

Solum Soil Nitrate Analysis Report Evaluation Study

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FINAL REPORT

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Overview

A laboratory study was conducted based to evaluate the Solum Tech soil nitrate analysis system on field soils. A secondary objective was to evaluate method precision and the method detection limit.

Project Management

The project was overseen by Dr. Robert O. Miller Affiliate Professor Colorado State University, Fort Collins, CO 80523, rmiller@lamar.colostate.edu.

Study

Thirty field soil samples representing grid points of two fields were collected by Aron Quist of Stanworth Laboratory near Blythe, California January 8, 2011, and shipped on ice to Windsor, Colorado. Soils were stored at 0-4 C and then samples were mixed (moist) and processed through Humbolt riffle splitter with 0.5 inch shoots. Each soil sample was individually poured through the rifle splitter three times and then split in sub samples A and B. Split sample bags were relabeled, and set A containing 30 soils was shipped to Solum Tech, California for analysis and set B to Ward Laboratories, Kearney, Nebraska, for nitrate analysis by the cadmium reduction method. Samples were weighed for soil analysis and extracted 1:5 prior to analysis by Ward laboratories.

Each laboratory was asked to provide triplicate analysis on three of the 30 samples representing a low, medium and high testing soil. Each was asked to determine the method detection limit as per criteria described in the California Analytical Methods Manual, Dairy General Order Compliance – Nutrient Management Plan Constituents, Method S-1.10 Nitrate-Nitrogen: http://anlab.ucdavis.edu/docs/uc_analytical_methods.pdf.

Comparison of nitrate analysis was made for the 30 samples and the method detection limit.

Results

Results for the seventeen soils collected from field R66 shown in Figure 1, show a R2 of 0.996 with a slope of 0.956 between Cd Reduction NO3-N and Solum nitrate method. Soil ranged from 11.9 to 70.4 ppm nitrate. It was noted that the method showed deviation on the high testing nitrate range.

Results for the thirteen soils collected from Field RW88 shown in Figure 2, show a R2 of 0.984 with a slope of 1.02 between Cd Reduction NO3-N and Solum nitrate method. Soil ranged from 4.9 to 16.7 ppm nitrate. It was note worthy the method showed a consistent 1.1 mg per kg low bias across the nitrate range. The high coefficient of correlation shows good agreement, but a consistent 1.0 low bias relative to the Cd Reduction method.

Combining the two fields the thirty soils shown in Figure 3, show a R2 of 0.995 with a slope of 0.952 between Cd Reduction NO3-N and Solum nitrate method. Again there was a consistent low bias on all samples of approximately 1.0 ppm nitrate. The source of low bias could not be readably identified, but it is suspected that it could be related to the preparation of the samples prior to analysis (drying and grinding), calibration standards or associated with background contamination. Note, that increases in nitrate during processing would likely effect low versus high testing soils differentially.

Overall there was very good agreement between the two methods equal to that one would expect between two separate laboratories performing nitrate analysis on thirty samples. The high R2 for the two sample sets indicates good homogeneity for the two sample splits.

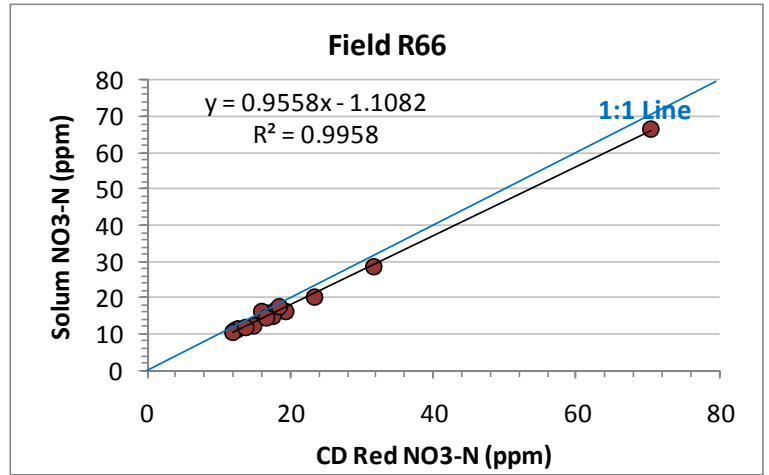


Figure 1. Field R66 soil nitrate comparison.

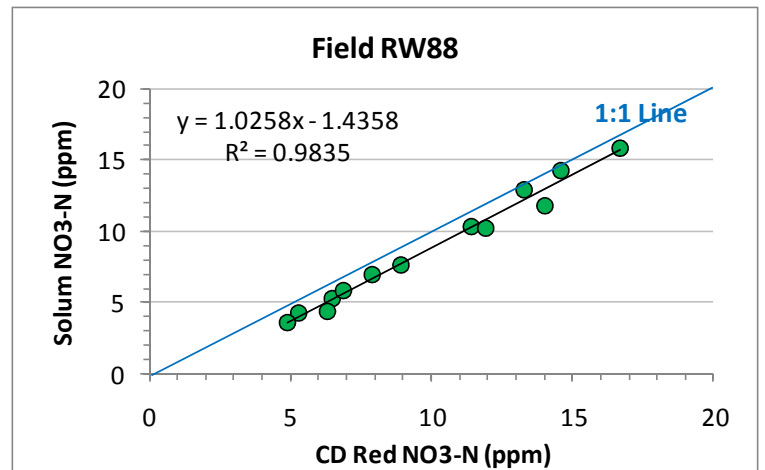


Figure 2. Field RW88 soil nitrate comparison.

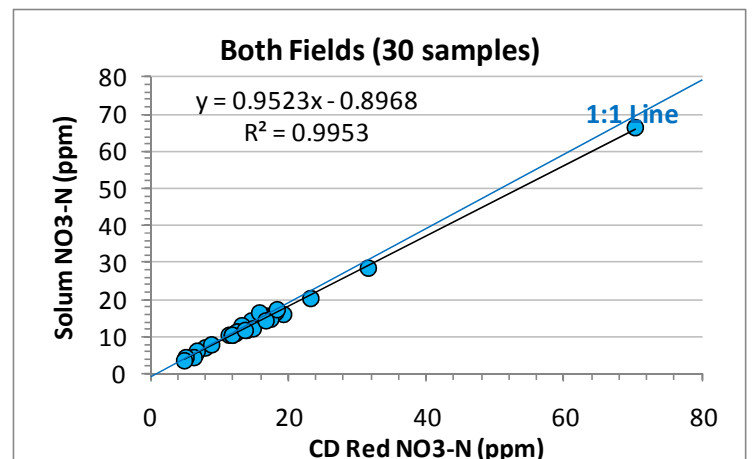


Figure 3. Nitrate thirty soil comparison.

A comparison of the thirty soils from both fields for nitrate by Cd Reduction was made to determine the precision of two duplicate runs, see Figure 4. Results show excellent duplication with an R2 of 0.999 between the two runs and an intercept of -0.13, which is only a slight deviation from zero. Although the slope is 0.9773 this is only a 3% deviation from an expected slope of 1.0. Overall these results show good reproducibility on two separate runs using different extracts.

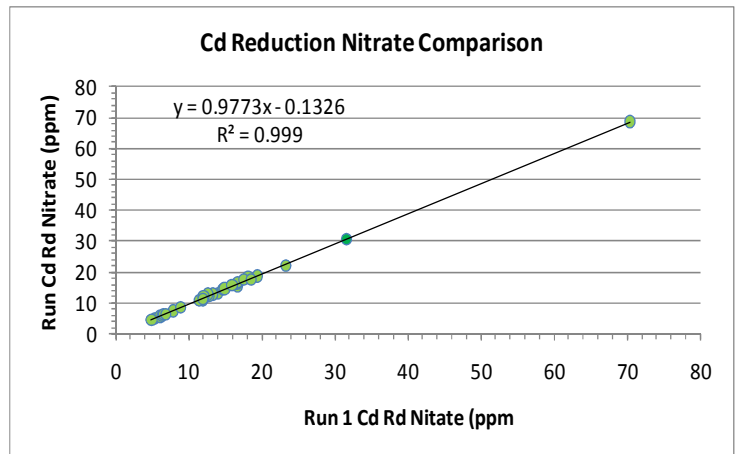


Figure 4. Cd Reduction nitrate duplicate analysis comparison.

A comparison was done to evaluate the lab precision of the Solum method of nitrate analysis to that of Cd Reduction as conducted by Ward Labs. Results indicate for the Solum evaluation (see Table 1) good precision for the three samples evaluated which range from 6.4 to 31.6 ppm. Uncertainty (based on a 95% confidence interval) was 0.24–0.26 ppm for the two low concentration samples and, 2.4 for the highest sample. A similar comparison of the same samples by the Cd Reduction method indicate a higher uncertainty on the two higher concentration samples, possible due to additional dilution factors.

Table 1. Solumn Nitrate precision three samples.

Solumn						
Sample ID	Rep 1	Rep 2	Rep 3	mean	stdev	uncertainty
#1203	6.19	6.39	6.62	6.40	0.22	0.24
#1213	17.15	17.42	17.60	17.39	0.23	0.26
#1226	29.41	31.89	33.66	31.65	2.13	2.42

Table 2. Solumn Nitrate precision three samples.

Ward Labs						
Sample ID	Rep 1	Rep 2	Rep 3	mean	stdev	uncertainty
#1203	5.50	5.63	5.33	5.49	0.15	0.17
#1213	18.22	18.85	17.25	18.11	0.81	0.91
#1226	30.80	36.40	30.40	32.53	3.35	3.80

Lastly an assessment was done to evaluate the method detection limit of the Solumn method for nitrate and compare to that for Cd reduction nitrate. The Instrument Detection Limit (IDL) was determined based on the method described by the US-EPA in Part 136, based on the standard deviation (*s*) of a minimum of seven (7) samples of soil NO₃-N, a Student's *t* 99% confidence interval. Ward labs measured the concentration of seven reps of a 3.0 ppm nitrate standard and Solum used a 2.0 ppm standard (see table 3). Statistic results show a standard deviation of 7 reps to be 0.06 for the 3.0 ppm solution and 0.16 for the 2.0 ppm, solution. Based on these results we estimate the instrument detection limit for the Cd reduction method to be 0.19 ppm and 0.50 ppm for the Solum method. Further work is need to resolve the IDL of the Solum nitrate method, such that it is equal or superior to that of the Cd reduction method.

Table 3. Stats for nitrate IDL.

	Ward	Solum
mean	3.02	1.86
stdev	0.06	0.16
IDL	0.19	0.50