Development of chemical tests for soil nitrate content as a factor for correlating soil and climatic properties with winter wheat (Triticum aestivum L.) yield
by Grant Dewayne Jackson

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE in Soils
Montana State University

Abstract:
Winter wheat at seven locations in the Gallatin Valley, Montana was top dressed with N in the spring of 1969.

Multiple regression analysis and correlations were made for grain yield with N fertilizer rate, soil nitrates, available soil water, pan evaporation, growing season rainfall, depth to lime and soil temperature at 50 cm.

The best correlation (R^2 = .99) was obtained with available soil water, growing season rainfall, pan evaporation, depth to lime and soil temperature at 50 cm.

Only two locations responded to N fertilizer; therefore, the multiple regression equations predict a very slight response to added N and soil nitrate.

Colorimetric analysis of nitrate in soil extracts using chromotropic acid was studied and compared to the phenoldisulfonic acid procedure.

Extracting solutions of 0.5 M $MgSO_4$, sat. $CaSO_4$, and 0.1 gm $Ca(OH)_2$/ 50 ml of water were investigated. The three extractants were equally effective in recovering NO_3. The $Ca(OH)_2$ - H_2O system was used for subsequent analyses since clear extracts were obtained.

A 1:5 soil to solution ratio and a shaking time of 15 minutes effectively extracted NO_3 and resulted in appropriate NO_3 concentration for color development.

Iron (II) and NO_2 were found to interfere at soil concentrations of 167 and 8.4 ppm respectively.

Three methods were used to compare the two procedures: 1) a nitrate recovery study, 2) linear regression and correlation analysis and 3) linear regression and correlation with winter wheat yield and response to N fertilizer. Equivalent amounts of NO_3 were recovered by both procedures. The correlation coefficient for the two procedures was 0.98**. Both procedures were similar in predicting wheat yield, but the phenoldisulfonic acid procedure produced a slightly better response correlation.

The new procedure has several advantages including rapidity, simplicity, sensitivity and relative freedom from interference.

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Date August 3, 1970

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August, 1970

ACKNOWLEDGEMENT

The author would like to express his sincere appreciation to the following people:

Dr. J. R. Sims, my major professor, for his patience and guidance throughout my graduate work;

The other members of my committee: Drs. A. H. Ferguson, R. A. Olsen and C. M. Smith for their helpful comments and suggestions;

Dr. E. P. Smith for his assistance with the statistical analysis, and Roger Stewart for his help with the computer programming;

Dr. E. O. Skogley and Mr. Glennis Boatwright for their comments and opinions;

Mr. Bud Giese and Fred Boettcher, Soil Scientists, SCS, for their assistance in describing the soils at each plot location;

My wife, Corline, for her patience and understanding.

The author also wishes to thank the Montana Wheat Research and Marketing Committee for their financial assistance.

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ABSTRACT

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INTRODUCTION

The response of wheat to N fertilizer depends upon several plant growth factors such as available soil N, available soil water, temperature, evapotranspiration, growing season rainfall, variety, disease, weed conditions, salts and alkali and possibly other variables.

In Montana, no attempt has been made to correlate more than 2 or 3 growth factors with wheat yield for the purpose of formulating N fertilizer recommendations.

The objective of this study was to check out the system necessary for measuring the growth factors in a state-wide research effort, and to develop a reliable, rapid NO_3-N soil test that can be adapted for routine analysis.

LITERATURE REVIEW

Winter wheat plant and its environment

The literature contains many examples of winter wheat response to nitrogen fertilizer. In fact, Thompson (29) attributes the increase in wheat production since 1945 to the use of nitrogen fertilizer. Schlehuber and Tucker (23) also stated that nitrogen fertilization has contributed greatly to the wheat production in every wheat producing area in the U.S. Montana is no exception. Kittams, et al. (12) reported significant yield increases at 29 locations in 1966. Other researchers have found similar results (P. L. Brown, unpublished data).

In recent years, NO_3-N levels in the soil profile are being used in formulating N fertilizer recommendations (13). The question that arises is what is the critical NO_3 level where no response to N occurs? Of course, this "gray zone" has received considerable attention. Sixty pounds of NO_3-N in six feet of soil has been quoted by one researcher (P. L. Brown, personal communication) as the level where N response is questionable. The work was done in Gallatin County, Montana. In Nebraska (7), a moderate response was noted with 67, 73 and 151 pounds of NO_3-N per six feet of soil. Similar results were reported in eastern Montana by the Soils Specialist, Montana Cooperative Extension Service (C. M. Smith, personal communication). Smika et al. (24) reported no relationship between grain yields and NO_3-N in the Great Plains states. Obviously, the prediction of wheat response with nitrate levels in the soil is not a "cut and dry" process, but it depends on other management and environment variables also.

The effect of available soil water and rainfall distribution has been well documented in the literature. Leggett (13) in Washington reported that winter wheat needed four inches of available water to produce grain and, thereafter, 5.8 bushels of grain was obtained per inch of additional available water. Smika et al. (24) also reported a positive relationship with stored available water in the Great Plains. Lehane and Staple (14) stated that five inches of available water was required before any grain was produced and each additional inch produced 3.5 - 4.0 bushels of Thatcher spring wheat in Canada. Young et al. (34) showed a significant relationship with winter wheat yield and available soil water to the depth of 122 cm in North Dakota. Eck and Tucker (9) in Oklahoma concluded that rainfall distribution was more important than total rainfall and that soil water in the spring was not indicative of yield. Apparently rainfall, rainfall distribution and intensity, and available soil water are important factors in wheat production. Apparently, the order of importance of these factors varies geographically.

Pelton (22) used a snow fence as a windbreak to study the effects of wind reduction on evaporation and wheat yield. The results were a 15-49% reduction in wind traveled, 12-23% reduction in evaporation measured from a pan, and a 24-43% increase in yield above the check. Staple and Lehane (26) studied the effect of field shelterbelts on wind velocity, evaporation, soil water and crop yield, but measured very little differences.

The effect of depth to lime on wheat production has not been well established; however, two individuals have reported a positive relationship (R. T. Choriki, personal communication and G. E. Warrington, 1967. Effect of N fertilizer, available water and soil condition on dryland winter wheat production M. S. Thesis, Montana State University, Bozeman).

The effect of deep soil temperatures on plant growth is not well defined in the literature. However, the effect of the soil surface temperature has been investigated. Burleigh et al. (5) showed that the maximum coleoptile length of several winter wheat varieties occurred when the temperature was maintained at 80°F. Lengths were decreased above and below this temperature. Stewart and Whitfield (28) reported an increase in wheat growth when the root temperature in a controlled water bath was increased from 55° to 60°F. Woolley (33) showed that the growth of spring wheat increased with increasing root temperatures of 45, 54, 67 and 80°F.

Laboratory nitrogen determination

There are several methods available for measuring or estimating available N in the soil. Estimation by organic matter content (O.M.) and Kjeldahl N are probably the oldest methods used. In recent times, initial NO_3-N and NO_3-N released after incubation are being used in predicting response to N fertilization.

The limitations of the O.M. and Kjeldahl methods are obvious in that available N is not measured directly. Smith (25) presented data to show that initial NO_3-N and NO_3-N released after incubation were better indexes of N availability on orchard grass grown in the greenhouse. Soil O.M. and total N were reported to be relatively nonrelated to N availability. Therefore, methods for determining NO_3-N will be reviewed extensively.

The phenoldisulfonic acid procedure (4) is a traditional method for NO_3-N. The procedure has several limitations such as the evaporation of soil extracts which extends the time of the analysis and, if the samples get too hot, NO_3^- may be voltalized. Another disadvantage is that the Cl ion interferes in the color development.

The NO_3-N method involving steam distillation as described by Bremner (4) apparently works quite well on soil extracts. Probably the main disadvantage is the amount of time required per sample.

In the past 20 years several colorimetric NO_3-N procedures have been proposed to replace the phenoldisulfonic acid procedure. Eastoe and Pollard (8) modified the phenoldisulfonic acid method. They showed that nitration would take place in aqueous solution at higher temperatures. The problem was that the method was not sensitive enough, and it required boiling the extracts for 3-15 minutes. Lewis (16) described a xylenol procedure but it was slower than the phenoldisulfonic acid method. Three procedures have been described for reducing NO_3^- to NO_2^- and colorimetrically measuring NO_2^-. Nelson et al. (21) used Zn-$MnSO_4$ to reduce the nitrate. Lowe and Hamilton (17) used a highly specific enzyme extracted from soybean nodules to reduce the NO_3^-. Apparently both methods work quite well and are relatively free from interferences. Middleton (18) reported a reduction procedure which used nascent hydrogen; however, the reaction is hard to control. Fisher et al. (10) reported a method for NO_3^- and NO_2^- using brucine hydrochloride for color development. The method is sensitive enough and the results agree with the phenoldisulfonic acid procedure. But the color is highly sensitive to temperature and light. A chromotropic acid procedure was developed for water analysis by West and Lyles (31). The procedure is rapid, almost free from interferences, and highly sensitive. There is no apparent reason why the procedure can't be modified for soil analysis.

A new method for NO_3-N has recently been developed. The procedure involves the use of a nitrate sensitive electrode (20) which measures the activity of the NO_3^- ion. The problem with the procedure is that anything that affects the activity of the NO_3^- will cause interference. Differential salt conditions, temperature and pH will affect the readings and calibration. Chloride and HCO_3^- anions both interfere greatly. The Texas Agriculture Experiment Station, Lubbock is using a nitrate sensitive electrode procedure, on a limited basis, for non-saline alkaline soils that do not contain gypsum or free calcium carbonate (A. B. Onken, personal communication). The staff at the Northern Great Plains Research Center, Mandan, North Dakota have been working with the probe but have found it unsatisfactory (J. F. Power, personal communication). Apparently, the procedure is hampered by calibration problems, and it is not very sensitive.

Systems for predicting response of small grains to N fertilizer applications

Collis-George and Davey (6) suggested in 1960 that the number of conventional field experiments should be restricted and replace some of them with completely instrumented experiments. They further stated that "until complete descriptions of experiments are available, the quantitative importance of environment and its interaction with fertilizer and cultivation practices cannot be determined". Since that time more research has been done to correlate soil and climatic factors with crop yield.

Leggett (13) devised one of the first systems for predicting N fertilizer additions for Washington state. He used available soil water + expected rainfall to determine maximum yield (Ym). He then figured the yield (Yn) one would expect from the initial soil NO_3-N content to the depth of six feet, and the yield expected from the N that would be mineralized (recrop yield). If Ym $>$ Yn, then additional N was added at the rate of 3 lb/bu ((Ym-Yn)3 = additional N). Perhaps this method could be improved by additional climatic variables.

Several models have been proposed by the Canadian scientists for estimating wheat production. However, those methods reviewed assumed adequate nutrient supply. Williams and Robertson (32) devised a system of predicting wheat production from soil moisture data and monthly precipitation data during the growing season. Lehane and Staple (15) correlated available soil water and rainfall during the growing season with wheat yields. Their highest R^2 value was obtained on the medium textured soils. Baier and Robertson (2) correlated yield components (kernels/head, number of heads/unit area and 1000-kernel wt) with several soil moisture variables. Highest R^2 (0.50) was obtained using five soil water variables. This method, as expected, was no better than using actual yield.

In North Dakota, Young et al. (34) showed that available soil water at seeding time to the depth of 122 cm, stored soil NO_3-N to 61 cm at seeding, precipitation from seeding to five days before harvest, and number of degree days above 21° C from five to sixty days before harvest had significant correlation with yield response to N fertilizer. The R^2 value was 0.49. This method was one of the first to successfully correlate N response to more than one climatic variable.

METHODS AND MATERIALS

Location of field plots

Seven sites were chosen in Gallatin County, Montana to provide a wide variation in rainfall and soil water. Table 1 lists the date of fertilizer application, cooperator, legal description and other pertinent data obtained from the cooperator. Figure 1 shows the approximate location of each site in the county. An attempt was made to locate the experiments on a Bozeman or Amsterdam silt loam. The soil at the Willow Creek site was classified as an Amsterdam loam. Plots were located on established stands.

Soils and sampling techniques

Table 2 contains the soil series name and other information about each site. All plots were located on soils with essentially the same parent material.

SCS Soil Scientists, Bud Giese and Fred Boettcher, described areas three, four and five, and noted the differences at all the other sites except location 6. Location six was omitted since it is the original site for the typical Amsterdam silt loam. The representative soil series descriptions and notes made by the SCS Soil Scientists are included in appendix table 16.

Two core samples for nitrate analysis and two core samples for soil moisture were taken from each location at the time the plots were fertilized. Nitrate samples were taken at 0-6 in, 6-12 in, 1-2 ft,

Table 1. Cooperator, date of top dressing, legal description, wheat variety, row spacing previous crop and fertilizer applied w/seed of winter wheat fertilizer experiments, 1969.

Location No.	Cooperator	Top-dressing date	Legal description	Wheat variety	Row Spacing	Previous[1] crop	Fertilizer w/seed		
							lb N	lb P	Source
1	V. Bates	4-21	NW¼S21T 2SR3E	Itana	7 in.	S C	24	11	24-24-0
2	A. Bates	4-21	SW¼S8T 1SR3E	Winalta	7 in.	W W	16	19	18-46-0
3	J. Kuipers	4-19	SE¼S20 T1NR3E	Winalta	14 in.	W W	14	16	18-46-0
4	J. Cooper	4-14	SW¼S28 T1NR1E	Winalta	10 in.	W W	8	5.0	16-20-0
5	J. Hamilton	4-16	NE¼S14 T2NR4E	Cheyenne	7 in.	W W	9	11	18-46-0
6	MT Ag Exp St	4-28	SE¼S7T 2SR5E	Psamont	18 in.	W W	none		
7	MT Ag Exp St Ft Ellis	5-5	NW¼S16T 2SR6E	Delmar	7 in.	B A	none		

1/ These crops were followed by a growing season fallow period prior to the crop fertilized in this study. S C = sweet clover, W W = winter wheat, B A = barley.

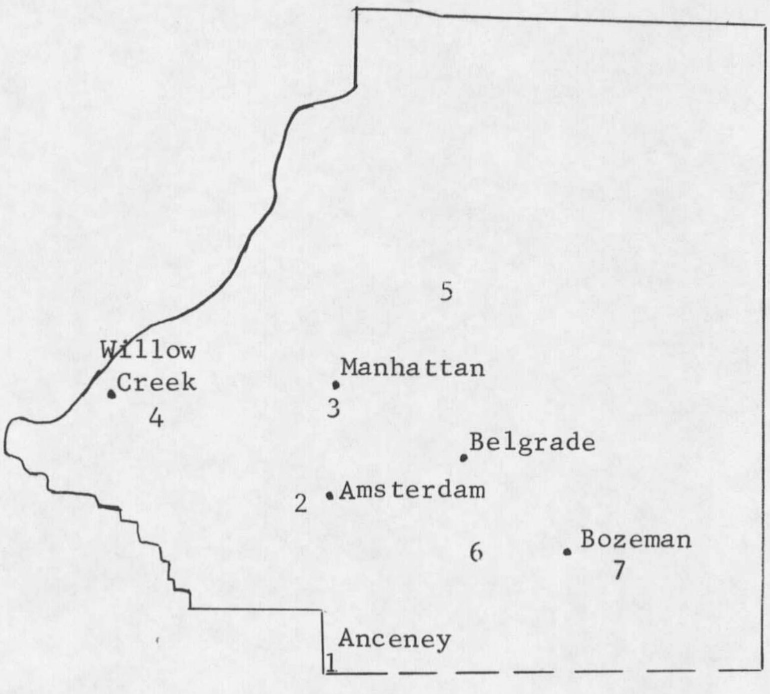

Figure 1. Approximate location of winter wheat N fertilizer experiments in Upper Gallatin County, 1969.

Table 2. Soil classification, texture, slope and aspect, and depth to lime for each location.

Location	Soil series name	Texture	Slope %	Aspect	Depth[1/] to lime
1	Amsterdam	sil	1.0	E	0 in.
2	Amsterdam	sil	2.0	E	7 in.
3	Amsterdam	sil	3.0	N	7 in.
4	Amsterdam	L	1.0	NW	0 in.
5	Amsterdam	sil	4.0	SE	7 in.
6	Amsterdam	sil	1.0	S	15 in.
7	Bozeman	sil	1.0	S	28 in.

1/ Strong effervescence with HCl.

2-3 ft, 3-4 ft, 4-5 ft and 5-6 ft depths and dried immediately. Soil water samples were taken in foot increments to the depth of six feet.

Ped samples were taken from pits at locations where an estimate of bulk density was needed. The samples were taken in each horizon.

Plot design and treatments

Each plot area was organized into a randomized complete block design with 8 treatments and 4 replications. Plot size was 7.5 ft x 40 ft. Treatments were 0, 6.7, 13.3, 20, 26.7, 33.3, 40.0 and 46.7 pounds of nitrogen per acre (lbs N/A) as ammonium nitrate. An error was made in application, therefore, the rates shown are only one-third of the amount of N we intended to apply.

The fertilizer was applied at the two leaf growth stage at all locations.

Precipitation

Rainfall was measured at each location with a Tru-Chek[1/] Accuracy rain gauge at approximately weekly intervals. Evaporation was prevented by placing a layer of mineral oil (approximately 1 cm thick) on top of 0.15 inches of water in the rain gauge. This procedure was necessary since the water would float on top of the oil if the oil was poured in the bottom of of the wedge-shaped gauge. Time of measurement was from the date of initiation of each site until August 5, 1969.

1/ Edwards Mfg. Co., Albert Lea, Minnesota

Evaporation

Evaporation was measured at each site from a No. 1 wash tub[1/] covered with a chicken wire screen. The water level in the tub was maintained between 12 and 17 cm of water to give an average diameter of the surface of about 46 cm (Figure 2). Measurements were taken at approximately weekly intervals throughout the growing season with a meter stick.

A tub was installed at both the Montana State University Campus and Agronomy farm weather stations to correlate the evaporation from the tub with the standard weather bureau pan. Readings from both stations were used in the linear regression analysis. The resulting equation is $Y = 1.12 X + .02$ where Y is the predicted evaporation in inches and X is the evaporation from the tub in inches. The correlation coefficient was 0.97 which was significant at the 1% level.

Soil temperature

Soil temperature was measured at the 50 cm depth at the same time as precipitation and evaporation were measured. This depth was chosen to minimize day and night fluctuation, and it is also the depth used by the SCS in classifying soils. Temperature was measured with a Wesson dial thermometer with an eight inch stem. Measurements were taken in the alley between replications or adjacent to the plots. The

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1/ Martin Ware C1, General Metalware Co., Minneapolis, Minnesota.