Evaluation of sulfur guidelines for major crops in Minnesota

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Introduction

Sulfur (S) is an essential element needed in the formation of proteins for all biological systems. Crop response to S fertilization was first identified in Minnesota in the 1920's. For years, S fertilization has been recommended for corn and wheat when grown on coarse-textured soils. However, recent studies found S deficiency symptoms in corn on medium and fine textured soils (Kim et al., 2013).

Sulfur deficiency is likely to occur due to a combination of several factors. Continued reduction in atmospheric SO_4-S deposition is a major detrimental factor for S nutrition. Data obtained from National Atmospheric Deposition Program showed that in 2014 in Minnesota, annual wet deposition of SO_4-S ranges from 1.7 lb per acre to 5.0 lb per acre. In contrast, S removal by corn can be several folds higher. Corn grain yield has increased steadily over the last 30 years further accelerated S removal from soil. In the past, commercial N, P, and K fertilizers contained considerable S as an impurity. Significant amount of incidental S was supplied with the application of N, P, and K fertilizers. In the recent years, advanced refinery of these fertilizers has enhanced the quality of these products but lessened the amount of S included as impurities.

Current recommendation suggests that an annual rate of 25 lb S per acre is adequate for corn and wheat production on sandy soil and can be broadcast or incorporated before planting or applied in a starter fertilizer at planting. For medium and fine textured soils with soil organic matter less than 4.0%, 10-15 lb per acre S has been suggested for corn. When organic matter concentration exceeds 4.0% there has been shown to be limited response to S fertilizer. However, there is some evidence that poorly drained soils with organic matter concentrations greater than 4.0% in major corn growing areas of West Central Minnesota may be sulfur deficient. Striping consistent with S deficiency has been noted even in fields were fertilizer S was applied. More research is needed to determine if S fertilizer should be applied for corn on high organic matter poorly drained soils.

Fall fertilizer application has economical and logistical benefits for both growers and fertilizer industry. Fall application of fertilizers saves time during the busy spring planting season. Many farmers would prefer to apply S with P and K and save a trip across the field. The question remains whether this is a good practice since SO42- can be leached. Because of the leaching potential of SO_4-S some are suggesting elemental sulfur be applied which will not leach.

However, elemental sulfur may not provide SO42- in time for peak demands early in the growing season.

Plant absorb S in the form of SO_4^{2-}. Fertilizers that supplies S in sulfate (SO_4^{2-}) form are preferred for corn production. Elemental S must be oxidized to SO_4-S before it can be available to plants. Conversion of elemental S to plant available SO_4-S depends on soil microbial activity which takes time and is slowed by cool spring temperatures. Source of S in a fertilizer program should be taken in to consideration as it can affect SO_4-S availability to corn. In the Midwest, farmers plant corn when temperatures are significantly low to be favorable for microbial activity. Therefore, it is unlikely that elemental S would be available at early-season corn growth stages. Research results in the Midwest showed that SO_4-S increased corn grain yield compared to elemental S when both fertilizers were either broadcasted or incorporated.

Grower interest when utilizing sulfur is primarily yield driven. However, sulfur is a part of proteins in the plant which can be important for crops such as spring wheat where protein is a consideration for marketing of the harvested grain. Nitrogen sufficiency is the primary driver of protein production in grain crops. Sulfur can impact N utilization thus it is possible for S to influence both yield and quality. The primary two amino acids that contain S are cysteine and methionine. Amino acids can be differentiated with chemical analysis but this process can be expensive. For soybean, NIR curves have been developed that offer quick screening of protein content and amino acids in the grain. NIR is a cost effective strategy to rapidly determine protein content in wheat but curves are not available for hard red spring wheat to concurrently estimate amino acid concentration.

Plant tissue tests are increasingly being used to determine the sufficiency of elements, such as sulfur, in major field crops. Analysis of plant tissue S content is typically accomplished through inductively coupled plasma optical emission spectroscopy (OES) which measures the concentration of multiple nutrients at one time. One issue we have noticed with ICP is that the recovery of S appears to be low compared to S measured with a combustion analyzer. It is unclear whether standard values used for determining sufficiency of S were created on an ICP, combustion analyzer, or something else. Reports of low S concentration can be questioned as to whether widespread reports of S deficiency could be due to an underestimation of the amount of S in the plant tissue due to the procedure being used. Direct comparisons of methods for analysis of S is needed on studies where crops respond to S to determine whether critical concentration of S in plant tissues varies based on the method of analysis.

Because agronomic, economic, and environmental factors are associated with fertilizer sources and application timing and no research could be found in the literature that examines the performance of fall vs. spring S applications on corn, an investigation was warranted. The objectives of this study were

1. Determine whether poorly drained soils in western Minnesota will respond to sulfur fertilizer.
2. Evaluate the efficacy of fall and spring applied elemental S and SO4-S containing fertilizer sources on corn.
3. Assess the impact of sulfur fertilizer on grain quality of hard red spring wheat
4. Develop NIR curves for hard red spring wheat to screen grain for common amino acids
5. Compare methods of detection of sulfur in plant tissue for the prediction of crop yield response to sulfur fertilizer.

Materials and Methods

Table 1 Summary of soil test data collected in Fall 2015. Samples were collected from the 0-6" and are a composite of 8 separate cores.

		Soil Test†				Sulfate-S	
Location	Soil Type	P	K	pH	OM	0-6"	0-24"
2016		--ppm--			-%-	-----ppm------	
Clarkfield	Seaforth-Canisteo	20	353	7.6	4.6	4.3	3.8
Renville	Okoboji	12	180	7.9	6.1	8.0	6.3
2017							
Clarkfield	Canisteo	18	345	7.7	6.1	35.0	91.5
Renville	Okoboji	18	234	7.4	5.7	9.0	30.1

† P, Olsen phosphorus; K, ammonium acetate extractable potassium; pH, soil pH; OM, organic matter.

Sulfur Rate and Timing for Corn: Two field trials were established using a randomized complete block with four replications in each of 2017 and 2018. Plot size was 10-11' wide by 40' long. An elemental S with bentonite mixture [Tiger 90 (0-0-0-90)] and K_2SO_4 (0-0-51-17) were compared applied at rates supplying 0, 5, 10, and 20 lb S per acre. Fertilizer is applied in the fall and spring of each year. Nitrogen and phosphorus were applied at non-limiting rates. Potassium was applied as KCl (0-0-60) to balance out K applied with the K_2SO_4.

At V10 growth stage, fully developed upper leaves were collected from 10 corn plants to analyze S concentration. At R1-R2 growth stage ear leaves were sampled from 10 corn plants and will be analyzed for S concentration. Canopy reflectance measurements will be collected using a SPAD chlorophyll meter and a Crop Circle active canopy sensor. SPAD measurements were collected at V10 and R2 while crop circle measurements were collected at V10 only. Soil samples were collected from 0-1' depth from all plots at V10 and analyzed for mono-calcium phosphate SO_4-S. Corn grain yield was measured in all plots and adjusted to 15.5% moisture content.

Spring wheat response to sulfur: Funding for the agronomic portion of this study was provided by the Minnesota Wheat Research and Communication Council. The funded study includes three locations per year where six wheat varieties are grown under three rates of sulfur (0, 7.5 and 15 lbs S per acre) applied as ammonium sulfate. Wheat flag leaf samples are collected at anthesis and grain samples are collected after harvest and analyzed for total sulfur content by dry combustion. The proposal to AFREC includes funding to cover the cost of analyzing the grain for amino acid content which is not covered in the grant to MN Wheat. Changes in amino acid content due to fertilizer application can tell us something about potential nutritional benefits from fertilizer application. In many S studies we have seen increased uptake of S in the grain but we do not know if the uptake is changing the protein composition. One other aspect we are interested in is the formation of acrylamide, which is a known carcinogen, which is partially related to the amount of asparagine in the grain. In our soybean research we have been utilizing NIR to quickly analyze protein and amino acids in the soybean grain. We are trying to develop curves for wheat to quickly screen samples. We have archived samples from 2014 and 2015 for further analysis. This data along with the 2016 funded studies would provide a large database to develop the NIR curves which can be used in future research.

Sulfur analysis methods: Samples collected from current studies and archived from previous S studies (crops studies will be corn, soybean, or spring wheat) will be analyzed by ICP-OES and combustion analysis for S. The samples selected will alll be analyzed by ICP or combustion. We are proposing to complete the analysis of archived samples for both ICP and combustion analysis of S on plant tissue taken from multiple timings. This will allow us to determine if analysis procedures need to be changed to better quantify S in tissue. We will be working with the University of Minnesota Research and Analytical Laboratory on procedures for the ICP work. Part of our interest is to determine if modifications made to the ICP to optimize the determination of S will greatly impact other nutrients. Field studies will be selected that have a measured grain yield response to S. Relative grain yield for individual plots will be correlated to S content to determine critical S concentration for each method. A correlation study will be conducted among the two methods for each plant tissue analyzed to compare the amount of S extracted for ICP versus combustion analysis.

Results and Discussion – Corn Sulfur Trials

Four field locations were established in areas where striping due to sulfur (S) deficiency has been noted in the past. Both field sites had soils which were relative high in soil organic matter [SOM (Table 1)]. Soil tests sulfate values are listed in Table 1 for the 0-6” and 0-24” depths. The values listed in Table 1 are reported in ppm and not in pounds per acre. A rough conversion to pounds per acre is multiplying ppm by 8. In when multiplying by 8, there would be a significant total of sulfate-S in the top two feet of the soil profile at both locations, but in particular Renville.

The data shown in Table 1 only account for three of the four replications at each location. Soil test sulfate-S concentration was extremely high in one of the four replications at each site. At

Renville, Replication 2 had an average sulfate-S soil test of 105 ppm (~800 lbs sulfate-S) while Replication 4 at Clarkfield averaged 147 ppm (~1200 lbs sulfate-S) both in the top two feet of the soil profile. These large quantities of S in the soil are likely attributed to a natural layer of gypsum in the soil profile near the soil surface. The area with the high sulfate-S concentration represented the lowest topographical area in the trial at Clarkfield. The field was relatively flat overall at Renville but it is likely that the high area was in a depressional area within the trial. Soil sulfate-S values were consistent among the three other replications in the field.

Soil test sulfate-S was evaluated in the top foot at the V6 growth stage. Data given in Tables 2 and 3 represent the values in ppm and do not reflect the change in soil test values from samples collected in the fall. The variation in the sulfate-S concentrations by replication made it difficult to calculate change in soil sulfate-S from the fertilizer application. Treatment differences for the soil concentrations will be used to evaluate changes based on source, rate, and timing of S application. Due to the unpredictability a difference in soil sulfate values was expected between sources. Plot treated with sulfate-S had a greater soil sulfate-S concentration at the Renville location but not at Clarkfield. Rate or timing did not impact soil sulfate-S concentrations at either location.

187	185	186	115	52	97	19	10	187
82	94	86	36	7	7	6	6	82
6	5	5	5	5	7	6	6	6
5	5	4	5	5	5	5	8	5
4	4	5	4	4	4	4	5	4
5	4	5	6	5	4	5	5	5
3	4	4	6	5	5	5	5	3
3†	4	3	2	3	5	3	2	3

Figure 1. 0-12" sulfate-sulfur soil test values for each plot at Clarkfield, MN for samples collected in June 2016. The † symbol denotes the location of plot 101 which was in the southeast corner of the study

5	6	5	6	7	7	7	8	5
5	5	6	6	6	6	6	6	5
5	6	6	6	6	6	5	7	5
5	5	4	6	6	9	7	9	5
7	7	26	113	204	237	198	126	7
6	5	6	19	63	79	78	173	6
5	4	6	9	7	4	5	7	5
6†	6	5	5	4	5	4	5	6

Figure 2. 0-12" sulfate-sulfur soil test values for each plot at Renville, MN for samples collected in June 2016. The † symbol denotes the location of plot 101 which was in the northeast corner of the study

Figures 1 and 2 highlight the spatial variation in sulfate-S concentration for both fields during 2016. High sulfate-S soil test values were isolated to specific areas of the fields. In the remaining areas the soil test sulfate-S values were relatively constant. An increase in sulfate-S was expected with the sulfate treatment and a rate response was expected. The data indicates that either the sulfate-S leached below the one foot depth and was not picked up in the soil test or that the soil test used, which is the on suggested for use in the Northcentral region, cannot be used to evaluate subtle changes in soil test values.

12	12	13	12	13	12	11	11	12
13	11	13	13	13	12	12	11	13
12	12	12	12	11	11	10	11	12
11	12	12	10	11	11	12	12	11
10	12	13	12	13	11	37	14	10
76	38	109	78	144	186	211	205	76
91	103	111	92	132	66	13	150	91
12	12	12	12	11	12	10	11	12

Figure 3. 0-12" sulfate-sulfur soil test values for each plot at Clarkfield, MN for samples collected in June 2017. The † symbol denotes the location of plot 101 which was in the southwest corner of the study

42	6	6	7	11	33	7	8	42
6	7	10	50	67	5	7	6	6
25	9	37	10	46	28	6	7	25
10	9	7	8	9	8	7	7	10
12	11	12	9	9	10	6	5	12
10	11	10	10	11	9	10	9	10
9	10	10	9	9	9	9	8	9
11	9	10	10	10	10	9	11	11

Figure 4. 0-12" sulfate-sulfur soil test values for each plot at Renville, MN for samples collected in June 2016. The † symbol denotes the location of plot 101 which was in the northeast corner of the study

Soil sulfate values by plot are presented for the 2017 growing season locations in Figures 3 and 4. Similar to 2016, the 2017 locations showed low soil sulfate levels except for in pockets of the field where there were very elevated values. The area in the plot with high soil sulfate values was more constant from side to side at Clarkfield (Figure 3) versus Renville (Figure 4).

There was no effect of sulfur source, timing, or rate on plant greenness at V10 or the concentration of S in the uppermost fully developed corn leaf at either of the 2016 locations (Table 2a). The only significant effects were for the timing by source interaction and timing by source by rate three way interactions at Renville. The three-way interaction is summarized in Table 4. It is highly likely that there are significant differences among the values listed in the table but not key trends are present where a rate, source, or timing performed better than another. The interactions are hard to explain with no significant main effects present.

Table 2b summarizes data collected at V10 from the 2017 locations. Similar to 2016, the 2017 data showed no clear evidence of an impact of fertilizer source, timing, or rate on soil sulfate-S at V10 or plant greenness (data was not measured at Clarkfield), or leaf S concentration. What was interesting is that soil sulfate S values were more elevated in 2017 than 2016 which may reflect more high values in the site shown in Figures 3 and 4. One item of note is that visually plants at the locations have shown striping in the past. From other research projects it appears that plants tend to recover starting around the V10 growth state. If that is the case, then it may not be likely that differences would be found in either leaf greenness or leaf S concentration when measuring the upper leaves. Sites like these pose an issue with determining deficiencies of S as plant tissue concentration will not likely be helpful and the suggested soil tests have seldom been shown to determine where a response will occur on fine textured soils.

Table 3a summarizes main effects for SPAD and leaf S concentration at R1 and corn grain yield data for 2016. Similar to effects at V10, there was not effect of sulfur rate, timing, or source on leaf greenness (SPAD) values. For leaf S, timing did differ were spring applied S produced a greater leaf S concentration than fall applied S. However, there was no effect of source or rate. The variation in soil sulfate-S concentration was used as a covariate in the analysis to attempt to clean the data to make it possible to detect difference among treatments. In all cases this analysis did not clean the data thus the data reported was for a standard analysis of variance procedure without any covariate analysis. In both cases for early and mid-season measurements it did not appear that a large quantity of sulfur was being taken up from the treatments applied relative to what was available from the soil.

Table 3b summarizes R1 and grain yield data from 2017. Similar to 2016 data there was no clear impact of treatments on R1 SPAD, R1 leaf S concentration, and grain yield. Lack of response for the SPAD meter readings at R1 on the leaf opposite and below the ear as well as leaf S concentration was not surprising given not effect at V10. The lack of yield response is surprising given other sulfur trials conducted at Renville which showed a response to S. One factor that could be impacting the inability to detect differences is the design of the study. The study was established using a factorial design which created 16 separate treatments. A split plot design would have been better where either source or time would have been in the sub-plot making detection of differences between treatments slightly easier.

Table 10. Summary of the Timing x Source interaction summarized for each location based on two growing season yield average (2016 and 2017) across S rates.

Timing	Source	Clarkfield	Renville
		----------------bu/ac----------------	
Fall	Elemental S	243c	226b
	Sulfate	249ab	228ab
Spring	Elemental S	253a	231a
	Sulfate	247bc	226b

Analysis of the data across years did show two major effects on yield. First, there was a significant source by timing interaction at both locations (Table 10). What was interesting is that Spring elemental S tended to have the greatest yield at both locations but fall elemental S was the least. Sulfur S did not appear to impact yield more when applied in either fall or spring. However, there was an impact of S rate when data was analyzed across all locations (Table 11). Corn grain yield was increased when 5 lbs of S was applied regardless of timing or source, but the increase was on average 3-4 bushels per acre. This slightly contradicts the data presented in Table 10 but it is consistent with data from other studies where yield was increased by a very small rate of S.

Table 11. Effect of S rate on corn grain yield across locations, S sources, and application rates.

S Rate (lb/ac)	Yield (bu/ac)
0	232b
5	235a
10	236a
20	236a

The source effects are puzzling as a response to elemental S would not be expected unless any S applied early in the sulfate form is tied up and rendered unavailable to plants. The previous statement cannot be proven but needs to be followed up with further research. It is clear that there is something affecting S availability in soils similar to what the studies were conducted. Follow up research focused on determining forms of S present in the soil would be beneficial to get a better understanding of how S cycles in soils across Minnesota.

Results and Discussion – Wheat Trials

Funding from this study included work on developing NIR equations for selected amino acids and sugars for hard red spring wheat. This work is progressing but is not yet completed. Data is being utilized from three years of a study funded by the Minnesota Wheat Research and Communication Council. The study looked at the impacts of S application on six wheat varieties at nine site years. Yield and protein response due to S were found at three site years. The remaining six site years showed no effect. The three responsive site years were all from sites conducted at Staples, MN where the wheat was grown on a sandy soil under irrigation.

Results and Discussion – S Methods Comparison

Table 12 summarizes data comparing ICP and combustion analysis of S taken from a separately funded AFREC study. Samples were collected from soybean and corn trials at different sampling dates. Sulfur concentration assessed with ICP was less than sulfur by combustion except for R2 corn ear leaf samples. For about half of the samples the relationship between the ICP and combustion was linear except for grain and R6 (end of season) samples which were quadratic. The quadratic relationship indicates that the ICP values tended to plateau even when S concentration by combustion tended to increase. Also, the recovery of S by combustion was greater for samples collected at later growth stages. This has been previously noted when analyzing grain for total S where the total S uptake was lower than anticipated. Since the majority of samples analyzed by labs use ICP it is questionable whether established normal values for total sulfur are adequate and need to be adjusted based on the sulfur analysis method.

Table 12. Summary of relationships between ICP- and combustion total S concentrations for plant and grain tissue samples from a long-term S study.

	Means		T-test	Regression			
	Comb.	ICP	P>F	Model	X_1	R^2	CL
	Soybean						
R2 Trifoliate	0.276	0.264	<0.001	Lin	0.37	0.32	--
R6 Whole Plant	0.178	0.152	<0.001	Lin	0.61	0.41	--
Grain	0.310	0.293	<0.001	Q	0.73	0.36	--
	Corn						
R2 Ear Leaf	0.134	0.159	<0.001	Lin	0.23	0.26	--
R6 Stover	0.076	0.052	<0.001	Q	0.54	0.29	--
Grain	0.107	0.088	<0.001	QP	0.67	0.31	0.18

L, linear; Q, quadratic; QP, quadratic plateau.

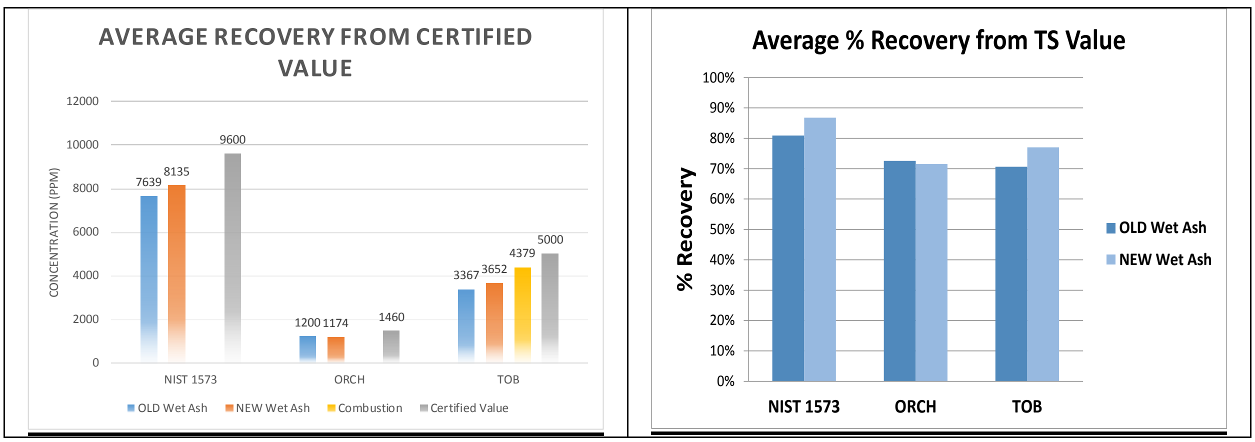

Figure 5. Comparison of new and old wet ash S analysis method compared to known standard values.

Adjustments were made to the typical wet ashing procedure used by the University of Minnesota Research and Analytical lab to determine total sulfur by ICP. The actual digestion of samples for the comparison was the same. The old method compared measured sulfur in the axial mode at a wavelength of 180.731nm. The new analysis measured sulfur in the radial mode at a wavelength of 182.034 with the addition of a yttrium internal standard. The modified method slightly improved recovery of sulfur from two of the three reference samples (Figure 5). Total recovery of sulfur was still less than 90% of the guaranteed values. Recovery did differ by reference material and was lowest for the orchard reference at near 70% of the reference value.

Sulfur by combustion was determined using a Elementar Rapid S Cube. For comparison only one of the references, the tobacco standard, was analyzed using the rapid S cube. Results from the combustion S analysis showed a greater recovery of S from the rapid S cube but the average value was still less than the S value stated by the tobacco standard.

Data for additional nutrients is not included in this report. Other nutrients were measured with and without the modified S procedure but there was no evidence that values for other nutrient varied. This indicates the S procedure could be modified without impacting other nutrients. The primary draw back to the modified procedure would be any additional cost or run time for samples. Since S recover still was not increased to 100% the modified procedure may not provide substantial enough increase over the standard procedure. Sulfur concentration would need to be related to crop yield to develop an index of response with the various procedures.

Table 13. Comparison of total S measured in corn tissue with the modified procedure versus the rapid S cube taken from an S responsive site near Staples, MN.

	Means		T-test	Regression			
	Comb.	ICP	P>F	Model	X_1	R^2	CL
V5 Whole Plant	0.19	0.19	ns	--	--	--	--
R2 Leaf	0.19	0.18	<0.01	Q	0.26	0.22	0.17
R6 Whole Plant	0.09	0.07	0.01	Lin	0.54	0.56	--
Grain	0.12	0.09	<0.001	Q	0.39	0.17	0.17

L, linear; Q, quadratic; QP, quadratic plateau.

Table 13 summarizes corn data collected from Staples. The revised ICP method still difference from combustion values except for the V5 whole plant samples. The relationship between the two values was again curvilinear for the R2 leaf and grain samples and linear for the R6 whole plant samples.

Table 14. Summary of the relationship between corn tissue S concentration measured by ICP and combustion and corn grain yield and the critical level (CL) defined as the point of maximum S concentration.

	ICP S vs Yield				Combustion S vs Yield			
	Model	X_1	R^2	CL	Model	X_1	R^2	CL
V5 Whole Plant	Q	944	0.26	0.25	Q	765	0.23	0.29
R2 Leaf	Q	1168	0.32	0.31	Q	394	0.07	0.34
R6 Whole Plant	ns	--	--	--	ns	--	--	--
Grain	Q	28743	0.25	0.10	Q	614	0.07	0.28

L, linear; ns, not significant; Q, quadratic; QP, quadratic plateau.

Table 14 summarizes the relationship between yield and plant tissue S concentrations measured by ICP and by combustion. Of the four samples collected, there was no relationship between R6 whole plant S concentration and yield for either method. The best correlation was between R2 leaf S concentration and grain yield for both methods. However, there was a slight difference in the critical level with a slightly higher critical level for the combustion measurement. The greatest difference between critical levels was for the grain measurements where the critical level for the ICP analysis was roughly half that of the combustion analysis. This is consistent with other findings and indicates that ICP analysis is likely to under predict total S removal. For the diagnostic testing periods, V5 and R2, the values were close but still different where, again, separate critical levels should be established based on analysis method and the method of analysis should be clearly stated prior to any recommendations made for S application based on tissue analysis.

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APPENDIX

Table 2a. Summary of treatment main effects for soil, sensing (normalized difference red-edge index measured with a crop circle 430), and corn newly developed upper leaf samples collected at the V10 growth state during 2016.

	V10 Soil SO_4-S		V10 NDRE		V10 Leaf S	
Effect	Clarkfield	Renville	Clarkfield	Renville	Clarkfield	Renville
	---ppm SO_4-S---				--------% S--------	
	Sulfur Source					
Sulfate-S	5.2	6.3a	0.442	0.410	0.27	0.27
Elemental-S	4.8	5.5b	0.442	0.412	0.26	0.26
	Time of Application					
Fall	5.1	6.0	0.438	0.410	0.27	0.26
Spring	4.9	5.8	0.445	0.411	0.26	0.28
--lb S/ac--	Sulfur Rate					
0	4.9	5.4	0.429	0.406	0.26	0.26
5	4.8	6.1	0.442	0.412	0.27	0.27
10	5.1	5.7	0.446	0.415	0.26	0.25
20	5.3	6.3	0.460	0.411	0.26	0.31
Stats	--*P*>F--					
Time	ns	ns	ns	ns	ns	ns
Source	ns	*	ns	ns	ns	ns
Time x S	ns	ns	ns	ns	ns	*
Rate	ns	ns	ns	ns	ns	ns
Time x Rate	ns	ns	ns	ns	ns	ns
S x Rate	ns	ns	ns	ns	ns	ns
T x S x R	ns	ns	ns	ns	ns	*

Table 2b. Summary of treatment main effects for soil, sensing (normalized difference red-edge index measured with a crop circle 430), and corn newly developed upper leaf samples collected at the V10 growth state during 2017.

	V10 Soil SO_4-S		V10 NDRE		V10 Leaf S	
Effect	Clarkfield	Renville	Clarkfield	Renville	Clarkfield	Renville
	---ppm SO_4-S---				--------% S--------	
	Sulfur Source					
Sulfate-S	38.4	13.7	--	0.337	0.25	0.21
Elemental-S	35.2	13.2	--	0.337	0.24	0.20
	Time of Application					
Fall	33.2	10.2	--	0.337	0.25	0.20
Spring	40.3	16.7	--	0.337	0.24	0.21
			--			
--lb S/ac--	Sulfur Rate					
0	24.3	14.0	--	0.335	0.25	0.20
5	45.2	15.6	--	0.339	0.24	0.20
10	41.3	15.2	--	0.336	0.25	0.20
20	35.8	9.1	--	0.339	0.24	0.21
Stats	--*P*>F--					
Time	ns	ns	--	ns	ns	ns
Source	ns	ns	--	ns	ns	ns
Time x S	ns	ns	--	ns	ns	ns
Rate	ns	ns	--	ns	ns	ns
Time x Rate	ns	ns	--	ns	ns	**
S x Rate	ns	ns	--	ns	ns	ns
T x S x R	ns	*	--	ns	ns	ns

Table 3a. Summary of treatment main effects for SPAD values and corn S concentration taken from the leaf opposite and below the ear and corn grain yield (adjusted to 15.5% moisture content) for the 2016 study locations.

	R1 SPAD		R1 Leaf S Concentration		Grain Yield	
Effect	Clarkfield	Renville	Clarkfield	Renville	Clarkfield	Renville
			--------% S--------		--------bu/ac--------	
	Sulfur Source					
Sulfate-S	56.0	52.0	0.19	0.18	248	223
Elemental-S	56.3	51.8	0.19	0.18	247	224
	Time of Application					
Fall	56.3	52.2	0.19	0.17b	248	225
Spring	56.1	51.6	0.19	0.19a	248	223
--lb S/ac--	Sulfur Rate					
0	55.4	51.6	0.19	0.18	245	221
5	56.6	51.5	0.19	0.19	249	226
10	56.0	52.8	0.19	0.17	249	225
20	56.7	51.8	0.19	0.19	249	223
Stats	--*P*>F--					
Time	ns	ns	ns	0.09	ns	ns
Source	ns	ns	ns	ns	ns	ns
Time x S	ns	ns	ns	ns	ns	ns
Rate	ns	ns	ns	ns	ns	ns
Time x Rate	ns	ns	ns	ns	ns	ns
S x Rate	ns	*	*	ns	ns	ns
T x S x R	ns	ns	ns	ns	ns	*

Table 3b. Summary of treatment main effects for SPAD values and corn S concentration taken from the leaf opposite and below the ear and corn grain yield (adjusted to 15.5% moisture content) for the 2017 study locations.

	R1 SPAD		R1 Leaf S Concentration		Grain Yield	
Effect	Clarkfield	Renville	Clarkfield	Renville	Clarkfield	Renville
			--------% S--------		--------bu/ac--------	
	Sulfur Source					
Sulfate-S	50.6	59.8	0.19	0.19	247	227
Elemental-S	50.4	59.1	0.19	0.18	248	225
	Time of Application					
Fall	50.6	59.5	0.19	0.19	246	225
Spring	50.4	59.3	0.19	0.18	249	227
--lb S/ac--	Sulfur Rate					
0	50.6	59.0	0.18	0.19	245	222
5	50.4	59.0	0.18	0.19	249	225
10	50.5	59.7	0.18	0.18	249	224
20	50.7	59.9	0.20	0.18	246	231
Stats	---*P*>F---					
Time	ns	ns	ns	ns	ns	ns
Source	ns	ns	ns	ns	ns	ns
Time x S	ns	ns	ns	ns	ns	ns
Rate	ns	ns	ns	ns	ns	ns
Time x Rate	ns	ns	ns	ns	ns	ns
S x Rate	ns	ns	*	ns	ns	ns
T x S x R	ns	ns	ns	ns	*	ns

Table 4. Summary of soil sulfate-S (SO_4-S) measured with the mono-calcium phosphate test on samples collected in June at a depth of one foot for each source x treatment x timing combination.

		2016				2017			
		Clarkfield		Renville		Clarkfield		Renville	
Source	Rate	Fall	Spring	Fall	Spring	Fall	Spring	Fall	Spring
	lb S ac^{-1}	------------------SO_4-S------------------							
Sulfate-S	0	5.7	4.2	5.0	5.7	61.5	12.5	8.5	17.8
	5	5.4	5.0	6.5	6.7	36.5	36.3	7.3	15.8
	10	4.7	5.0	6.0	6.0	12.3	24.5	8.8	15.8
	20	5.8	6.0	7.0	7.3	11.5	48.3	9.3	13.5
Elemental-S	0	5.2	4.8	5.4	5.5	29.0	18.0	9.0	9.3
	5	4.4	4.4	5.6	5.8	78.0	59.5	29.0	9.0
	10	5.3	5.3	6.3	4.7	48.3	79.3	8.5	23.8
	20	4.8	4.7	6.0	5.0	11.8	31.8	8.0	11.5

Table 5. Summary of sensing data for the normalized different red-edge index collected with a Crop Circle 430 and summarized for each source x treatment x timing combination.

		2016				2017			
		Clarkfield		Renville		Clarkfield		Renville	
Source	Rate	Fall	Spring	Fall	Spring	Fall	Spring	Fall	Spring
	lb S ac^{-1}								
Sulfate-S	0	0.410	0.393	0.280	0.240	--	--	0.333	0.328
	5	0.404	0.428	0.258	0.288	--	--	0.341	0.338
	10	0.407	0.418	0.293	0.258	--	--	0.339	0.335
	20	0.420	0.398	0.280	0.263	--	--	0.338	0.335
Elemental-S	0	0.415	0.406	0.253	0.268	--	--	0.332	0.340
	5	0.404	0.413	0.263	0.258	--	--	0.331	0.335
	10	0.413	0.420	0.263	0.243	--	--	0.332	0.334
	20	0.411	0.416	0.243	0.268	--	--	0.347	0.331

Table 6. Summary of the sulfur concentration in the newest fully developed corn leaf collected at the V10 growth stage for each source x treatment x timing combination.

		2016				2017			
		Clarkfield		Renville		Clarkfield		Renville	
Source	Rate	Fall	Spring	Fall	Spring	Fall	Spring	Fall	Spring
	lb S ac^{-1}	-------------------% S-------------------							
Sulfate-S	0	0.28	0.24	0.23	0.31	0.27	0.23	0.21	0.21
	5	0.26	0.29	0.23	0.30	0.23	0.24	0.21	0.22
	10	0.29	0.26	0.24	0.25	0.23	0.26	0.22	0.20
	20	0.28	0.26	0.31	0.33	0.23	0.25	0.22	0.23
Elemental-S	0	0.25	0.27	0.25	0.23	0.25	0.25	0.21	0.21
	5	0.26	0.26	0.31	0.24	0.25	0.25	0.20	0.22
	10	0.26	0.24	0.25	0.25	0.25	0.25	0.22	0.21
	20	0.24	0.27	0.28	0.32	0.24	0.23	0.20	0.22

Table 7. Summary of SPAD chlorophyll meter values collected from the leaf opposite and below the corn ear at the R1 growth staged for each source x treatment x timing combination.

		2016				2017			
		Clarkfield		Renville		Clarkfield		Renville	
Source	Rate	Fall	Spring	Fall	Spring	Fall	Spring	Fall	Spring
	lb S ac^{-1}								
Sulfate-S	0	55.3	55.4	51.6	50.3	50.5	51.2	59.6	57.6
	5	56.1	56.1	52.5	50.7	49.9	50.0	58.0	60.2
	10	55.9	56.0	52.4	54.8	51.5	50.6	62.1	59.5
	20	57.8	55.8	52.7	51.2	51.5	49.5	59.8	61.1
Elemental-S	0	55.4	55.3	52.5	52.3	50.4	50.2	58.9	59.9
	5	57.4	56.9	52.2	50.5	50.6	51.0	58.3	59.5
	10	56.1	56.2	52.6	51.3	50.2	49.7	58.8	59.0
	20	56.5	56.8	51.4	51.8	51.0	50.7	60.5	58.2

Table 8. Summary of corn leaf sulfur concentration for the leaf opposite and below the ear collected at the R1 growth stage for each source x treatment x timing combination.

		2016				2017			
		Clarkfield		Renville		Clarkfield		Renville	
Source	Rate	Fall	Spring	Fall	Spring	Fall	Spring	Fall	Spring
	lb S ac^{-1}	-------------------% S-------------------							
Sulfate-S	0	0.20	0.20	0.20	0.20	0.19	0.18	0.19	0.18
	5	0.19	0.19	0.19	0.19	0.18	0.17	0.19	0.18
	10	0.19	0.19	0.19	0.19	0.19	0.19	0.19	0.19
	20	0.20	0.20	0.20	0.20	0.20	0.20	0.19	0.19
Elemental-S	0	0.20	0.20	0.20	0.20	0.18	0.18	0.19	0.18
	5	0.18	0.18	0.18	0.18	0.20	0.19	0.18	0.18
	10	0.18	0.18	0.18	0.18	0.17	0.19	0.18	0.18
	20	0.20	0.20	0.20	0.20	0.19	0.20	0.19	0.18

Table 9. Summary of corn grain yield (adjusted to 15.5% moisture) for each source x treatment x timing combination.

		2016				2017			
		Clarkfield		Renville		Clarkfield		Renville	
Source	Rate	Fall	Spring	Fall	Spring	Fall	Spring	Fall	Spring
	lb S ac^{-1}	-------------------bushels/ac-------------------							
Sulfate-S	0	244	241	223	210	246	241	218	219
	5	248	255	231	223	247	259	216	218
	10	246	250	220	234	255	240	225	214
	20	253	249	230	215	249	248	223	226
Elemental-S	0	250	245	228	224	251	247	216	221
	5	245	247	223	228	248	249	218	219
	10	253	247	226	220	245	256	218	223
	20	244	248	219	226	251	250	221	223