7	37	15
P - Olsen, mg/kg	71	12	69	11
K -Am. acetate, mg/kg	266	22	274	26
SOM, %	2.46	0.50	2.36	0.44

Olsen extractable PO_4-P and NO_3-N levels above what is considered the normal agronomic range (see **Table 1**) which has traditionally been the range of greatest interest to users of soil testing. However, today the range of interest extends to much higher levels and failing to adjust calibration protocols can introduce additional error. For ammonium acetate K, 3 of the 19 reporting labs exceeded the 90% confidence interval of 992 ± 437 mg/kg.

Results of the PAP duplicate soil, NAPT 2003-108 Thorndike, generally indicated good reproducibility for the median values for all soil analysis methods (**Table 4**). The exception was soil pH (1:1 H_2O) which showed a 0.20 unit shift in the median values and soil EC (1:1) which showed a 0.91 dS/m shift. Since these samples were prepared and randomly selected, we can only conclude that this error is either linked to within-lab handling problems or method variability.

Based on an evaluation of laboratory performance using median and MAD values of the 20 participating labs and the method-weighting factors listed, the preliminary results of the PAP program indicate an overall median proficiency score for the 20 labs of 91%, with 5 labs failing to achieve a proficiency score of 80%. Those labs (5 of 20) not meeting the 80% proficiency score are being retested in 2005. Those which meet the PAP performance standards have been listed on the program website as soil analysis labs approved for NRCS-NMP. Visit the website at: >www.NAPT-PAP.org<.

Overall, the PAP program has identified that the majority of testing labs doing business in the western U.S. are capable of providing soil test results within 25% of the actual value. There are a few labs that currently are not meeting that standard. Since the PAP program assessed performance using known standards and defined methods, these variations can only be attributed to individual lab bias.

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References

Davis, J.G., D.B. Bosley, R. Buhler, and A.W. Cooley. 1999. Comparison of soil testing laboratories. p. 93-99. *In* T. Tindall and D. Westermann (eds) Proc. of the Western Nutrient Management Conference, March 4-5, 1999. Salt Lake City, Utah.

Koenig, R.T. 2003. Certification of soil testing labs. p 83-87. *In* J. Ellsworth (eds) Proc. of the Western Nutrient Management Conference, March 6-7, 2003. Salt Lake City, Utah.

Lorbeer, S., S. Schaff, and J. Jacobsen. 1999. A comparison of soil test lab results and recommendations. p. 101-106. *In* T. Tindall and D. Westermann (eds). Proc. of the Western Nutrient Management Conference, March 4-5, 1999. Salt Lake City, Utah.

Miller, R.O. and J. Kotuby-Amacher. 1997. Western states proficiency program. p. 192-197. *In* T. Tindall and D. Westermann (eds). Proc. of the Western Nutrient Management Conference, March 6-7, 1997. Salt Lake City, Utah.

Miller, R.O., J. Kotuby-Amacher, and N. B. Dellavalle. 1996. A proficiency testing program for the agricultural laboratory industry: results of 1994 program. Com. Soil Sci. Plant Anal. 27:451-461.

USDA-NRCS. 2005. Nutrient Management Standard 590. Utah NRCS. Available on the Internet at: >http://efotg.nrcs.usda.gov/treemenuFS.aspx?Fips=49035&MenuName=menue.ut.zip<