### THE ILLINOIS NITROGEN SOIL TEST

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#### Introduction

Nitrogen fertilizer recommendations for corn production are often estimated on the basis of a realistic yield goal, with adjustments to allow for N credits from other sources, such as legumes or manure. A yield-based recommendation may have merit on a long-term basis, but under- or over-fertilization is apt to occur in any given growing season since soil N availability is not taken into account. Insufficient application of N can have serious economic consequences for the farmer, whereas excessive fertilization increases the risk of environmental pollution.

Public concern that excessive N fertilization may contribute to NO<sub>3</sub> enrichment of ground and surface water has stimulated interest in soil testing to improve the accuracy of N fertilizer recommendations for corn. This concern may well be justified, since crop responsiveness to N fertilization can vary widely even within the same field (Harrington et al., 1997), and nonresponsive sites have been detected throughout the north-central and northeastern U.S. (Bundy and Malone, 1988; Fox et al., 1989; Meisinger et al., 1992; Brown et al., 1993; Schmitt and Randall, 1994). For many years, a preplant NO<sub>3</sub> test (PPNT) has been used in western Canada and the Great Plains region of the U.S. to account for carryover of mineral N from previous cropping (Dahnke and Johnson, 1990; Bundy and Meisinger, 1994). Though originally developed for use in semihumid areas where leaching is limited, the PPNT has recently been applied in humid regions of the north-central U.S. to detect residual NO3 In the surface 60 cm (2 feet) of medium- to fine-textured soils (Bundy and Malone, 1988; Schmitt and Randall, 1994). To improve the reliability of NO3<sup>-</sup> testing as a basis for fertilizer recommendations in humid regions, a presidedress NO3 test (PSNT) was developed by Magdoff et al. (1984), in which soil sampling is postponed until corn is 6-12 inches tall, so as to estimate plant-available NO3 as closely as possible to peak uptake by the crop. If the test indicates a low concentration of soil NO<sub>3</sub>-N (< 20-30 ppm), supplemental N is applied as a sidedressing. The PSNT has been recommended more widely than the PPNT in the eastern U.S., but usage has been limited by the need to collect soil samples during the growing season at a time when farmers are occupied with many other tasks that cannot be neglected. Fertilization must be postponed until after testing, and this can lead to crop N deficiency if adverse weather conditions prevent sidedressing. Neither test is currently recommended in Illinois.

Numerous chemical methods have been proposed to estimate the availability of soil organic N (Bundy and Meisinger, 1994), but these have been based on an empirical approach, and their use has been very limited due to low correlations with crop N uptake and/or the production of mineral

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N during soil incubations. A more rational approach would require chemical fractionation of soil organic N to identify a labile pool; however, little progress has been made in this respect, due largely to fundamental defects in the available methodology that vitiated analyses for amino sugar-N and amino acid-N. These defects were identified and eliminated through a substantial effort that ultimately led to simple diffusion methods of fractionating the N in soil hydrolysates (Mulvaney and Khan, 2001). When the newly developed diffusion methods were applied in comparing N-distribution analyses for soil samples from sites that differed in crop responsiveness to N fertilization in a N-response study by Brown et al. (1993), a much higher concentration of amino sugar-N was observed for nonresponsive than for responsive soils (Fig. 1), whereas no consistent difference was detected in their concentrations of total hydrolyzable N, hydrolyzable NH4<sup>+</sup>-N, or amino acid-N (Mulvaney et al., 2001). In subsequent incubation studies, nonresponsive soils produced a much larger quantity of mineral N than did responsive soils, and mineralization was accompanied by a net decrease in amino sugar-N but not in amino acid-N (Table 1).

The method described by Mulvaney and Khan (2001) for determining hydrolyzable amino sugar-N was too tedious and time-consuming for routine use in commercial soil testing laboratories, so a simpler technique was subsequently developed, whereby amino sugar-N is estimated without the need for acid hydrolysis (Khan et al., 2001). The objectives of the present paper are to describe how this technique was developed, and to identify some of the factors that must be considered in using it.

# **Development of a Simple N Soil Test**

The soil samples used had been collected from the surface 12 inches in late March to early April of 1990, 1991, or 1992 from a series of N-rate experimental sites that had been characterized as responsive or nonresponsive by Brown et al. (1993). Soon after collection, the samples were airdried, ground to pass a 0.15 mm screen, and stored in a Mason jar sealed with an air-tight lid. A total of 25 samples, including 12 from nonresponsive and 13 from responsive sites, were then subjected to a series of studies in developing a simple test that would estimate amino sugar-N without the need to hydrolyze the soil for 12 h with 6 M HCl (Mulvaney and Khan, 2001). In the soil test thereby developed, a 1-g sample of air-dried soil is treated with 10 ml of 2 M NaOH in a 1-pint wide-mouth Mason jar, and the jar is gently swirled to mix the contents (but not so vigorously as to deposit some of the sample on the wall of the jar) and then sealed within 15-30 seconds by attaching a lid modified to support a small petri dish containing 5 ml of H<sub>3</sub>BO<sub>3</sub>indicator solution. The sealed jar is heated on a hot plate (i.e., a commercial griddle) for 5 hours at a temperature of 48-50°C. After removing the petri dish from the jar lid, the H<sub>3</sub>BO<sub>3</sub> solution is diluted with 5 ml of deionized water and subsequently titrated with 0.01 M H<sub>2</sub>SO<sub>4</sub>, either manually or using an automatic titrator. The micrograms of N liberated by diffusion is calculated as  $S \times T$ , where S is the volume of H<sub>2</sub>SO<sub>4</sub> used in titration of the sample and T is the titer of the titrant (for 0.01 M H<sub>2</sub>SO<sub>4</sub>,  $T = 280 \mu g$  N/ml). For complete details, see Khan et al. (2001).

In comparative studies using the 25 soil samples previously mentioned, soil N-test values were highly correlated with hydrolyzable amino sugar-N (Fig. 2). Higher test values were obtained for nonresponsive than for responsive soils, and the two groups were completely resolved assuming a critical concentration of 240 ppm (Fig. 3). Test values for nonresponsive soils ranged from 237

to 435 ppm, as opposed to a range of 72 to 223 ppm for responsive soils, which tended to be inversely related to N-fertilizer response. The former range suggests a means of estimating residual N availability, while the latter is indicative of a quantitative basis for N rate recommendations. In either case, much more research will be required to ensure adequate reliability in production agriculture.

## Diffusion timing

A 5-hour diffusion period was adopted in the N soil test, as this period was adequate to clearly distinguish between responsive and nonresponsive soils (Fig. 4), while providing stable test values for responsive soils and allowing commercial laboratories the option of analyzing two sets of samples per day. The latter option could be easily implemented, for example, if heating is initiated late in the day, and automatically discontinued after 5 hours using hot plates equipped with electrical timers. The titrations could be done the next morning, preferably after initiating another set of diffusions. In this manner, five hot plates could be utilized to process more than 100 samples within a single working day. Studies have shown very little change in soil-test N when samples are allowed to stand for several hours at room temperature following heating, whereas a substantial increase can occur when heating is prolonged beyond 5 hours, or is carried out above  $50^{\circ}C$  (Khan et al., 2001).

### Sampling depth

The N test described was developed using soil samples collected to a depth of one foot, but should be equally suitable for samples collected to a depth of 6 or 7 inches in routine soil testing for pH, P and K, since, unlike NO<sub>3</sub>, amino sugars are not subject to extensive leaching. Support for this view is provided by Table 2, which summarizes data obtained for three different depths of a responsive and a nonresponsive soil. As expected, test values decreased when samples were collected to a greater depth, but in each case, a clear distinction was observed between the two soils. An important implication of Table 2 is that the critical value for detecting nonresponsive soils will depend on sampling depth. In our work to date, this value appears to be around 240 ppm for 1-foot samples, whereas a value around 300 ppm would probably be appropriate with 6-or 7-inch samples. Further research will be required to ensure a sampling depth that provides the best possible correlation with crop response.

#### Sampling time

If the soil test described measures a form of N that mineralizes readily, test values should decrease substantially during the growing season. This is exactly what was observed for soil samples collected from three rotations in the Morrow Plots during the 1998 growing season, beginning at emergence and concluding with fall tillage (Fig. 5). Regardless of rotation, soil test values decreased after emergence, reaching a minimum at silking, when plant uptake of N would have been maximal. A subsequent increase in these values to near their original levels presumably reflects microbial assimilation of N during crop senescence and residue decomposition.

The data in Fig. 5 suggest that soil sampling for the test described can be done before or after, but not during, the growing season, although further work is needed to confirm this critical issue.

Also of interest is the fact that Fig. 5 shows a consistent difference between the three rotations studied, in that test values decreased according to previous cropping in the order, alfalfa > soybean > corn. The latter finding indicates the possibility that the soil test described may provide a quantitative basis for estimating N credits due to cropping.

Spatial variability

A field of approximately 40 acres was sampled (to 6 inches) on a 1-acre grid in the spring of 2001 to characterize spatial variability in amino sugar-N. The results are summarized by Fig. 6, which shows a range from 242 to 410 ppm, and a consistent pattern of variability relative to a barn and pasture area (not sampled) that occur in the existing farmstead. There has been no livestock production at this site for at least 17 years, but prior to that time the field sampled had received regular applications of swine manure. Surprisingly, the soil test data in Fig. 6 seem to indicate a residual effect of manuring. It is probably not coincidental that the highest test value was obtained for the area where the gate was once located. If the findings in Fig. 6 are confirmed by additional studies, the soil test described may well become a useful basis for variable-rate application of N, and may also revolutionize our understanding about the value of manure as a nutrient source.

#### Summary

A simple soil test has been developed to estimate amino sugar-N, as a means of identifying sites where corn does not respond to N fertilization. This test avoids the need for acid hydrolysis or chemical extraction, and is ideally suited for routine soil testing. Besides providing an unprecedented capability to detect nonresponsive sites, the test described may have important value for estimating how long such sites may remain nonresponsive, and may also provide a quantitative basis for N fertilizer recommendations. Further work is in progress to calibrate the test for a wide range of soils and management practices, develop appropriate protocol for soil sampling and sample processing, and ascertain the effect of climatic conditions on the mineralization of amino sugar-N. Such information is a crucial prerequisite to successful applications on production fields.

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Fig. 1. Effect of amino sugar-N concentration on relative yield response.



Fig. 2. Relationship between amino sugar-N and Illinois soil test N concentrations. Y = 98.73 + 0.56x,  $r^2 = 0.82$ .



Fig. 3. Relationship between Illinois soil N test and fertilizer N response.



Fig. 4. Effect of heating time on liberation of amino sugar-N by the Illinois N soil test procedure.



Fig. 5. Change in Illinois N soil test values over the growing season. Soil samples were collected in 1998 from the Morrow Plots, and were supplied by Dr. Michelle Wander.



Fig. 6. Variation	<u>in Illinois N so</u>	oil t <b>es</b> t across a 40-acre field.	
No sample	<300	300-350	

	Extractable Inorganic N	Amino Acid	Amino Sugar	
		mg N/kg		
3 Nonresponsive soils	123	40	-46	
2 Responsive soils	40	11	-23	

 Table 1. Net change in inorganic, amino acid, and amino sugar N following a 12-week incubation of soils differing in N fertilizer responsiveness.

 Table 2. Effect of sampling depth on N test values for soils differing in N-fertilizer

 responsiveness.

	Sample Depth, inches	Illinois N Soil Test, ppm
2 Nonresponsive soils	0-6	339
	6-12	224
	12-24	130
2 Responsive soils	0-6	160
	6-12	111
	12-24	78

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