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THE EFFECT OF SUPPLEMENTAL SULFUR APPLICATIONS
ON THE YIELD AND SULFUR CONTENT
OF MICHIGAN FIELD CROPS

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THE EFFECT OF SUPPLEMENTAL SULFUR APPLICATIONS

ON THE YIELD AND SULFUR CONTENT

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By

Anne Marie Grates

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ABSTRACT

THE EFFECT OF SUPPLEMENTAL SULFUR APPLICATIONS
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Field experiments were conducted in 1981 in seven counties in Michigan to determine the response to supplemental sulfur by field crops. The sources of sulfur used in this study were gypsum, ammonium sulfate and ammonium thiosulfate. Crops treated in the investigation included corn, soybeans, navy beans and cranberry beans.

Prospective sites were first analyzed for sulfate-sulfur in the surface soil in order to characterize them as potentially sulfur deficient. Desirable characteristics of the experimental site also included sandy soil and low organic matter content.

No significant yield increases were obtained at any of the experimental sites although yield differences were observed. Significant differences in plant sulfur content due to increased sulfur rates were observed at six out of eight sites. Significant differences between fertilizers were observed at three sites.

This thesis is affectionately dedicated to my father,

Robert J. Grates

whose example was a great inspiration.

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INTRODUCTION

Sulfur (S) is essential for protein synthesis and is involved in the activation and/or formation of enzymes including those involved in photosynthesis and other metabolic processes. Sulfur is also involved in chlorophyll development and is a constituent of essential vitamins.

Sulfur research has been performed since the 1700's, then why does S research continue today? The answer can be found in changing trends in farming and changing attitudes in general. To begin with, look at the sources of S available for plant growth. The soil, crop residues (organic matter), manure, irrigation water, rain, the atmosphere, pesticides, fertilizers and various other soil amendments all can provide S which plants utilize for their growth.

Arguments can be made which indicate why each of these sources no longer provide the amount of S they once did. Cultivation and crop uptake have decreased the amount of S originally present in the soil. In some soils, the amount of organic matter is being reduced and crop residues may not be left in the soil to decompose. Many farms have converted their operations to cash crops so no livestock manure is available to spread on the soil. Concern for the environment has led to restrictions on S emissions to the atmosphere which in turn have decreased the amount of S in rain and irrigation water. Increased use of low S fuels has played an important part in reducing S from these sources. Many of the pesticides being used today contain less S than those used in the past.

Changes in fertilizers have had a great influence on the amount of S provided to plants. Normal superphosphate (0-20-0) was the major source of P for many years. Concentrated superphosphate (0-46-0) has now emerged as the major P source in an effort to reduce transportation costs. The S content of normal superphosphate is 12% whereas the S content of concentrated superphosphate is 3%. This trend has also occurred in N fertilizers. The use of ammonium sulfate (21% N) dropped sharply when ammonium nitrate (34% N) was introduced. Ammonium nitrate contains no S while ammonium sulfate is 24% S.

Another factor contributing to the increased S requirement is the great increase in crop yields which have occurred over the years. Increasing yields place an increasingly greater demand for essential nutrients. Higher rates of high analysis fertilizer with additional attention to the micronutrients seem to solve the problem. However, S is slighted again.

With these thoughts in mind, additional S research was undertaken in Michigan. The objectives of this study were:

(1) To determine the yield response of crops to S application.

(2) To compare various S sources in their ability to provide S to crop plants.

(3) To determine the effect of S application on the S content of crops.

LITERATURE REVIEW

Sulfur has long been known to be an essential element. Initial work with S began in France using gypsum. Alway (1940) reviewed the results of this early work. Pierre, in 1768, treated clover with land plaster and was rewarded with dark green color and vigorous growth. His subsequent lecturing on these results spread the practice of plastering to England and Germany and eventually overseas. Inspired by Pierre's work, Boussingault experimented with gypsum on clover, wheat, oats and rye during the years 1841-1843. His conclusion was that Paris plaster acts usefully by adding lime to the soil. Benjamin Franklin brought the idea of plastering back to the United States after serving as Ambassador to France. In a clover field outside Washington, D. C., dark green growth spelled out "THIS LAND HAS BEEN PLASTERED".

Sufficient amounts of S were considered to be present in the soil for crop growth up to this time. Dry ashing plant material was a common practice. Comparisons of soil analysis with crop uptake always indicated more S was present in the soil than the crop would use. Recommendations for gypsum were ignored and the use of gypsum dropped. Brown and Kellogg (1915) using the Osborne method of plant analysis for their work in Iowa found that up to 90% of the S in plant material was lost by ashing. Once again the question returned. Was there adequate S in the soil to meet the requirements of the crop? Brown and Kellogg analyzed and compared a number of Iowa soils with regard to S content.

All of the soils had a higher total S content in the subsurface than in the surface and four out of five were higher in the subsurface than in the subsoil. They compared the total S values of the soil to the amount of S taken up by various crops and determined that S was sufficient for a number of years but not indefinitely. When comparing virgin to cultivated soils, they found that constant cultivation without manuring resulted in a loss of S from the soil. This agreed with work done on Kansas soils (Swanson and Miller, 1917) where cropped soils contained 40% less S than native sod. Brown and Kellogg also looked at S added to the soil by rain and lost from the soil by drainage. They agreed with previous work which said the loss of S by drainage at least equalled and probably exceeded that added to the soil in the form of rain. They recommended the application of livestock manure to insure an adequate amount of S in the soil. In the absence of manure, acid phosphates should be applied.

Duley (1916) experimented with three S sources on red clover in Missouri. A nutrient solution with $MgSO_4$, flowers of S or $CaSO_4$ (gypsum) as the S source provided all nutrients for sand culture experiments. Growth of the gypsum treated plot was initially slower but at harvest time was the best. Using soil cultures, a treatment of flowers of S alone increased the yield of clover by 50% over no treatment and when added to a complete fertilizer increased the number of nodules by 700%.

A surge of interest in the application of S to crop land occurred in the western states. Crocker (1923) reviewed work done in the previous decade in Oregon, Washington and adjoining states where increases in alfalfa yield were commonly in the range of 50 to 500% in response to S application. Percent N and percent protein also increased. In California (Lipman and Gericke, 1918), adding S and N instead of N alone greatly increased the number of stalks and heads produced on barley. The most profound effects were the result of treatment with ammonium sulfate where 14 times more total dry matter was produced compared to the control. Varied results were obtained on Idaho soils (Neidig, et al., 1923). Sulfur applications produced yield increases in alfalfa on soils from the non-irrigated part of the state but had no effect when applied to irrigated arid soils. However, application of S increased the percent S in the plant in all cases.

Work with S was also occurring sporadically in other states during this time. Kansas researchers (Swanson and Miller, 1917) delved into the movement of sulfate (SO_4^{-2}) in the soil. They concluded that SO_4^{-2} leached more readily than any other soil component. Their explanation of this was that SO_4^{-2} tends to dissolve whereas other nutrients, such as phosphate, tend to precipitate. The detrimental effects of excessive S application became evident in research performed in New Jersey (Lipman, et al., 1921). All barley plots germinated but injury became evident as the season progressed on plots that received 1000 lb/acre or more of S. Most of the plants on the 4000 lb/acre plot died before harvest. Soybeans planted after the barley showed depressed germination starting at the 1000 lb/acre rate. These adverse effects might have been related to decreased pH. Erdman (1923) found that gypsum favorably affected the growth of clover, small grains and alfalfa in Iowa. Greaves and Nelson (1925) looked at the effect of irrigation water on S content of wheat, oats and barley. Analysis of the grain indicated that increasing the amount of irrigation water increased the S content of wheat but had varied effects on oats and barley, which in general showed a decline in S content. These results were tentatively attributed to the hull on oats and barley. Researchers in Utah (Greaves and Gardner, 1929) conducted a complete inventory of the sources of S available for plant use. Soil, rainfall and irrigation waters were analyzed in additon to plant samples. It was determined that S may become a limiting factor in crop production with time.

Beginning in the 1940's, research on S increased throughout the United States. In order to better indicate the areas of S response, this discussion will continue according to individual geographical areas beginning in the forties and continuing to the present.

The Pacific states have all shown a response to S fertilizers but investigators in California have performed an extensive amount of research in this area. Conrad (1941) investigated the ability of soil to retain S. He found that compounds containing reduced forms of S were retained by soils whereas compounds containing oxidized forms of S were not. However, all but two sources of S, sodium thiocyanate and thiourea, gave a significant yield response. Extremely large yield responses were found by Conrad, et al. in 1947; three to four fold increases in legumes the first year and doubled yields of nonlegumes in the following years resulted from an application of ammonium sulfate. Conrad (1950) continued his work with S by establishing plots in five different localities distributed throughout the state. Fifty per cent yield increases occurred in all instances and doubling of yields was not uncommon. Rendig (1956) fertilized alfalfa with gypsum. This treatment increased yields for all five successive cuttings. Analysis of plant tissue indicated that most of the increase in S content could be attributed to SO_4^{-2}. Jones (1964) found winter forage yield could be significantly increased by applying 20 lb of S per acre. Moreover, residual effects of the 80 lb of S per acre treatment gave yields the following year comparable to an application of 20 lb of S. Jones and Ruckman (1966) found that 40 lb of S per acre as gypsum was sufficient for only one year whereas an equal amount of S as elemental S was sufficient for two years. Their final assessment of 6 years of data showed consistent yield increases due to S application (Jones and Ruckman, 1973).

Investigators in neighboring states obtained similar results with S application. Yield increases in alfalfa and wheat were obtained in Idaho (Jordan and Baker, 1959). Yield responses were also obtained on 11 of 16 Oregon soils (Harward, et al., 1962). Ulrich, et al. (1967) grew alfalfa in solution culture with unfiltered and smog-free filtered air as the sources of S. Significant growth increases resulted from the absorption of air-borne S. Work in Washington involving SO_2 injections into the soil resulted in significant yield increases in wheat (Roberts and Koehler, 1965). Sulfur from this source compared favorably to S from gypsum when applied at the same rate.

A number of southern states have experienced growth responses due to S application. Research results in the Carolinas included increased yield of cotton seed in North Carolina (Kamprath, et al., 1957) and significant yield increases for corn and soybeans in South Carolina but only when P was also applied (Jones, et al., 1982). Similar results with cotton in Georgia were obtained but only on a lighter, loamy sand soil (Anderson and Webster, 1959). Two Florida locations, 100 miles apart, gave significant yield increases with S application to cotton and led to speculation of widespread S deficiency (Harris, *et al*., 1945). Bardsley and Kilmer (1963) determined that the best indicator of maximum crop yields was the concentration of S in the top one foot of soil. Cotton was also studied in Arkansas (Younge, 1941). Six of 10 soil types showed significant yield reduction where S was not applied. In Alabama, it was determined that S was a necessary supplement to high analysis fertilizers caused in part by the low amounts of S brought down in rain in rural areas (Volk, *et al*., 1945). Gypsum and elemental S were found to be equally effective as S sources. However, Burmester, *et al*. (1981) failed to elicit a growth response to S applications even at high rates of N. Research on S application in Mississippi covered a wide range of crops. Intensive work by Bardsley and Jordan (1957) and Jordan and Bardsley (1958) demonstrated yield increases in clover, cotton and tobacco at a number of locations. Corn and grasses were generally less responsive to S applications. Work by Bardsley (1960) indicated that gypsum was a very good source of S although cystine and methionine in solution culture also performed well.

Sulfur studies in the midwest states were generally over short term periods and in some cases failed to demonstrate a need for supplemental S. In Indiana, alfalfa did not respond to S fertilizer (Bertramson, *et al*., 1950). Ohio researchers received a 10% increase in sugar beets and alfalfa with gypsum applications (Barnes, 1956). They also determined that after 6 or 7 years of applying high analysis fertilizer, supplemental S was needed for optimum growth. Six of 8 Wisconsin soils gave a significant increase in alfalfa yield when treated with K_2SO_4 and gypsum (Rand, *et al*., 1969). The two sources were equally effective in providing S. Rehm and Caldwell (1968) determined that only the north central and northeastern sections of Minnesota have a low S supplying capacity as determined by plant uptake. Statewide applications of S were not necessary. Sulfur treatment tripled the yield of alfalfa (Seim, et al., 1969) while increasing the S and N content of corn and alfalfa but decreasing the P content (Caldwell, et al., 1969).

Initial work in Michigan by McCool (1920) and McCool and Millar (1924) failed to demonstrate a need for supplemental S. Slight yield increases occurred but were negligible. Cressman and Davis (1962) did not find a yield response from S applications however, potatoes and red clover did exhibit an increase in S content. Beaton and Fox (1971) performed field trials and conducted a tissue survey in southern Michigan. They concluded that many of the soils contained inadequate amounts of S for top corn production. Of the soil samples analyzed, 79% were rated as S deficient with another 13% declared borderline. Extensive research by Robertson and Vitosh (1974) failed to demonstrate a significant yield increase after S application even where soil tests indicated S was low. Janssen and Vitosh (1974) determined that S increased N fixation by almost 60%. The only yield response from S application in Michigan, from dark red kidney beans, was reported in this work. Robertson, et al. (1975) conducted a survey of SO_4^{-2} levels in the plow layer of soils throughout the state. Unexpectedly low levels in some instances led to the suggestion of the need for additional research on the use of S containing fertilizers.

Plant response to S fertilizers was not the only facet of S research occurring in the 1900's. During the same time that gypsum began to lose some of its popularity as a soil amendment, interest began to rise in regard to air pollution. Sulfur dioxide is a component of air and it was known that rainfall captured some of this S and deposited it on the soil. Research was conducted on the ability of plants to absorb SO_2 from the air and the amount of S deposited by rainfall.

It was known that pollution centered around industrialized areas and its main source was the burning of coal. Pollution was most severe downwind from the source. Crowther and Stewart (1913) identified SO_2 as the component of air pollution causing damage to vegetation near the industrial city of Leeds, England. Rainwater analysis indicated that S concentrations were heaviest to the east of the city due to the prevailing westerly winds but were not as high in rural areas where no damage to vegetation was observed.

The scientific community was deluged with reports of S in rainwater during the 1920's. Ithaca, New York began monitoring S in rainwater in 1918 (Wilson, 1921). Over two years, an average of 26 lb of S/acre/year was supplied to the soil through rainwater. Subsequent publications by Wilson (1923, 1926) provided additional data on S in rainfall. In 1923 it was found that a higher concentration of S in rainfall during the winter months provided 60% of the total addition to soil. A slight but consistent increase in the concentration of S in rainwater over a five year period had increased the average yearly deposition to 29 lb of S/acre. The 1926 data showed an increase in the S concentration in rainwater reflecting the opening of a heating plant.

Similar experiments were conducted in other states during this time although without the dedication of Wilson. Erdman (1922) collected precipitation at the Iowa Agricultural Experiment Station and concluded that monthly depositions of S were fairly constant and that in general, S supplied to rural communities through rainfall was about 15 lb/acre/year. In Tennessee (MacIntire and Young, 1923), ten locations were monitored to determine the amount of S in rainfall. Yearly averages ranged from 232 lb/acre to 13 lb/acre of soluble SO_4-S. Distance and direction from industrialized areas played an important role in the amount of S collected. In Kentucky (Johnson, 1924), the average annual amount of S in rainfall was 35 lb/acre. Seasonal variation of the S content of rain indicated that winter rainfall has a higher S content than summer, agreeing with previous reports.

After a ten year lull, the next paper to be published on atmospheric S indicated that only a small area of Minnesota was in need of supplemental S (Alway, et al., 1937). They maintained that crops received adequate S due to rainfall and direct absorption of SO_2 from the air based on calculations of crop uptake and measurements of S in rainfall and SO_2 in the air. Even when crop uptake exceeded the amount of S supplied by these sources, supplemental S did not result in a yield increase. They concluded that atmospheric S was sufficient for even maximum crop yields.

An extensive study by Eriksson (1952) monitored precipitation in 14 states. The amount of S deposited annually by rain ranged from a high of 260 kg/ha (232 lb/acre) in Tennessee to a low of 3.2 kg/ha (2.9 lb/acre) in Alabama. The average of the midwest states excluding Chicago, Ill. and Gary, Ind. was 29 kg/ha (26 lb/acre). Again, the increase in winter deposition due to increased coal consumption was noted.

A number of states have since monitored rainfall individually. Leland, in 1952, released data from New York covering 18 years. Average yearly depostion of S was 48 lb/acre. This was an increase of 22 lb/acre when compared to earlier work and was attributed to an increase in rainfall. Kentucky reported a statewide annual average of 12.6 lb/acre of S from precipitation (Seay, 1957). Wisconsin researchers measured an overall average of 30 kg/ha (27 lb/acre) of S deposited from precipitation annually (Hoeft, et al., 1972). They noted that S emissions from industrial sources could not be measured beyond 10 km (6.2 mi) from the source. Although this amount of S would be sufficient for crop growth, it was possible that some of it was lost since much of the S deposition occurred in the winter and was subject to runoff if spring rains came before the soil thawed. Tabatabai and Laflan (1976) determined that 12 to 15 kg/ha (11 to 13 lb/acre) was deposited annually in Iowa. This was the same amount as was measured 50 years ago. Cressman (1961) analyzed precipitation in five counties in Michigan over a two year period. Depositions of elemental S ranged from 7.5 to 13.1 lb/acre annually. Richardson and Merva (1976) in Michigan determined that rural areas received an average of 18 kg/ha (16 lb/acre) of S annually and urban areas received 68 kg/ha (61 lb/acre). Sulfur content of Michigan river water ranged from 4.5 to 75.6 lb/acre foot (Robertson, et al., 1976). The average of 13 rivers was 33.5 lb of S/acre foot. The Sulphur Institute analyzed river water in the western United States (1975). The average S content was 50 lb/acre foot. The S content was lowest at the source while drainage from agricultural areas increased the S content downstream.

Sulfur in precipitation is an important source of this essential element however, SO_2 in the atmosphere is also available for plant use. Attempts have been made to determine the amount of SO_2 in polluted and unpolluted environments. Measurements in Hawaii and Florida detected 0.7 mg/m^3 (.266 ppm) of S as SO_2 in an unpolluted atmosphere (Eriksson, 1963). Junge (1960) measured up to 3 mg/m^3 (1.14 ppm) of S in a polluted atmosphere. Fried (1948) used radioactive S as a tracer to determine the fate of atmospheric S in the plant. He concluded that SO_2 from the atmosphere can be taken up by plants and converted to organic S compounds. The amount of SO_2 utilized is dependent on the S status of the plant (Olsen, 1957). Healthy cotton plants obtained 30% of their S from the atmosphere whereas S deficient plants absorbed 50% of their S as SO_2 from the atmosphere. Other researchers reported that plants in the greenhouse obtained as much as 73% of their total S from the atmosphere (Hoeft, et al., 1972). Maugh (1979) also came to this conclusion but SO_2 in the atmosphere was inadequate as the sole source of S.

Sulfur dioxide can enter leaf stomata directly or be absorbed by moisture on the leaves (Terman, 1978). However, the primary factor controlling SO_2 absorption by plants is the degree of stomatal opening (Spedding, 1969). High humidity will open the stomata more to allow increased respiration. Other factors increasing the absorption of atmospheric SO_2 by vegetation include increased wind velocity above the plants, height of the canopy and temperature (Hill, 1971).

EXPERIMENTAL METHODS

Field Studies

Sites for the field studies were chosen on the basis of a preliminary analysis of the surface soil for sulfate-sulfur (SO_4-S). In addition to having the surface soil contain less than 8 ppm SO_4-S, most of the sites also fulfilled the additional requirements of being sandy and low in organic matter. Sites were selected in seven counties in Michigan (Table 1).

A randomized complete block experimental design with four to six replications per site was used. The three sources of S used were ammonium thiosulfate (12% N, 26% S), ammonium sulfate (21% N, 24% S) and gypsum (16% S). Additional nitrogen fertilizer was added in order to prevent nitrogen from being a variable. All experiments were performed in 1981.

A short description of each site and the treatments applied to them follows.

Soybeans in Barry County were planted with a drill in seven inch rows. Ammonium sulfate was the S source and the treatments were 0, 15, 30 and 45 pounds of S per acre and was not incorporated. To distinguish between an N response and an S response, nitrogen only plots were established with rates of N equal to that provided from the S source. Ammonium nitrate (34% N) was used at 0, 13, 26 and 39 pounds of N per acre with no additional N fertilizer provided by the farmer. No

Table 1. Location of sites, soil series and crop(s) grown.

County	Location from Lansing, MI	Soil series	Crop grown
Barry	35 miles west	-----	soybeans
Branch	70 miles southwest	-----	corn
Ionia	25 miles northwest	Miami-Owosso	navy beans
Jackson	35 miles south	Arkport	corn
Montcalm	40 miles northwest	Montcalm-McBride	cranberry beans
Saginaw	45 miles northwest	Charity	corn, soybeans
Washtenaw	30 miles southwest	Spinks	corn

irrigation was used. The experiment was replicated six times at this site but yields were harvested from only two replications.

Corn at the Branch County site was planted in thirty inch rows and was irrigated with a center pivot system. Gypsum and ammonium sulfate were applied as 0, 15, 30 and 45 pounds of S per acre and was plowed down. Urea (46% N) was added to the gypsum treatments to provide N at the same rate as provided to the ammonium sulfate treatments. Where ammonium sulfate was used at the rate of 45 pounds of S per acre, 39 pounds per acre of N was added. All treatments were brought to this level of N by the addition of ammonium nitrate. The farmer supplied an additional 220 lb/acre of actual N. The experiment was replicated five times.

Navy beans at the Ionia County site were planted in thirty inch rows and were irrigated with a center pivot system. Ammonium sulfate was applied at 0, 15, 30 and 45 pounds of S per acre and was not incorporated. Nitrogen only plots were again used at rates of 0, 13, 26 and 39 pounds of N per acre from ammonium nitrate to compensate for the N added from ammonium sulfate. The farmer supplied an additional 50 lb/acre of actual N. The experiment was replicated six times.

Jackson County corn was supplied with S from both ammonium sulfate and gypsum at 0, 15, 30 and 45 pounds of S per acre and was incorporated only by the action of the planter. Ammonium nitrate and urea were added to establish a uniform rate of N of 39 pounds per acre as was discussed for Branch County. The farmer supplied an additional 131 lb/acre of actual N. The row spacing was thirty inches and no irrigation was used. The experiment was replicated six times.

Cranberry beans in Montcalm County were planted in twenty-eight inch rows. Sulfur was provided as gypsum and ammonium sulfate at 0, 15, 30 and 45 pounds of S per acre and was incorporated with secondary tillage implements. Ammonium nitrate was added to the plots to provide uniform N application over the entire site. The farmer supplied an additional 100 lb/acre of actual N. Irrigation was provided through a travelling gun system. The experiment was replicated six times.

Corn in Saginaw County was planted in twenty-eight inch rows and received applications of gypsum and ammonium sulfate at 0, 15, 30 and 45 pounds of S per acre and was incorporated with a harrow. Ammonium nitrate and urea were used to standardize the N rate. A total of 150 lb/acre of actual N was applied. No irrigation was used. The experiment was replicated four times.

Soybeans in Saginaw County were planted in twenty-eight inch rows. The source of S was ammonium thiosulfate with rates of 0, 5, 10 and 20 pounds of S per acre. A nitrogen solution (28% N) was used to make the N rates constant across the site. An additional 25 lb/acre of actual N was supplied to the crop in a band to the side and below the seed. Irrigation was not used. The experiment was replicated four times.

Washtenaw County corn was planted in thirty-eight inch rows. Ammonium sulfate and gypsum were applied at 0, 15, 30 and 45 pounds of S per acre and was not incorporated. Urea and ammonium nitrate were used to make the N rates uniform over the entire site. The farmer supplied an additional 185 lb/acre actual N. A center pivot irrigation system was used. The experiment was replicated five times.

Sulfur fertilizer was hand broadcast at all locations except at the Saginaw County site where soybeans were planted. Application was made either shortly before or shortly after planting. Soybeans in

Saginaw County received a banded application of ammonium thiosulfate to the side and below the seed at planting time.

Plots were observed throughout the growing season. Visual differences among the plots were noted on only two occasions. On August 20, 1981 it was possible to pick out the zero pounds per acre of S plots (check plots) at the Branch County site. Husks and stalks were yellow and the leaves showed a mild striping. When the Jackson County plots were visited to collect tissue samples (July 24, 1981) it was noted that the check plots exhibited a general yellowing with some striping. However, these differences had disappeared by the time the plots were checked on August 20, 1981.

Plant tissue samples were collected and analyzed for total S. The leaf below the ear leaf of corn (referred to as ear leaf in figures and tables) was sampled at tasselling. Bean crops were sampled during early flowering, taking the most recently matured trifoliate. Yield data was collected at the end of the growing season. Dates at which plant tissue samples were collected and harvest occurred are found in Table 2.

Laboratory Studies

Once the site had been characterized as potentially sulfur deficient, soil samples were collected to a depth of 40 to 42 inches at all locations except Saginaw County. Samples were dried in a forced air dryer and crushed to pass a 10 mesh sieve. Sulfate-S analysis was performed by extracting the soil with a solution of 500 ppm phosphorus in 2 *N* acetic acid (Liegel, *et al*., 1980). Charcoal was added to the soil-extract mixture to clear the solution. After filtering the mixture, equal volumes of the filtrate and a barium chloride-gum

Table 2. Plant tissue sampling and harvest dates.

County	Crop	Plant tissue	Yield
Barry	soybeans	7/31/81	10/31/81
Branch	corn	7/24/81	10/29/81
Ionia	navy beans	7/15/81	9/23/81
Jackson	corn	7/24/81	10/13/81
Montcalm	cranberry beans	7/14/81	9/9/81
Saginaw	corn	7/20/81	10/7/81
Saginaw	soybeans	7/9/81	10/11/81
Washtenaw	corn	7/17/81	10/8/81

arabic-acetic acid solution were transferred to a test tube. Air was bubbled through the solution to assure uniform mixing. The turbid solutions were analyzed on a Bausch and Lomb spectrophotometer at 420 nm (millimicrons) and compared to standard solutions. Surface samples were analyzed for organic matter (O. M.) content on a Leco carbon analyzer. Soil pH was determined from a 1:1 soil to water solution on a Sargent-Welch pH meter.

A nitric-perchloric acid digest was used to analyze the plant samples for total S (Blanchar, et al., 1965). An aluminum heating block with a fume hood and scrubber was used for the digestion. After digestion, an aliquot of a salt buffer solution was added to the sample and brought to a constant volume. Barium chloride crystals were added and the solution was stirred. The turbid solution was analyzed on a spectrophotometer as above.

For complete analytical procedures, see Appendix A.

RESULTS AND DISCUSSION

Soils

Results of the soil analyses are displayed in Table 3. All the experimental sites except Branch County have higher SO_4-S concentrations in the subsurface than in the surface. Humid region soils may contain large amounts of hydrated oxides of iron and aluminum. Sulfates are adsorbed by these oxides and may accumulate in the illuviated horizon (Beaton, et al., 1974). However, this situation is found more often in tropical soils and may not be a factor in the soils involved in this study. Also, SO_4 is a very mobile ion and may easily leach from the surface soil since most of the sites were sandy. Both of these factors can contribute to an increased SO_4-S concentration in the subsurface.

Use of a soil SO_4-S analysis as a reliable index of S available for plant growth has been questioned. This uncertainty ranges through all phases of the analysis, from sampling the soil to the actual chemical reaction that occurs during the analysis. To begin with, how deep should the sample be taken? Plant roots are not restricted to the upper eight inches of soil, a common sampling depth for soil analysis. Roots may tap the SO_4 reserves of the subsurface to fulfill the S requirements of the plant. The SO_4 ion is also very mobile; its concentration in the soil is dependent on the relative amounts of rainfall and evapotranspiration. Sampling the surface of a sandy soil shortly after a heavy rain will not accurately reflect the S status of

Table 3. Soil analysis data for each experimental site.

County	Depth (inches)	ppm SO_4-S	pH	%O.M.
Barry	0-9	6	6.1	3.02
	9-18	9	6.9	
	18-27	23	7.9	
	27-40	12	7.5	
Branch	0-8	2	6.6	3.72
	8-16	2	5.9	
	18-27	1	5.6	
	27-40	2	5.5	
Ionia	0-9	4	5.9	1.95
	9-18	14	5.6	
	18-27	10	6.0	
	27-40	11	5.9	
Jackson	0-8	4	6.0	1.88
	8-18	8	5.8	
	18-30	14	5.6	
	30-42	12	5.2	
Montcalm	0-9	3	6.3	1.95
	9-18	8	5.8	
	18-27	12	5.6	
	27-40	10	5.6	
Saginaw				
corn	0-8	3	7.7	4.30
soybeans	0-8	0	7.7	4.30
Washtenaw	0-9	2	6.4	1.48
	9-18	2	6.2	
	18-27	3	6.2	
	27-40	5	5.9	

that soil.

Analysis involves extracting the soil with a calcium phosphate extraction solution. Sulfate is not as strongly bound as PO_4 and is replaced by PO_4. However, PO_4 is present in excess and the extent to which it interferes with the $BaCl_2$ reaction is not known. The $BaSO_4$ precipitate which is formed results in the solution turning milky, a condition referred to as turbid. Variations in temperature are known to affect the amount of turbidity produced. Retaining homogeneity of the turbid solution also may be difficult as the $BaSO_4$ precipitate eventually settles out of solution. The problems and technique involved in analysis of soil samples for SO_4-S resulted in a colleague describing the analysis as more of an art than a science.

Yields

Statistical analysis of the yield data determined that no significant yield increases were obtained at the 5% level at any experimental site. Tables of means for the interaction effects between rate of S applied and source of S are presented in Appendix B. Although yield differences between treatments were observed, neither the main effects nor the interaction effects were significant.

There were a number of instances where the zero treatments had the highest yield or had only one yield unit less than the highest yield for that site. This situation was observed for both gypsum and ammonium sulfate in Montcalm County, gypsum on corn and ammonium thiosulfate on soybeans in Saginaw County and ammonium sulfate in Washtenaw County. These sites were four of five which tested the lowest in SO_4-S concentration. This is additional evidence pointing to the lack of a reliable soil test for S.

Plant S Content

Figures 1 through 8 are graphical representations of the effect of increasing rates of S or N plus S on the S content of the plant. Tables of the plotted data are presented in Appendix C.

Before beginning the discussion of each site, it should be pointed out that none of the recorded plant S content values for corn, navy beans or soybeans indicate a deficiency in the plant. Sufficiency ranges have been established for these three crops (University of Wisconsin, 1980). The sufficiency range for corn is from .16 to .50 %S and for soybeans and navy beans, the range is .21 to .40 %S. No data was available for cranberry beans.

There was no significant difference in plant S content due to application of ammonium sulfate when compared to ammonium nitrate in Barry County (Figure 1). Only at the highest rate of ammonium sulfate did plant S content exceed that of plants treated with ammonium nitrate. Significant differences were observed in plant S content due to the increasing rate of application when averaged over both fertilizers. With no differences between fertilizers, this response seems to be due to increasing N rate.

There were no significant differences in plant S content when comparing S sources in Branch County (Figure 2). Gypsum and ammonium sulfate are essentially equal in their ability to provide S to the plant. There is an increase in S content at the 45 lb of S/acre rate averaged over both sources however this increase is not statistically significant.

Even with low SO_4 levels to a depth of 40 inches, S application had no effect on plant S content. The site was sandy and low in organic

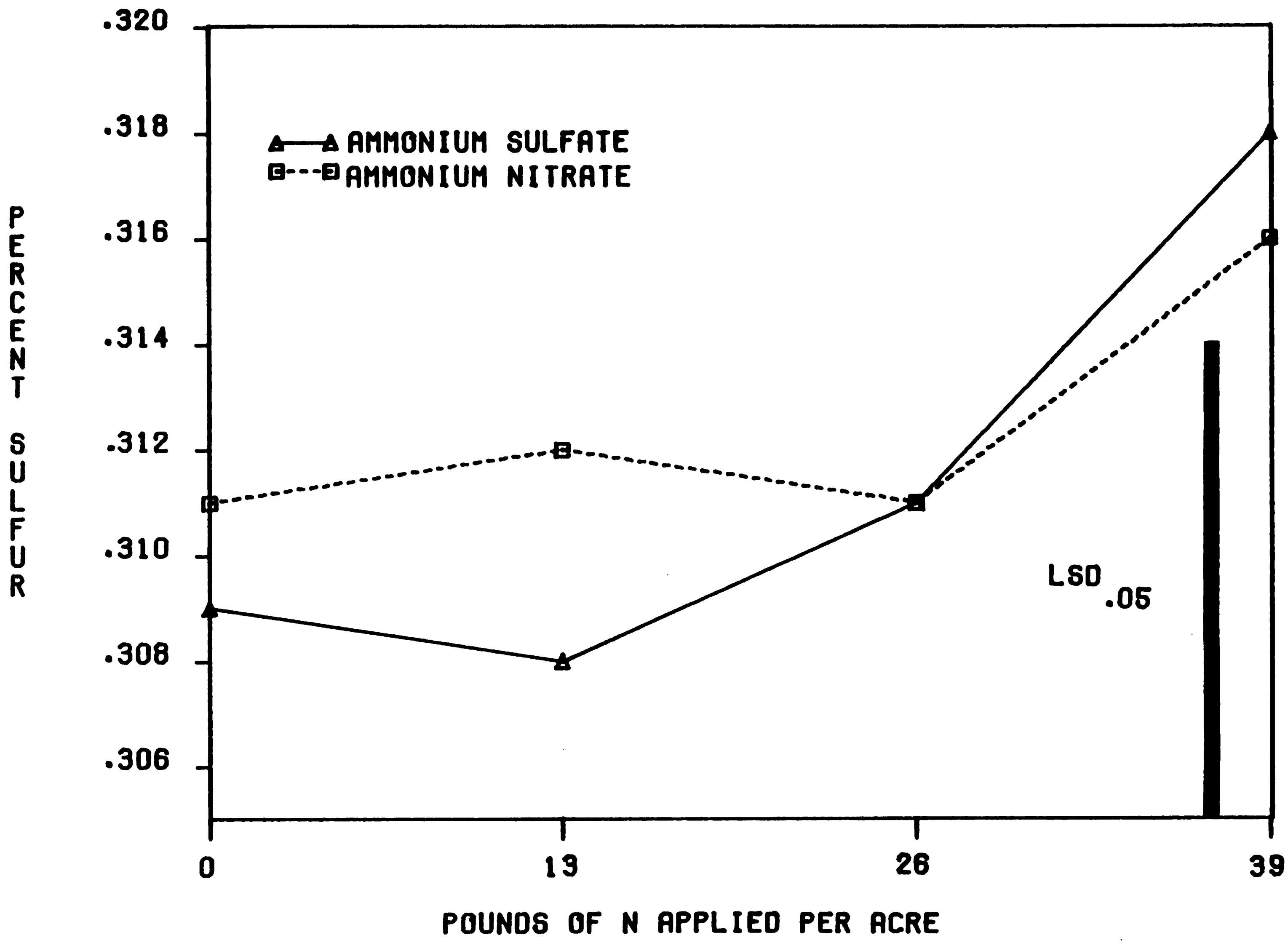

Figure 1. Effect of N and N plus S application on leaf tissue S content of soybeans in Barry County.

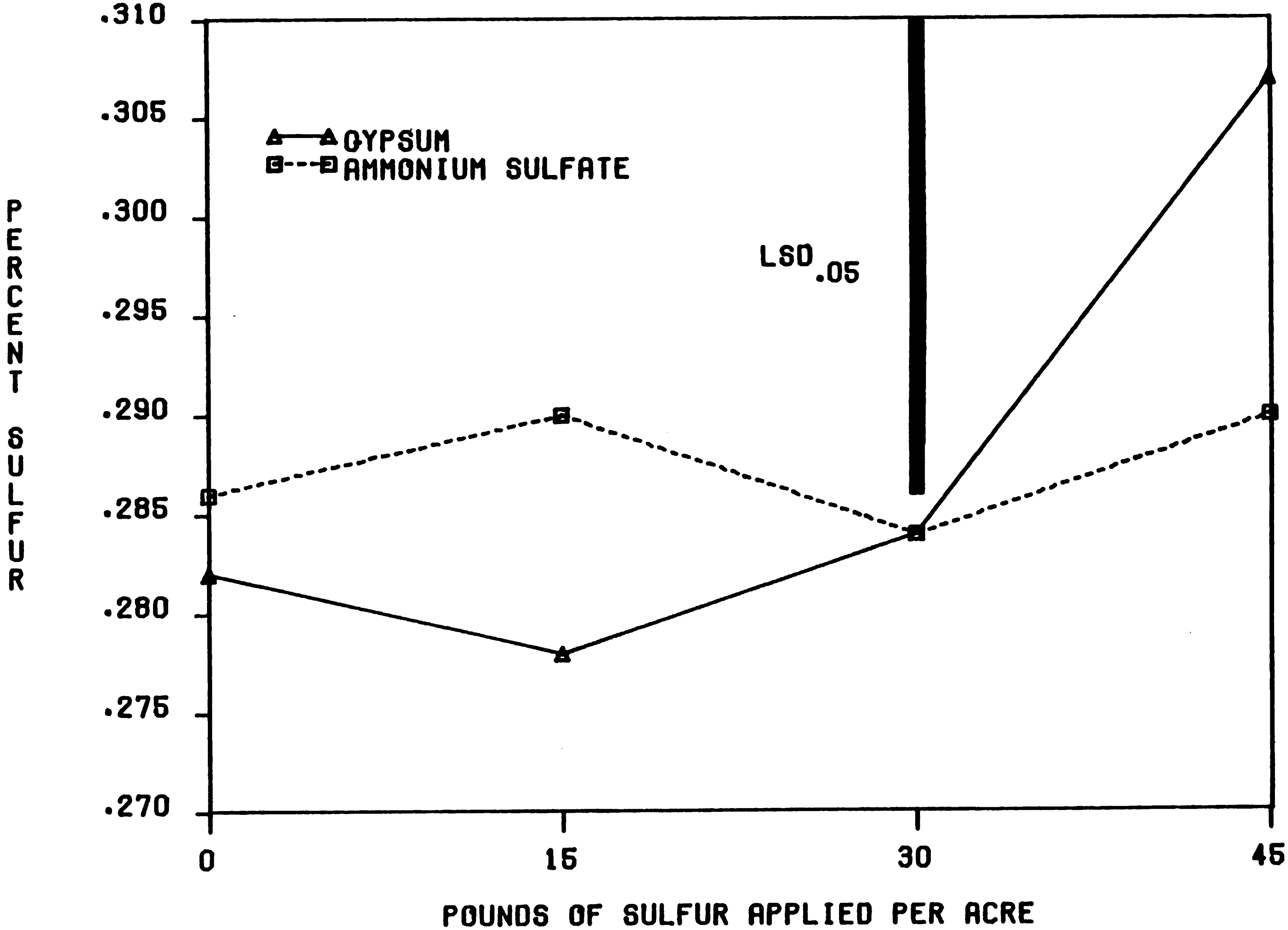

Figure 2. Effect of S application on ear leaf S content of corn in Branch County.

matter so leaching and lack of mineralization would be expected to contribute to a S response. Two factors may play a part in explaining the lack of response. This site was irrigated. Sufficient S may have been provided through the irrigation water. This site was also in a northeasterly direction from Gary, Ind., a highly industrialized city. Winds from the southwest may have blown S emissons from Gary toward Branch County. High levels of sulfur dioxide (SO_2) in the air and high SO_4 levels in rainfall could have contributed sufficient amounts of S for adequate plant growth.

The presence of S in ammonium sulfate did significantly increase the S content of navy beans when compared to ammonium nitrate in Ionia County (Figure 3). Although there was an increase in S content, this did not result in a significant yield increase. An increase of the S content of the plant without a corresponding increase in yield is indicative of luxury consumption, which is known to occur with S in plants. Increasing the rate of fertilizer did not have a significant effect on the S content of the plant.

Both gypsum and ammonium sulfate showed significant increases in plant S content with increased rates of S application in Jackson County (Figure 4). Ammonium sulfate was consistently a better source of S than gypsum. Again there was not a yield increase even though the plant content increased.

Significant increases in plant S content due to increasing rates of S application were evident in Montcalm County (Figure 5). Although there were differences between gypsum and ammonium sulfate as sources of S, these differences between sources were not significant. Again there was no yield increase even though there was an increase in plant S

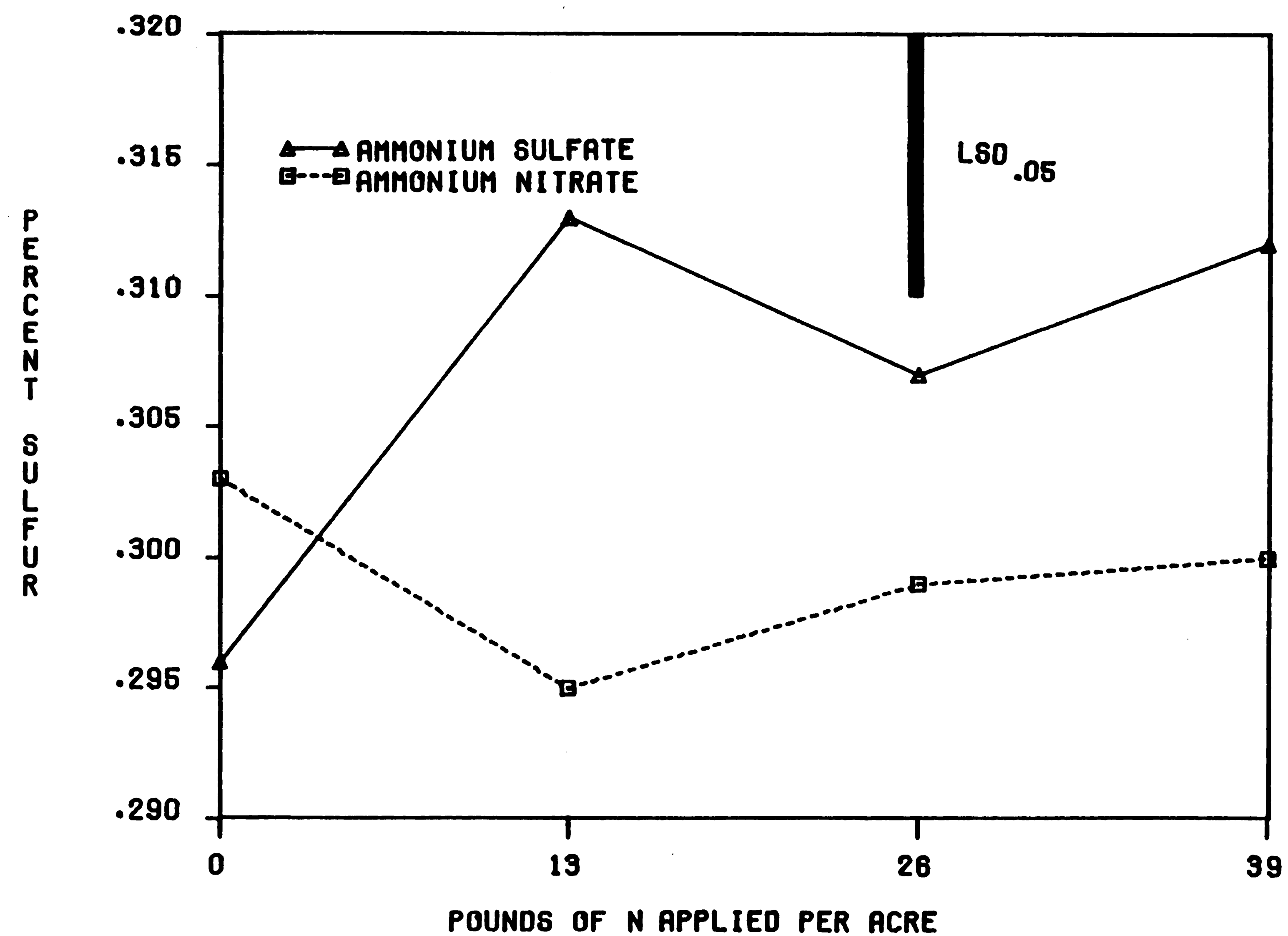

Figure 3. Effect of N and N plus S application on leaf tissue S content of navy beans in Ionia County.

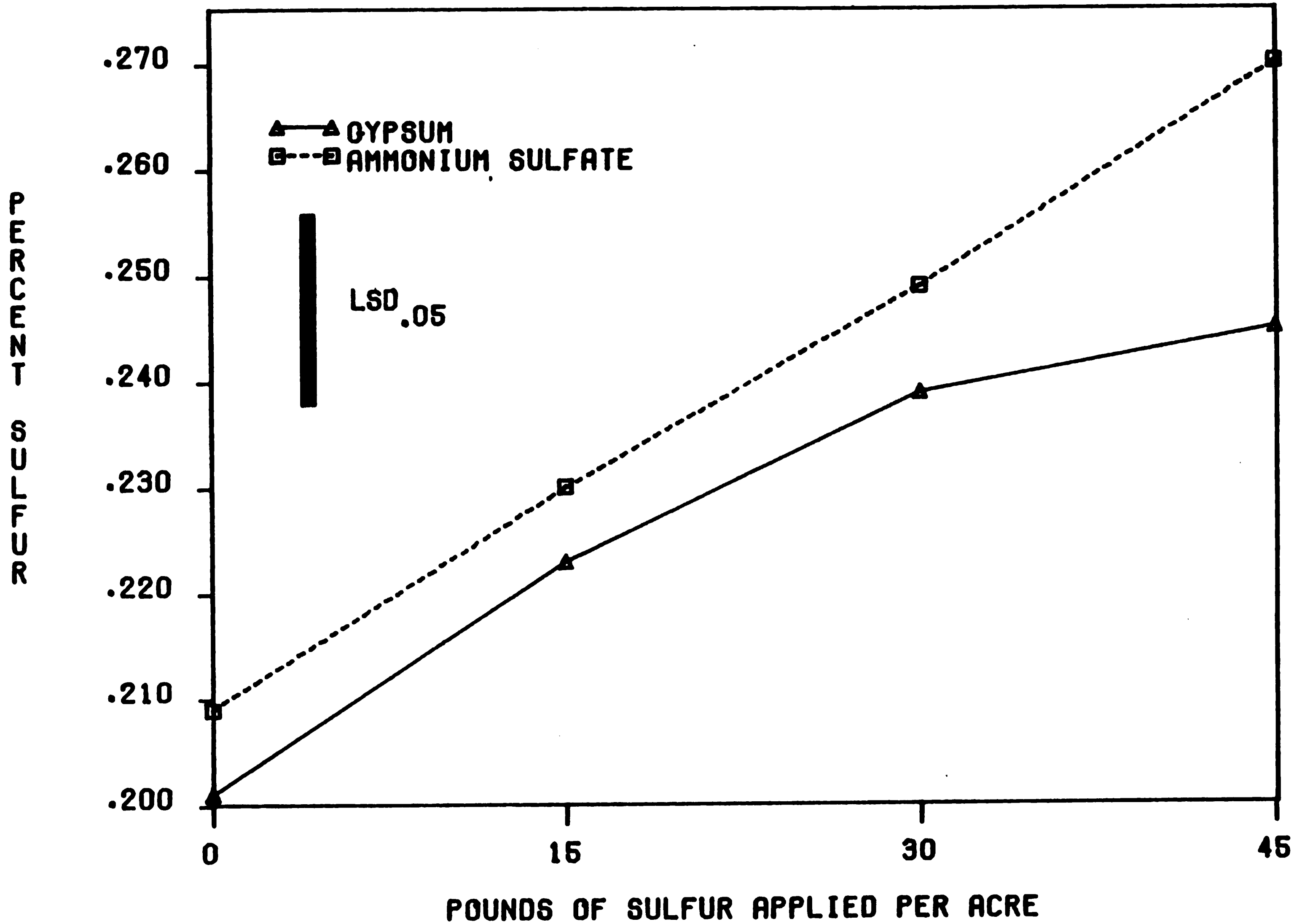

Figure 4. Effect of S application on ear leaf S content of corn in Jackson County.

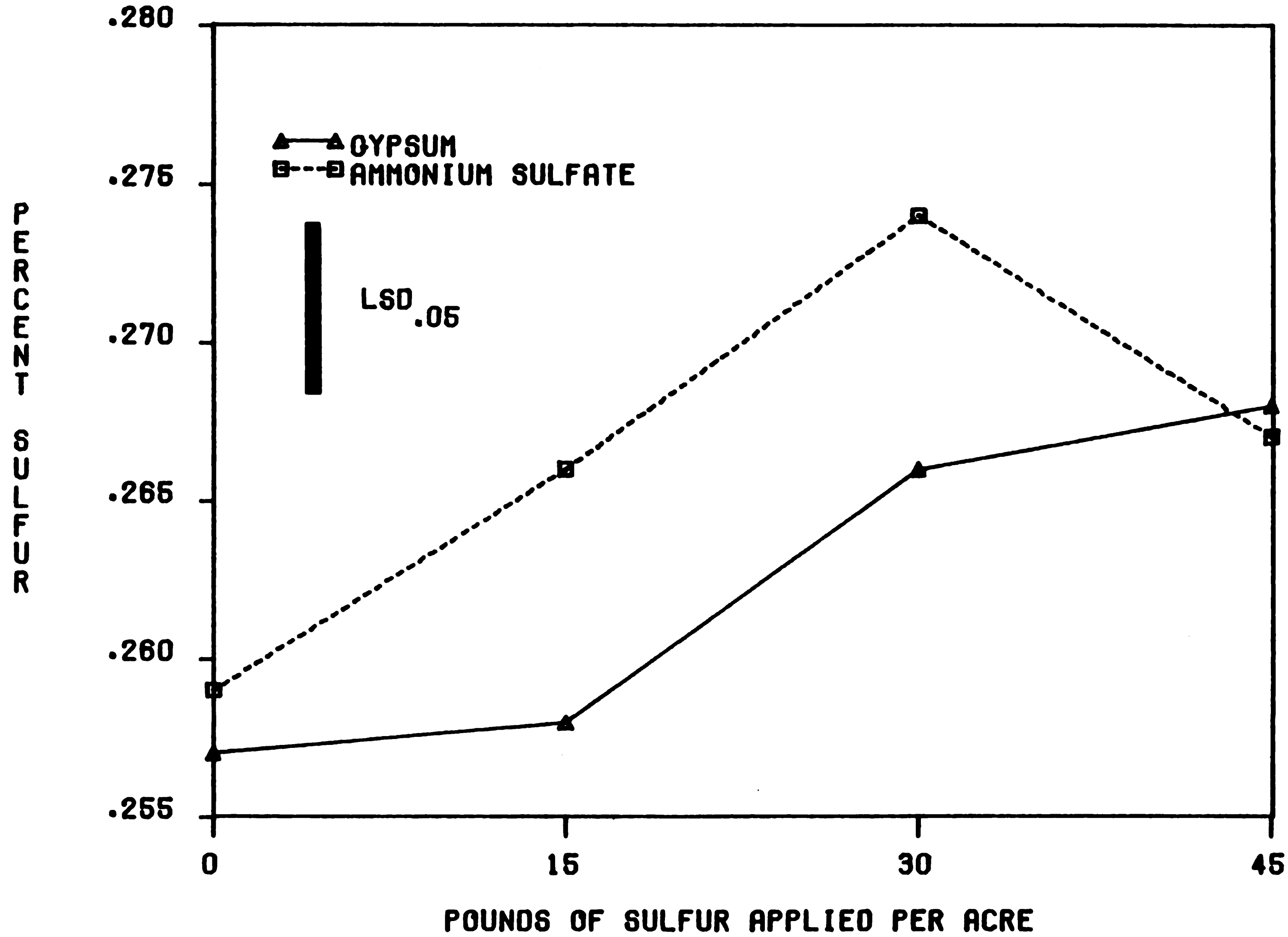

Figure 5. Effect of S application on leaf tissue S content of cranberry beans in Montcalm County.

content.

Corn in Saginaw County showed significant increases in plant S content due to increased rate of S application (Figure 6). From the graph it can be seen that neither gypsum nor ammonium sulfate was significantly better in providing S for plant use. Again there is no connection between increased plant S content and increased yield.

Increased rates of ammonium thiosulfate on soybeans in Saginaw County resulted in a significant increase in plant S content (Figure 7). Although analysis of the surface soil indicated that no SO_4-S was present, the soil was heavy and contained 4.3% organic matter. Adsorption of SO_4 to the clay at greater depths and mineralization of the organic matter are possible sources of S in addition to the fertilizer.

Significant differences between gypsum and ammonium sulfate were observed in Washtenaw County (Figure 8). Gypsum was a consistently better S source than ammonium sulfate. A significant increase in plant S content was observed for each successive increment of S applied. These dramatic increases however, did not result in a yield increase.

A summary of the information provided above can be found in Table 4.

The lack of yield response was disappointing although not entirely unexpected. Only once in the history of S research in Michigan has a yield response occurred.

Sulfur contained in the supplemental fertilizer may not have been the only source of S available to the plant. Fertilizer applied by the farmer may have contained S as an inpurity. A number of sites were irrigated. Significant amounts of S may have been provided by the

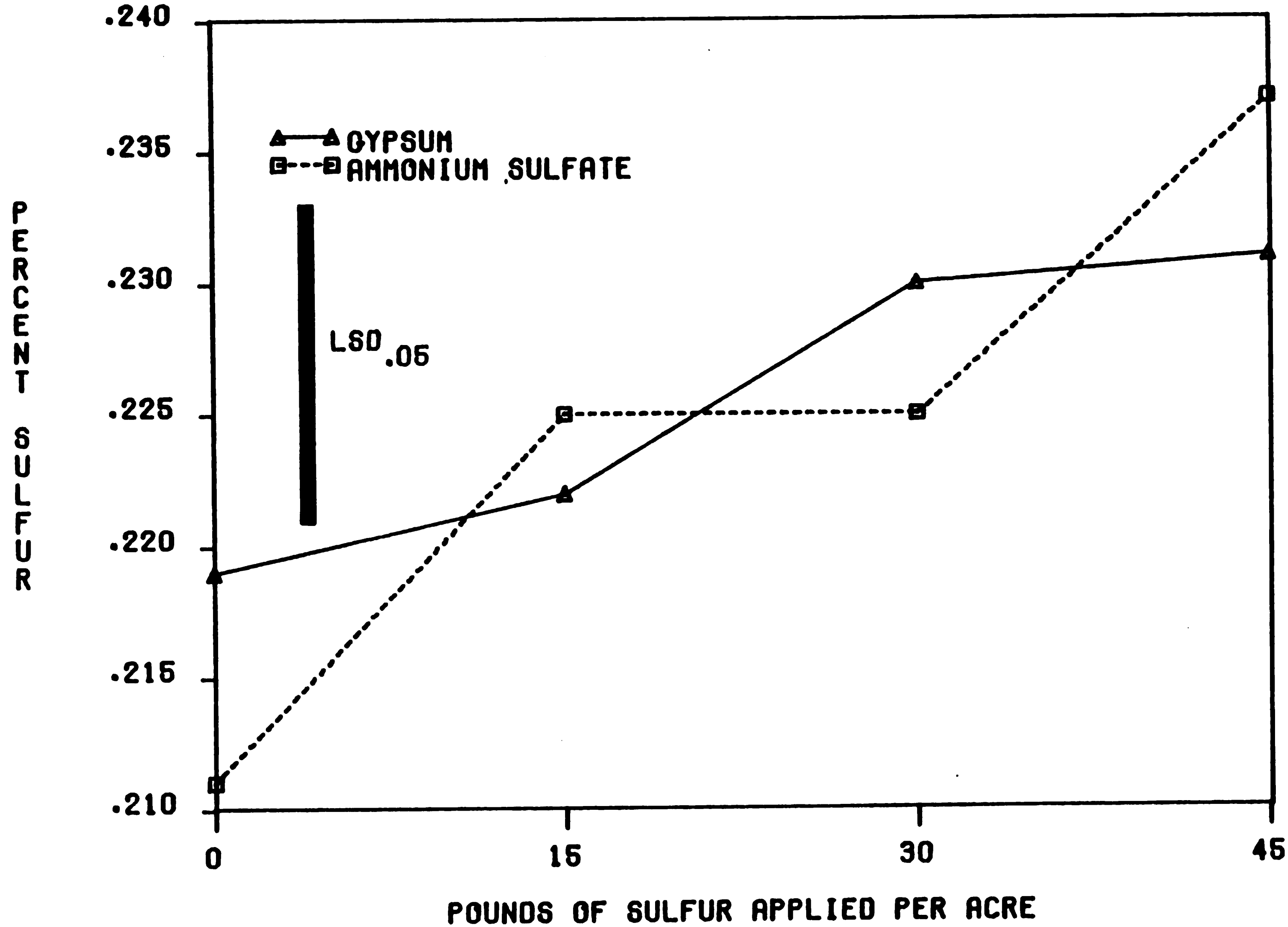

Figure 6. Effect of S application on ear leaf S content of corn in Saginaw County.

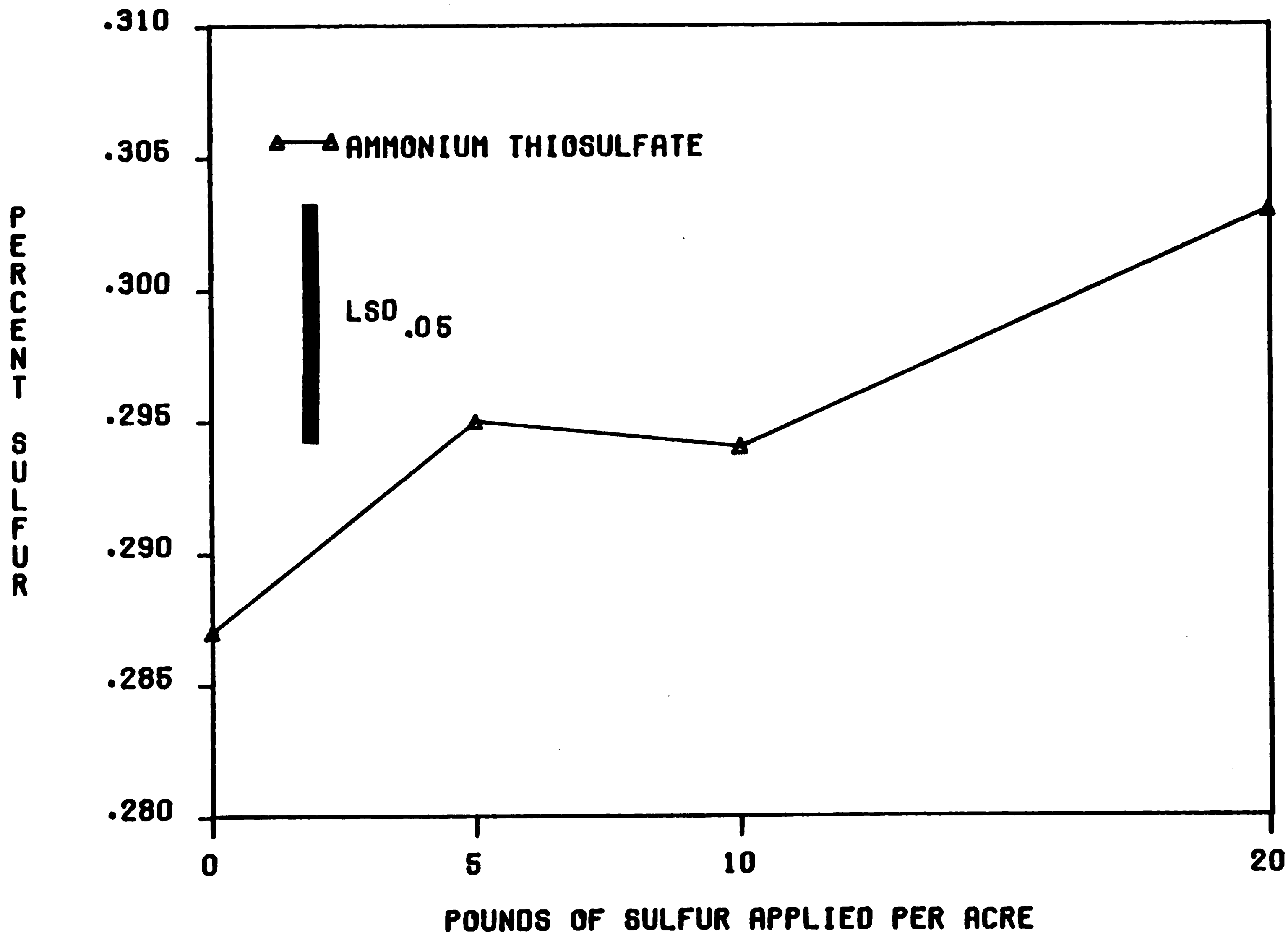

Figure 7. Effect of S application on leaf tissue S content of soybeans in Saginaw County.

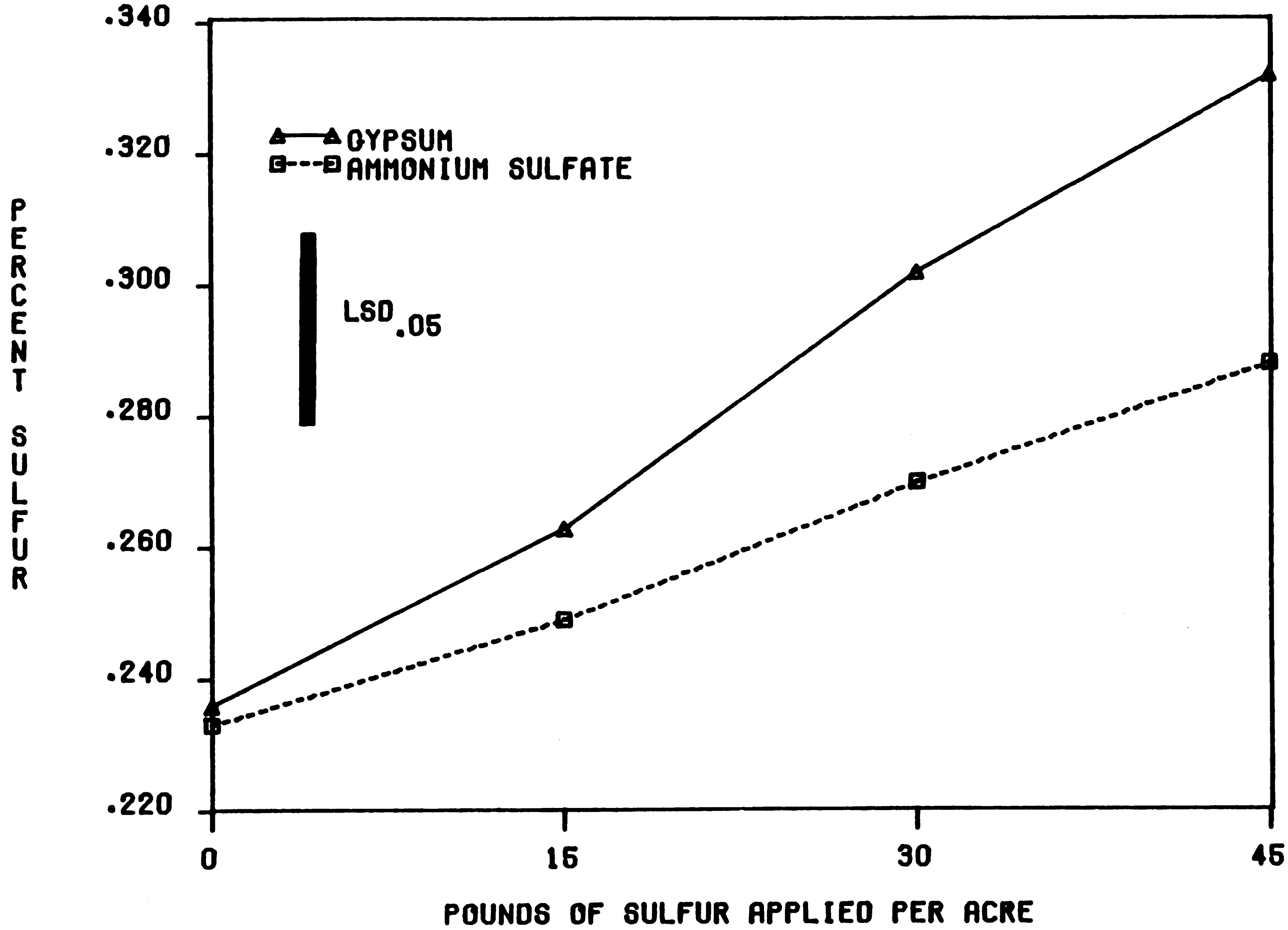

Figure 8. Effect of S application on ear leaf S content of corn in Washtenaw County.

Table 4. Summary of significant differences at $LSD_{.05}$ in plant S content data.

County	Source	Rate+
Barry	n.s.	*
Branch	n.s.	n.s.
Ionia	*	n.s.
Jackson	*	*
Montcalm	n.s.	*
Saginaw		
corn	n.s.	*
soybeans	--	*
Washtenaw	*	*

+ Rate is significant over both sources.

irrigation water during the growing season. Sulfur dioxide in the atmosphere also provides S to the plant. It has been shown in previous work that the S status of the soil will determine the amount of SO_2 utilized by the plant. Depressed soil levels enhance the use of SO_2 by plants until the soil supply has been replenished. The combination of SO_4 in the soil, SO_2 in the atmosphere and SO_4 in rainfall and irrigation water may combine to provide adequate S for the plant, making the application of supplemental S unnecessary.

SUMMARY

Significant yield increases were not obtained at any of the eight experimental sites in Michigan when supplemental S was applied although yield differences were observed. Differences in plant S content due to increasing rate of fertilizer application were observed in Barry, Jackson, Montcalm, Saginaw and Washtenaw Counties. Differences in the ability of fertilizers to provide S to the plant were significant in Ionia, Jackson and Washtenaw Counties. Branch County, which had the lowest soil analysis for S, did not produce a significant difference in either category.

Research in 1981 failed to demonstrate a need for supplemental S application to corn, soybeans and dry edible beans in Michigan.

APPENDICES

APPENDIX A

Analytical Methods

Soil Analysis (Liegel, et al., 1980)

Extracting Solution (500 ppm P in 2 N acetic acid): Dissolve 2.03 g of $Ca(H_2PO_4)_2 \cdot H_2O$ in 800 ml of distilled water. Add 115 ml of glacial acetic acid and dilute to one liter.

$BaCl_2$-Gum Arabic-HOAc: Dissolve 5 g of gum arabic in 500 ml of hot water. Filter if cloudy. Add 50 g of $BaCl_2 \cdot 2H_2O$ and 450 ml of glacial acetic acid and dilute to one liter.

Activated Charcoal: Boil 20 g of charcoal in 200 ml 6 N HCl for 10 minutes. Filter under suction and wash until free of chloride. Dry in an oven.

Standard S Solution (100 ppm S): Dissolve 0.544 g of oven-dried K_2SO_4 in 500 ml of water. Add 10 ml of glacial acetic acid as a preservative and dilute to one liter.

P Solution for Standards (2000 ppm P in 8 N HOAc): Dissolve 8.12 g of $Ca(H_2PO_4)_2 \cdot H_2O$ in 500 ml of water. Add 460 ml of glacial acetic acid and dilute to one liter.

Working S Standards (0, 2, 4, 6, 8, and 10 ppm S): Transfer 0, 2, 4, 6, 8, and 10 ml of standard S solution to 100 ml volumetric flasks. Add 25 ml of the P solution for standards and dilute to 100 ml.

Procedure: Weigh 10 g of soil into a 50 ml flask. Add 25 ml of extracting solution and 0.1 g of activated charcoal. Shake for 15 minutes and filter through Whatman No. 2 filter paper. Transfer 10 ml of filtrate to a 50 ml test tube. Add to this 10 ml of the $BaCl_2$-Gum Arabic-HOAc solution and bubble for 5 seconds. Read the turbid solution on a spectrophotometer at the 420 nm setting. A set of standards must be run with each batch of samples. Pour 25 ml of each standard into an extraction flask and add charcoal. Throughout the rest of the procedure, treat the standards the same as the samples.

Plant Analysis (Blanchar, et al., 1965)

Buffer Solution: In turn dissolve 40 g of $MgCl_2 \cdot 6H_2O$, 4.1 g of CH_3COONa and 0.83 g of KNO_3 nitrate in 800 ml of distilled water. Add 28 ml of 95% ethanol and dilute to one liter.

Standard S Solution (1000 ppm S): Dissolve 5.44 g of oven-dried K_2SO_4 in water and dilute to one liter.

Working S Standards (10, 15, 20, 30 and 40 ppm S): Transfer 10, 15, 20, 30 and 40 ml of Standard S Solution to one liter volumetric flasks. To each flask add 500 ml of water, 200 ml of buffer solution and 20 ml of perchloric acid and dilute to one liter.

Procedure: Dry the plant tissue and grind it to pass a forty mesh screen. Weigh 0.50 g of sample into a digestion tube. Add two glass beads and 3 ml of nitric acid. Put a glass funnel in the mouth of the tube and allow to stand overnight. Add 2 ml of 60 to 70% perchloric acid through the funnel. Preheat the digestion block to 150C and digest for one hour. Remove the tubes from the holes and place them on top of the block beside the holes in contact with the hot block. Raise the temperature to 235C and insert the tubes in the block. Digest for two hours. Remove the tubes from the block and allow them to cool. Remove the funnels. Add 1 ml of HCl and digest for 20 minutes at 150C. Remove the tubes from the block and allow them to cool. Add 35 ml of distilled water and 10 ml of buffer solution. Bring the sample to a final volume of 50 ml with distilled water. Filter into glass bottles through Whatman No. 2 filter paper.

A single sample is placed on a magnetic stirrer and allowed to come to a constant state. Add 0.30 g of 20 to 30 mesh $BaCl_2$ crystals and stir for one minute. Remove the solution from the stirrer and read the turbid solution on a spectrophotometer set on 420 nm exactly two minutes after adding the $BaCl_2$. Readings are compared to standard solutions which are run with each group of samples.

APPENDIX B

Yield Data

Table B1. Yield data for soybeans in Barry County.*

N Rate (lb N/acre)	Ammonium Sulfate (bu/acre)	Ammonium Nitrate (bu/acre)
0	51	49
13	52	53
26	52	54
39	53	52

* Average of 2 replications.

Table B2. Yield data for corn in Branch County.*

S Rate (lb S/acre)	Gypsum (bu/acre)	Ammonium Sulfate (bu/acre)
0	138	125
15	145	142
30	149	137
45	135	139

* Average of 5 replications.

Table B3. Yield data for navy beans in Ionia County.*

N Rate (lb N/acre)	Ammonium Sulfate (cwt/acre)	Ammonium Nitrate (cwt/acre)
0	26.8	25.2
13	27.3	27.8
26	29.3	26.7
39	25.5	27.4

* Average of 6 replications.

Table B4. Yield data for corn in Jackson County.*

S Rate (lb S/acre)	Gypsum (bu/acre)	Ammonium Sulfate (bu/acre)
0	123	121
15	118	119
30	128	124
45	124	122

* Average of 6 replications.

Table B5. Yield data for cranberry beans in Montcalm County.*

S Rate (lb S/acre)	Gypsum (cwt/acre)	Ammonium Sulfate (cwt/acre)
0	21.7	22.0
15	20.4	20.5
30	20.3	19.9
45	19.7	20.0

* Average of 6 replications.

Table B6. Yield data for corn in Saginaw County.*

S Rate (lb S/acre)	Gypsum (bu/acre)	Ammonium Sulfate (bu/acre)
0	133	122
15	134	127
30	126	130
45	134	128

* Average of 4 replications.

Table B7. Yield data for soybeans in Saginaw County.*

S Rate (lb S/acre)	Ammonium Thiosulfate (bu/acre)
0	63
5	61
10	60
20	58

* Average of 4 replications.

Table B8. Yield data for corn in Washtenaw County.*

S Rate (lb S/acre)	Gypsum (bu/acre)	Ammonium Sulfate (bu/acre)
0	83	85
15	85	81
30	83	86
45	87	83

* Average of 5 replications.

APPENDIX C

Plant Sulfur Content Data

Table C1. Leaf tissue S content for soybeans in Barry County.*

N Rate (lb N/acre)	Ammonium Sulfate (%S)		Ammonium Nitrate (%S)
0	.309		.311
13	.308		.312
26	.311		.311
39	.318		.316
$LSD_{.05}$		.009	

* Average of 6 replications.

Table C2. Ear leaf S content for corn in Branch County.*

S Rate (lb S/acre)	Gypsum (%S)		Ammonium Sulfate (%S)
0	.282		.286
15	.278		.290
30	.284		.284
45	.307		.290
$LSD_{.05}$		.024	

* Average of 5 replications.

Table C3. Leaf tissue S content for navy beans in Ionia County.*

N Rate (lb N/acre)	Ammonium Sulfate (%S)		Ammonium Nitrate (%S)
0	.300		.303
13	.313		.295
26	.307		.299
39	.312		.300
$LSD_{.05}$		.010	

* Average of 6 replications.

Table C4. Ear leaf S content for corn in Jackson County.*

S Rate (lb S/acre)	Gypsum (%S)		Ammonium Sulfate (%S)
0	.201		.209
15	.223		.230
30	.239		.249
45	.245		.270
$LSD_{.05}$		.017	

* Average of 6 replications.

Table C5. Leaf tissue S content for cranberry beans in Montcalm County.*

S Rate (lb S/acre)	Gypsum (%S)	Ammonium Sulfate (%S)
0	.257	.259
15	.258	.266
30	.266	.274
45	.268	.267
$LSD_{.05}$	.009	

* Average of 6 replications.

Table C6. Ear leaf S content for corn in Saginaw County.*

S Rate (lb S/acre)	Gypsum (%S)	Ammonium Sulfate (%S)
0	.219	.211
15	.222	.225
30	.230	.225
45	.231	.237
$LSD_{.05}$	.012	

* Average of 4 replications.

Table C7. Leaf tissue S content for soybeans in Saginaw County.*

S Rate (lb S/acre)	Ammonium Thiosulfate (%S)
0	.287
5	.295
10	.294
20	.303
$LSD_{.05}$	.009

* Average of 4 replications.

Table C8. Ear leaf S content for corn in Washtenaw County.*

S Rate (lb S/acre)	Gypsum (%S)	Ammonium Sulfate (%S)
0	.236	.233
15	.263	.249
30	.302	.270
45	.332	.288
$LSD_{.05}$	.029	

* Average of 5 replications.

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