RELATION OF SULFATE CONTENTS BEFORE AND AFTER INCUBATION OF SOILS TO CROP RESPONSES TO SULFUR

by

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RELATION OF SULFATE CONTENTS
BEFORE AND AFTER INCUBATION OF
SOILS TO CROP RESPONSES TO SULFUR

INTRODUCTION AND OBJECTIVES

Fertilizer materials which contain sulfur as an incidental component are decreasing in use due to the trend toward high analysis fertilizers. The applications of many high analysis fertilizers fail to maintain the sulfur contents of the soils. Many areas in the United States now show signs of sulfur deficiencies and separate applications of sulfur carrying fertilizers must be made to maintain or increase the sulfur content of the soil for the optimum growth of crops. The northwestern states, California, and the southeastern states are well known to have areas deficient in sulfur.

Sulfur occurs in soils in many forms. The transformations of the unavailable forms of sulfur to the available form are primarily due to the oxidation of inorganic sulfur compounds, such as sulfides and elemental sulfur, and the aerobic decomposition of organic sulfur compounds to sulfates.

There is a general belief that most of the sulfur in soils is in organic forms. In such a case, the rate and degree of release of sulfates by microbial mineralization of organic sulfur may be of prime importance and may be the controlling factor that determines the amount of sulfates that will be available to the plants. Although little work has been done on the rate of mineralization of soil organic matter in relation to the release of sulfates for plant use, nitrifiable nitrogen has been successfully used in Iowa to predict the nitrogen needs of corn. Because nitrogen and sulfur are similarly related to organic matter, the amount of sulfates released by mineralization might also provide some basis for making recommendations for sulfur fertilization.

Although the need for sulfur in numerous areas has been known for many years, there is no reliable soil-test to detect sulfur deficiencies. Because of this situation, a study was initiated to investigate the feasibility of using the levels of sulfur mineralized as a means of detecting sulfur deficiencies in soils. This study was regarded as preliminary in nature. Yield data and soil samples from other studies which contained sulfur variables were utilized; therefore, controlling the plant species or soil series was not possible. Such control would be desirable for more precise correlation work. However, within the limits that this study was intended, that is, indications of feasibility of a soil test, the samples should provide some useful information.

This study, as initially outlined, had the following objectives:

(1) To devise a method of determining the amounts of sulfates released by soils upon incubation.

(2) To determine if there is any correlation between the levels of sulfate sulfur before and after incubation of soils and crop responses to sulfur fertilization.

REVIEW OF LITERATURE

Sulfur in Soils

Amounts of Sulfur

Studies on sulfur have been too often neglected in favor of other plant nutrients, notably nitrogen, potassium and phosphorus. Although most soils contain less total sulfur than phosphorus, the amounts of sulfur and phosphorus removed by plants are not very different. Lipman and Conybeare (43) reported that in 1930, crops removed an average of 2.8 pounds of sulfur and 3.8 pounds of phosphorus per acre. Some crops, such as the crucifers (30) and onions (39) remove more sulfur than phosphorus; legumes, especially alfalfa (30), may remove more sulfur than phosphorus.

Many areas have been reported to be deficient in sulfur for the optimum growth of plants. Millar (49) reported that at least eight states needed sulfur in addition to that added incidentally with other fertilizers. California, the northwestern states and Florida were listed in this category. If fertilizers were not applied, 18 or possibly 23 states, would need sulfur. Bardsley and Jordan (5, 6) and Jordan and Bardsley (38) found that the surface soils in the southeast were commonly low in sulfate extracted by sodium acetate solution. Most of the soils contained not more than six pounds of sulfur per acre. Soils in Alabama, Oklahoma, and Maryland were found by Eaton (19) to be deficient in sulfur. Shedd (63) found that 27 of 31 soils representing the principle soil areas of Kentucky contained less than 30 ppm of sulfate sulfur; 23 of the 31 soils had less than 20 ppm of sulfate sulfur. Here in the Willamette Valley, the basaltic red hill soils are rather low in total sulfur in the plowed surface layer. Powers (56) reported that sulfur contents of these soils generally ranged from 150 to 400 pounds per acre, frequently being as low as 100 to 130 pounds per acre.

Distribution of Sulfur in Soil Profiles

Analysis of soils representing different sections of Kansas showed that the sulfur contents were about the same at all depths (65). However, typical soils of Kentucky (60), Ohio (2), and Alabama, Maryland, and Oklahoma (19), have been reported to contain more sulfur in the surface layer than in the subsoil. Contrary to the above results, most soils of the southeast (5, 6, 38) had an accumulation of sulfur in the lower horizons.

From what has been reported in the literature on the distribution of sulfur in the soil profile, a general statement for all soils cannot be made. The distribution may vary, depending on the soil type, type of crops grown, and other management factors.

Interrelations of Organic Matter, Total Sulfur and Sulfate Sulfur

Eaton (19) found most of the sulfur in soils of Alabama, Maryland, and Oklahoma in the organic form; there was a direct correlation between total sulfur and organic matter content. Similar results were reported for soils of Ohio (2) and Illinois (33). By analyzing 39 samples of podzol, chernozen and black prairie soils of Minnesota, Evans and Rost (23) found a direct correlation between organic matter and total sulfur, and between total sulfur and sulfate sulfur.

The foregoing clearly indicates that organic matter is an important factor or component associated with total soil sulfur.

Biological Transformations of Sulfur

Organisms Responsible. Unavailable forms of soil sulfur are transformed to the available form by the oxidation of inorganic sulfur compounds and by the aerobic decomposition of organic sulfur compounds. These transformations of soil sulfur are primarily due to biological, rather than chemical reactions.

The oxidation of inorganic sulfur to sulfates is due primarily to the microorganisms belonging to the genus *Thiobacillus* (67). The aerobic decomposition of organic sulfur is done by the numerous heterotrophic microorganisms present in the soil.

Factors Affecting the Oxidation of Inorganic Sulfur. Of the factors affecting the oxidation of soil sulfur, temperature, soil moisture, and aeration can be controlled in laboratory studies. Most workers used temperatures of 25° to 30° C. and soil moisture content of 50% to 60% saturation. Allison (1) incubated the soils at 27° to 28° C. for six weeks to study the effect of nitrates on the oxidation of sulfur. To study the effect of sulfur oxidation on some soil properties, McGeorge and Greene (47) incubated their soils at 30° C. for 11 weeks. Both papers did not state what the moisture contents were. Ames and Boltz (3), Ames and Richmond (4) and Fife (24) moistened the soils to 60% of saturation and incubated at 28° to 30° C., while Halversen and Bollen (28), and Lipman, McLean and Lint (44, 45) used soils at 50% saturation. Brown and Kellogg (11) and Joffe (37) reported that soils at 50% water saturation produced the greatest amount of water soluble sulfates upon incubation.

There are conflicting reports on the effect of lime on sulfur oxidation. Shedd (63) found that over half of the soils tested was benefited by the addition of calcium carbonate. He found that lime did not significantly affect sulfur oxidation when elemental sulfur was added to the same soils. Brown and Johnson (10) reported that calcium carbonate or magnesium carbonate in small amounts benefited the oxidation of applied sulfur; similar results were found by Ames and Richmond (4) with black clay soils, but not with silt and peat soils. Vavra and Frederick (66) reported that large amounts of calcium carbonate increased sulfur oxidation. The inconsistent effect of lime on the oxidation of sulfur in different soils may be due to differences in such factors as pH and levels of calcium and magnesium between the soils.

Effect on Other Soil Constituents. The oxidation of inorganic sulfur reduced the soil pH with a consequent increase in soluble bases (48) and soluble phosphates (42, 44, 45, 47). Ligon (42) found that soluble calcium and potassium had increased. Other cations affected similarly were aluminum and manganese (3), magnesium and sodium (47). Decreases in exchangeable cations were reported by McNeur (48) and Saunders and Blakemore (59).

Ammonification is increased and nitrification is decreased with the application of elemental sulfur or some sulfates. Fife (24) found that sulfur increased ammonification 10% to 100%, depending on the organic matter content of the soil and the amount of sulfur added. Duley (18) reported that nitrates in the soil decreased when sulfur or gypsum were added. Cornfield (16) reported that application of sulfur, aluminum sulfate or ferrous sulfate decreased the soil pH and nitrate content and increased the ammonia content.

Methods of Extraction and Determination of Sulfate Sulfur

Soil sulfates have been extracted by numerous reagents, of which water, dilute acids and sodium acetate solution buffered with glacial acetic acid seemed to be the most common. Most of the early workers, such as Brown and Kellogg (11) in Iowa and Halversen and Bollen (28) in Oregon used water as the extracting agent. Some, like Shedd (63) in Kentucky digested the soil with 1% HCl solution. Evans and Rost (23) and Little (46) also used dilute hydrochloric acid solutions. Chesnin and Yien (13) employed sodium acetate solution buffered at pH 4.8 with glacial acetic acid, and an extractant to soil ratio of 5:1. Other workers using sodium acetate-acetic acid extraction included Ensminger (20), Hesse (31) and Kamprath, Nelson and Fitts (40).

The relative strengths of some extractants were compared by Ensminger (20), who found that solutions of sodium acetate at pH 4.8, neutral sodium acetate, monopotassium hydrogen phosphate containing 50 ppm of phosphorus and monopotassium hydrogen phosphate containing 100 ppm of phosphorus extracted about the same amount of sulfate. However, 0.1 N HCl and water extracted less sulfate from the soils.

Sulfates present in soil extracts have been determined by four general methods, which include gravimetric, titrimetric, colorimetric and turbimetric procedures. In the gravimetric method, barium chloride is added to the soil extracts to precipitate the sulfates as barium sulfate, which is then washed, dried and weighed. The low solubility of barium sulfate minimizes the loss of precipitate during the washing process. Using this method, Shedd (63) determined the sulfate content of Kentucky soils, and Evans and Rost (23) did the same for Minnesota soils. The latter also determined total organic sulfur, humus sulfur and total sulfur gravimetrically after converting the different forms of sulfur to sulfate. The gravimetric method seems to be accurate, but lengthy.

Several titrimetric methods for the determination of sulfates in soil extracts are available. A method that is simple, rapid and free from the interference of other ions was proposed by Little (46). The acidified soil extracts were titrated with standard barium chloride solution, using sodium rhodizonate as an internal indicator and bromocresol purple to increase the sensitivity of the rhodizonate. Cantino (12) made the soil extracts alkaline to phenolphthalein, whereupon the sulfate was precipitated with standard barium chromate. The excess barium chromate was then titrated with sodium thiosulfate. After destroying organic matter in the soil extracts, Christie and Martin (14) precipitated the sulfate as benzidine sulfate by adding benzidine hydrochloride. The precipitate was then titrated with standard potassium permanganate solution. The latter method was improved by Hibbard (32) by removing ferric and phosphate ions which interfered with the analysis. The versene (EDTA) method (34) was reported to be accurate and precise but lengthy, requiring three separate analyses. One aliquot was used to determine the combined calcium and magnesium by titration with EDTA. The total carbonates and bicarbonates was determined on another aliquot. After the carbonates and bicarbonates were removed from the third aliquot, excess standard barium chloride was added to precipitate the sulfate. The excess barium chloride was then titrated with standard EDTA solution.

In the colorimetric method of Bertolacini and Barney (7), the color was developed by the addition of barium chloranilate to the soil extracts, which were first passed through exchange resins for the removal of interfering ions.

A rapid turbimetric method with no appreciable interference from several ions was devised by Chesnin and Yien (13). Barium chloride crystals passing through 30 mesh but retained by 60 mesh screens were added to the soil extracts to form a turbid suspensions of barium sulfate. The suspensions were stabilized by the addition of gum acacia solution. The turbidity readings were taken with the aid of a colorimeter.

Whether any of the methods can be adopted to predict the sulfur needs of crops has not been determined. Correlation work must be done before soil analysis can be used to detect sulfur deficiencies.

Crop Responses to Sulfur and Sulfates

Many areas in this country are deficient in sulfur for the optimum growth of crops; this is verified by the reported responses to sulfur fertilization of various crops grown on numerous soils.

Jordan and Bardsley (38) reported that 10 of 29 field experiments located in seven southeastern states showed crop responses to calcium sulfate equivalent to 4 to 8 pounds sulfur per acre. Crops which responded were cotton seeds, clover and tobacco. Clover, grown on 12 soils in the greenhouse, responded to calcium sulfate. Peanuts grown in Florida responded to calcium sulfate; the magnitude of response varied with the variety of peanuts (41).

When various carriers of sulfur were added to Kentucky soils, Shedd (61) found the following crop responses: 10% to 25% for tobacco; 8% to 30% for soybeans; as high as 67% for turnips; and 2% to 110% for mustard. The magnitude of response varied with the soil and the sulfur carrier. Some crops, such as clover and cabbage did not respond. Shedd (62) also reported that the yields of soybeans, clover, oats, alfalfa, and wheat were increased on some soils when 100 or 200 pounds of elemental sulfur were applied.

Application of 150 pounds sulfur per acre on a Missouri soil increased the yields of clover 50% and corn and rape slightly (18). Alfalfa was grown on six principle Idaho soils in the greenhouse by Neidig, McDole and Magnuson (53). The yields on five of the soils were increased by the application of either 100 pounds of elemental sulfur per acre or 200 pounds of gypsum per acre. Sulfur increased the yields 30% to 120% and gypsum increased the yield 15% to 80%. Field experiments in Montana (51) showed that the yields of alfalfa were doubled with the use of gypsum and tripled with the use of sulfur. Numerous field trials in Iowa (22) have shown that crops such as oats, barley, clover, and alfalfa responded very well to gypsum. Responses of clover and oats to gypsum were also reported by Erdman (21) and responses of alfalfa to gypsum and sulfur were found by Bollen (8).

Conrad (15) reported that many areas of California were deficient in sulfur; either sulfur or gypsum increased the yields. Rendig (58) found that alfalfa grown on Delhi sand in the San Joaquin Valley were highly responsive to gypsum applied at rates of 200 and 400 pounds per acre. The average yield was more than twice that of the untreated plots.

Neller (54) reported responses of alfalfa and clover to 160 pounds of sulfur per acre and 200 pounds of gypsum per acre in greenhouse and field trials on Palouse, Ritzville, and Sagemoor soils of Washington. Greenhouse trials resulted in yield increases of 12% to 70% for alfalfa and 22% to 117% for clover, and generally resulted in higher yields from sulfur than from gypsum. The magnitudes of responses were less in the field experiments. Responses of barley, oats, wheat, and peas to sulfur treatments on Palouse soils in greenhouse trials were reported by Olson and St. John (55). They also reported alfalfa responses to gypsum and sulfur on college farms and alfalfa responses to gypsum on many of the neighboring farms. Alfalfa grown on Lynden and Olympia soils responded over 75% when sulfates were applied (27).

Here in Oregon crop responses to sulfur and sulfates were reported as early as 1917. Alfalfa responded to gypsum and sulfur in field experiments at the Hood River Station (9). Miller (50) increased the yields of red clover and oats on Beaverdam, Medford and Antelope soils with the application of sulfur, sodium sulfate or calcium sulfate. When sulfur and various forms of sulfates were applied to the soils in southern Oregon, the yields of alfalfa and clover increased 50% to 1000% on many of the soils (57). The field experiments were conducted on various types of soil, from course granites to the heaviest adobe. Powers (56) reported that many trials with alfalfa on 22 leading soil types throughout the state showed responses to sulfur and gypsum. He estimated that the yields of at least 100,000 acres of alfalfa in Oregon could have been increased with sulfur fertilization. Spring barley and oats in certain areas in the Willamette Valley (35, 36) and alfalfa in eastern Oregon (17) responded to gypsum.

PRELIMINARY METHODOLOGY STUDIES

Since a universally accepted method of determining the sulfur supplying power of soils is not available, one logical alternative is to develop an incubation method similar to that used for nitrogen in Iowa.

A series of papers (25, 26, 29, 52, 64) has been written on the methods of determining nitrifiable nitrogen and on the results of correlation with the yields of corn. The method described by Stanford and Hanway (64) was precise and the nitrates released were highly correlated with the yields of corn. In their method, 10 g. of air-dried soils were mixed with equal volumes of exfoliated vermicullite. The mixtures were then leached free of nitrates with three successive portions of 20 ml. water; suction was then applied to remove excess water. The samples were incubated at 35° C. in a nearly saturated atmosphere. After two weeks, the samples were leached free of nitrates as before; the nitrates recovered represented nitrifiable nitrogen. The advantageous features of the method are: (1) Only one nitrate analysis is necessary to determine nitrifiable nitrogen. (2) Moisture losses during incubation are negligible due to the high moisture-holding capacity of vermicullite, the highly humidified environment and the placement of one-hold rubber stoppers or plastic caps over leaching tubes containing the soil samples. (3) The use of vermicullite also permits better diffusion of air due to increased porosity.

The method of Stanford and Hanway (64) for release of nitrate was initially investigated to determine if the method could be adopted or modified for the release of soil sulfates.

Leaching of Soil Sulfates

Whether 60 ml. of water would be enough to leach the soil sulfates from the soil-vermicullite mixture was investigated. Four soils (Aiken, Sams, Walla Walla, and Willamette), varying in chemical and physical properties in many respects were used. The samples were leached with 60 and 80 ml. of water using a leaching apparatus and procedure similar to that used in Iowa. The 60 ml. of water required over three hours to leach through the column, while 80 ml. required over four hours. Similar results were obtained when Morgan's solution (sodium acetate buffered at pH 4.8 with acetic acid) were used. Because of the time involved, leaching was not considered a suitable method of sulfate extraction for general soil testing procedures.

Method of Sulfate Analysis

Since leaching procedures were not suitable for the extraction of soil sulfates, other methods of extraction were sought. A method for the analysis of sulfate in the soil extracts was also sought. Chesnin and Yien (13) proposed a method which seemed to be simple, rapid, and adaptable for correlation work and for routine soil-testing procedures.

Extraction by shaking with water was initially investigated. When samples of soil-water suspensions were shaken, the extracts contained suspensions of colloidal soil particles even after filtration through Whatman #42 filter papers. When clarifying agents (cupric acetete, calcium hydroxide and ammonium carbonate) were added, erroneous turbidity readings were obtained. The higher turbidity readings might have been corrected if further investigations were conducted. Because a simple and rapid method was desired, water extraction was not considered suitable, mainly because of the time required to clarify the extracts.

The question, "Can the method of Chesnin and Yien (13) be improved with respect to speed and simplicity without the sacrifice of accuracy?", arose. The method would be more simple and more rapid if the same procedure was used regardless of the volume of test solution and regardless of the concentration of sulfur. Experiments were conducted simultaneously to determine the effects of the following: (1) addition of 0.25% gum acacia solution before instead of after the addition of barium chloride crystals, (2) addition of 2 ml. of gum acacia to all samples instead of 1 ml. of gum acacia to solutions containing 0-20 ppm of sulfur and 2 ml. to solutions containing 20 to 40 ppm of sulfur, (3) addition of 0.5 g. instead of 1 g. of barium chloride and (4) addition of 2 ml. of acacia and 0.5 g. of barium chloride to 25 ml. of test solutions. Morgan's solution containing 0, 5, 10, 20, 30, and 40 ppm of sulfur as potassium sulfate were employed as the test solutions. The results of the study (Table 1) show that none of the four factors caused the readings to deviate more than six units on the colorimeter employed to measure the turbidity of the solutions. The greatest deviation in the 0-20 ppm range was three units, and the greatest deviation in the 20 to 40 ppm range was six units. These deviations were not considered serious. The results clearly indicate that in the analysis of sulfates by the turbimetric method, 2 ml. of 0.25% gum acacia solution and 0.5 g. of barium chloride may be added to either 25 or 50 ml. of soil extracts containing up to 40 ppm of sulfur.

Table 1

Effects on the Colorimeter readings from the addition of 1 and 2 ml. of 0.25% gum acacia solutions before and after the addition of 0.5 and 1 g. of barium chloride to 25 and 50 ml. standard sulfur solutions

ml. of buffered solution	ml. of gum acacia added	When acacia added relative to $BaCl_2$	Grams of $BaCl_2$ added	ppm S in solution					
				0	5	10	20	30	40
				(--Colorimeter Readings--)					
50	1	Before	0.5	7	52	95			
50	1	After	0.5	7	52	96			
50	1	Before	1.0	7	51	94			
50	1	After	1.0	6	51	94			
50	2	Before	0.5	7	53	96	175	235	274
50	2	After	0.5	7	53	95	174	236	273
50	2	Before	1.0	6	52	95	173	232	270
50	2	After	1.0	7	52	94	173	231	269
25	2	Before	0.5	7	53	97	176	235	275

Although duplicate readings seldom deviated more than two units even in the higher range, the readings of standard solutions often deviated slightly from day to day. Because of the latter fact, standard curves were made with each series of analysis. Every standard curve had the same general shape. A typical standard curve is shown in Figure 1. The shape of the curve, unlike the one shown by Chesnin and Yien (13) can be described as being concave from 0-5 ppm S and convex from 5-40 ppm. The deviation from linearity of the 20-40 ppm portion is greater than the 5-20 ppm portion of the curve. The reasons for such a shape are not known. However, the results of soil analysis should be reliable as long as the colorimetric readings are compared with that of the standard curve prepared at the same time. Since soil extracts may contain less than 5 ppm of sulfur, the standard curve should include two or three points between zero and 5 ppm to get reliable results.

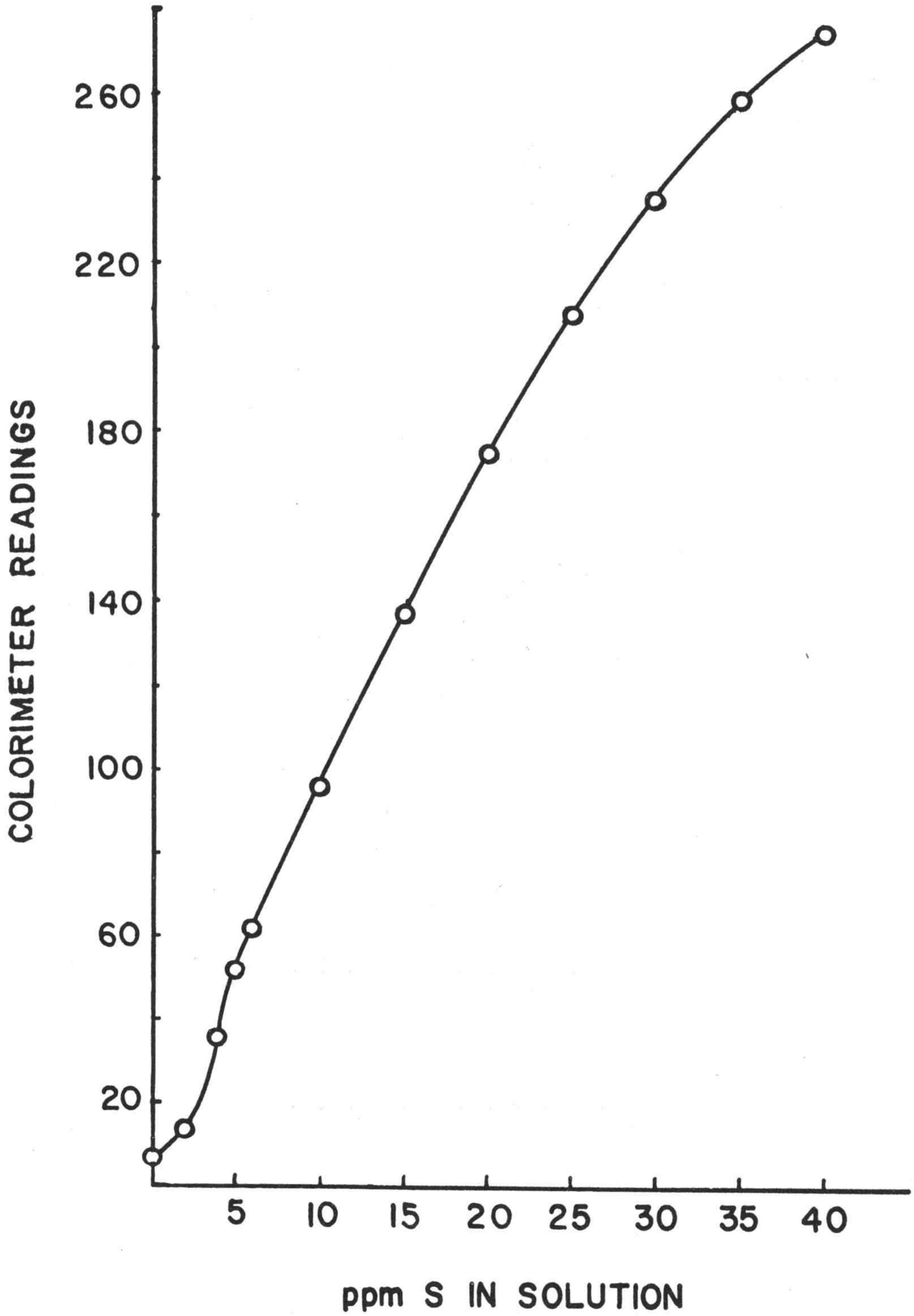

Figure 1. A typical standard curve for turbimetric sulfur

Effect of Charcoal and Vermicullite on Sulfate Analysis

Results of an initial attempt to determine the solution to soil ratio necessary for the optimum extraction of soil sulfates indicated that charcoal and vermicullite interfered with sulfate analysis.

For the determination of the solution to soil ratio, duplicate 10 g. samples of Aiken and Walla Walla soils were mixed with equal volumes of vermicullite. Charcoal was added to the mixtures; the amounts added depended on the suspected amount of organic matter in the soil. The mixtures were then shaken with 60 ml. of Morgan's solution for one-half hour. Another set of mixtures were shaken with 60 ml. of Morgan's solution, which contained potassium sulfate equivalent to 200 ppm of sulfur in the soil.

The results are shown on line 1 in Table 3. The term "unwashed" was used because the materials were used in their original condition. The percent recovery of added sulfur amounted to 109.5% for the Walla Walla soil while only 76.2% was recovered from the Aiken soil. The former figure indicates that charcoal and/or vermicullite may have contaminated the soil, i.e. the materials may have contained enough sulfur to give a high recovery. The low recovery from Aiken soil may have been due to the adsorption of sulfate by the charcoal and vermicullite. If so, the effects were opposite to the high recovery from the

Walla Walla soil. However, the sulfate could have been adsorbed by the Aiken soil, which has a fairly high amount of free oxides of aluminum and iron. Workers have shown that when anions, such as phosphates or sulfates were added to soils containing aluminum and iron oxides, much of the anions were retained or rendered "unavailable" by the soil. T. T. Chao[1] placed gypsum on top of the columns of various soils and leached the soils with water. Most of the sulfate in Aiken and other lateritic soils was located in the upper portion of the column and very little movement occurred. However, soils like Walla Walla had the sulfate concentrated in the lower portion of the column and appreciable amounts of sulfate were found in the leachate. His results indicate that sulfate may be retained by soils high in aluminum and iron oxides.

Since there were indications that unwashed charcoal and unwashed vermicullite may have affected the results of analyses for soil sulfates, the nature of the interference (adsorption or contamination) was studied further.

The interferences of charcoal and vermicullite on the turbidity of standard sulfate solutions were initially investigated. Sixty ml. of Morgan's solution containing 0, 5, and 30 ppm of sulfur as potassium sulfate were each

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1. Research Fellow, Soils Department, Oregon State College, Corvallis, Oregon. Personal communication.

shaken with unwashed charcoal, with unwashed vermicullite and with unwashed charcoal plus vermicullite for one-half hour. One teaspoon of charcoal and two teaspoons of vermicullite were used. The procedure was repeated with charcoal and vermicullite which were previously washed free of sulfate. The mixtures were filtered and the sulfur contents of the extracts were determined. The charcoal and vermicullite were washed free of sulfate by shaking the materials with Morgan's solution for a period of one and a half to two hours. The solution to material ratio was 2:1. The vermicullite was then thoroughly washed free of sodium acetate with tap water, followed with distilled water. When the charcoal had settled the liquid portion was decanted. The sodium acetate was removed by shaking the charcoal with water and then decanting the water; the process was repeated several times to remove most of the sodium acetate. The washed charcoal and vermicullite were thoroughly dried before they were used in the studies.

The results of the study are shown in Table 2. When unwashed charcoal and unwashed vermicullite, either alone or in combination, were used, the sulfur contents were appreciably greater than those of the standard solutions. This indicates that unwashed charcoal and unwashed vermicullite contaminated the solutions with sulfate sulfur. However, the contamination was reduced when the washed materials were used and the amounts were considered insignificant. There was no indication of sulfate adsorption by any of the materials.

Whether charcoal and vermicullite, when added to the soil, would affect the result of analysis for soil sulfate was also investigated. Before the materials could be used, a knowledge of the nature and amount of interference under the conditions of sóil analysis was considered essential. Unwashed charcoal, washed charcoal and washed charcoal plus washed vermicullite were added to 10 g. samples of Aiken and Walla Walla soils. The mixtures were then shaken with 60 ml. of Morgan's solution for one-half hour. Another set of mixtures were shaken with Morgan's solution containing potassium sulfate equivalent to 200 ppm of sulfur in the soil. The two types of charcoal were also added to the soil extracts instead of the soils. The mixtures were shaken for one-half hour.

The results are shown in Table 3. The amounts of sulfate sulfur obtained when washed charcoal was added to the soil extracts are assumed correct. Such an assumption can be made because previous results (Table 2) indicated that the sulfate contents of standard solutions were not significantly affected when the solutions were shaken with washed charcoal. When unwashed charcoal was added to the soil extracts the values of sulfate sulfur were consistently

Table 2

Comparative interferences of washed and unwashed charcoal and vermicullite on the turbidity of standard sulfate solutions. Averages of duplicate samples.

Materials added to the standard solutions and separated prior to determination of sulfur	Solution of 0 ppm S		Solution of 5 ppm S		Solution of 30 ppm S	
	Turbidity Readings	ppm S	Turbidity Readings	ppm S	Turbidity Readings	ppm S
None	7	0	50	5	234	30
Washed charcoal	7	0	51	5.1	236	30.3
Washed Vermicullite	7	0	51	5.1	237	30.5
Washed charcoal plus Vermicullite	7	0	52	5.2	237	30.5
Unwashed charcoal	9	0.7	57	5.5	242	31.5
Unwashed vermicullite	14	2.0	80	8.0	242	31.5
Unwashed charcoal plus vermicullite	23	3.0	85	8.5	245	32.0

Table 3

Comparative interferences of unwashed and washed charcoal and vermicullite on the analysis of soil sulfates extracted from two soils. Averages of duplicate samples.

	Soil Only		Soil plus 200 ppm S in Extractant			
	Aiken	Walla Walla	Aiken		Walla Walla	
	(ppm S)		(ppm S)	(Recovery)	(ppm S)	(Recovery) (Per Cent)
Materials added to the soils:						
Unwashed charcoal plus vermicullite	9.6	9.0	162	76.2	228	109.5
Unwashed charcoal	9.0	9.0	157	74.0	204	97.5
Washed charcoal plus vermicullite	8.4	9.6	156	73.8	192	91.2
Washed charcoal	8.4	9.6	156	73.8	192	91.2
Materials added to the soil extracts:						
Unwashed charcoal	9.0	10.0	158	74.5	191	90.5
Washed charcoal	8.4	9.6	156	73.7	190	90.2

higher than when washed charcoal was used. Although the differences were small and may be negligible from practical viewpoints, they indicate that unwashed charcoal contaminated the analysis. Similar results were obtained when washed charcoal was added to the soil and the extracts; this indicates that washed charcoal may be added either to the soil or to the extracts.

The addition of washed charcoal plus vermicullite and washed charcoal resulted in equal values of sulfate sulfur, which indicates that washed vermicullite did not contaminate the analysis. The results from the addition of unwashed charcoal and vermicullite and washed materials to the soil indicates that the use of unwashed materials gave higher values with Aiken soil and lower values with Walla Walla soil. The opposite effects of unwashed materials on soil analysis indicate that the interference of unwashed materials may differ, depending on the soil to which the materials are added.

When the mixtures were extracted with Morgan's solution containing 200 ppm of sulfur, the addition of unwashed materials resulted in higher values than with the addition of washed materials. The difference indicates sulfate contamination from unwashed materials. Contamination from vermicullite was greater than from charcoal. The reason for the low recovery of the sulfate added to the Aiken soil was discussed previously.

Results of the studies (Tables 2 and 3) indicated that unwashed charcoal and unwashed vermicullite contain sufficient amounts of sulfate to result in high analysis. Because of the contamination, it was decided that charcoal and vermicullite would be washed before using in future studies and soil analysis.

Optimum Ratio of Extractant to Soil

Since the previous series of studies indicated that 200 ppm of sulfur was not completely recovered when the solution to soil ratio was 6:1, a higher ratio was employed to determine whether the recovery would be higher. Duplicate 10 g. samples of Aiken, Sams, Walla Walla and Willamette soils, which are widely different in their properties, were each mixed with equal volumes of washed vermicullite. Charcoal was added to the mixtures, which were then shaken with 60 and 80 ml. of Morgan's solution. Another set of mixtures were shaken with Morgan's solution containing sulfate equivalent to 200 ppm of sulfur in the soil.

The results (Table 4) show that there were no appreciable differences in the amounts of sulfate extracted by 60 and 80 ml. of Morgan's solution. The recovery of sulfate was not appreciably increased from the use of a ratio of 8:1. Whether the recovery could be improved by the use of ratios wider than 8:1 was not investigated because wider ratios would result in decreases in the precision of the analysis of soils containing relatively small amounts of sulfate sulfur. Since most soils are believed to contain small amounts of sulfate sulfur, it is best to keep the ratio as narrow as possible. To remedy the incomplete recovery, a correction factor could be applied. Such practice, however, is impractical because a correction factor would have to be determined for each of the soils analyzed. Since this research is primarily concerned with correlation studies, a correction factor may not be necessary. Furthermore, it is not known if the sulfate which is not recovered by extractants is actually extracted in exactly equivalent amounts by plants. For the purposes of these preliminary studies, it was decided not to apply any correction factor to the soils. Since the amounts of sulfate extracted with extraction ratios of 6:1 and 8:1 were not significantly different, it was decided that the lower ratio would be used in future analysis.

Temperature and Duration of Incubation

In order to complete the methodology phase prior to conducting the correlation studies, the temperature and duration of incubation for the release of soil sulfates

Table 4

Comparative amounts of sulfate extracted from 10 g. samples of four soils, which were shaken with 60 and 80 ml. of Morgan's solution containing sulfate equivalent to zero and 200 ppm of sulfur in the soils. Averages of duplicate samples.

	Extractant/Soil = 6/1			Extractant/Soil = 8/1		
	Soil Only	Soil + 200 ppm S in Extractants	Percent Recovery of S	Soil Only	Soil + 200 ppm S in Extracants	Percent Recovery of S
	(- - - - ppm S - - - -)			(- - - - ppm S - - - -)		
Aiken	8.3	154	72.9	8.4	157	74.3
Sams	10.2	186	87.9	10.3	187	88.4
Walla Walla	9.6	192	91.2	9.6	192	91.2
Willamette	10.8	175	82.1	10.8	177	83.1

were determined. Triplicate 10 g. samples of four soils were mixed with equal volumes of vermicullite and incubated at temperatures of 30 and 35° C for periods of two, three and four weeks in a highly humidified chamber. Before incubation, water was added to each sample to make the soil about 50 percent saturated. There were two reasons for not using temperatures greater than 35° C. Soil moisture losses during incubation might be appreciable at higher temperatures and the incubator used could not be maintained at higher temperatures. Longer periods of incubation were considered impractical for a soil-testing procedure. Since the results are to be used for correlation studies the amounts of sulfate released need not necessarily be optimum nor maximum; relative amounts of sulfate should be sufficient for the correlations.

The results (Table 5) indicate that the release of soil sulfates was higher as the temperature or period of incubation was increased. Since the greatest increase in sulfate release occured between three and four weeks and the release was highest at 35° C, it was decided that the soils should be incubated at 35° C for four weeks.

Summary of Preliminary Methodology Studies

Preliminary laboratory investigations were conducted for the primary purpose of obtaining a method of determining the amount of sulfate released upon incubation of soils.

Table 5

Relative amounts of sulfate released from four soils incubated at 30 and 35° C for two, three and four weeks. Averages of triplicate samples.

Temperature (°C)	Incubation Period (weeks)	Amounts of Sulfate Released (ppm S, air-dried basis)			
		Aiken	Sams	Walla Walla	Willamette
30	2	10.0	11.6	13.0	14.0
	3	11.2	12.0	13.2	14.6
	4	13.2	13.2	14.5	16.0
35	2	12.2	13.2	13.9	15.4
	3	13.4	14.0	14.4	16.4
	4	14.5	15.8	16.5	18.5

Certain phases of the method of Chesnin and Yien (13) and the effect of charcoal and vermicullite as sources of contamination on sulfate analysis were also investigated. The method of Stanford and Hanway (64) for nitrates was investigated to determine if their method could be applied for the release of soil sulfates.

From the results of these investigations, the conditions for the incubation of soil samples and a method of determining the levels of soil sulfates were formulated. The following procedure was selected for use in the remainder of the investigations for studying the relationship between the levels of sulfates before and after incubation and yield response.

> Ten grams of air-dried soils passing through a 2 mm. screen are mixed with equal volumes of sulfur-free vermicullite in leaching tubes equipped with one hole plastic covers. Water is added to the mixtures to make the soils about 50 percent saturated. The plastic covers are placed on the tubes, which are then incubated in a highly humidified chamber for four weeks at 35° C. The soil-vermicullite mixtures are transferred to shaking bottles. One half to three teaspoons of charcoal (depending on the amount of organic matter in the soils) and 60 ml. of Morgan's solution (sodium acetate buffered at

pH 4.8 with acetic acid) are added. The suspensions are shaken for half an hour and filtered through filter papers. Two ml. of 0.25 percent gum acacia solution are added to 25 ml. aliquots of the filtrates. After shaking the solution for a few seconds, 0.5 g. of 30-60 mesh crystals of barium chloride are added to the solutions. The solutions are shaken for about one minute. After five but not later than thirty minutes, the turbidity readings are taken with a photoelectric colorimeter using a blue filter at 420 mμ and compared with a standard curve ran at about the same time. Sulfate extracted from these soils represents "total sulfate sulfur"; that is, it represents the sulfates present in the soils before incubation plus the sulfates released during incubation due to the oxidation of various forms of sulfur to sulfates. Sulfate contents before incubation of soils are determined as above omitting vermicullite and incubation.

RESULTS AND DISCUSSION OF SOIL ANALYSIS AND CORRELATIONS

Sources of Soil Samples

Soil samples and their corresponding yields were obtained for field and greenhouse experiments which contained sulfur as a variable. The field experiments largely involved barley and oats and clover to a lesser extent.[1] The experiments were located on different soil series in the Willamette Valley, most occuring on "bottom land" soils. Treatments included zero and 20 pounds of sulfur as gypsum per acre. Nitrogen, phosphorus and potassium were also applied to both the zero and sulfur treated plots. If the yield data and corresponding soil samples for each replication of an experiment were available, they were considered individually. If not, the total yields and composite soil samples were utilized.

Samples of numerous soil series cropped with alfalfa and clover in the greenhouse were also utilized.[2] Sulfur

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1 Appreciation is expressed to Dr. T. L. Jackson, Department of Soils, Oregon State College for the use of soil samples and yield data for the field experiments.

2 Appreciation is expressed to Messrs. T. T. Chao, D. W. James, E. A. Jenne and J. E. Yahner, Department of Soils, Oregon State College for soil samples and yield data of greenhouse experiments.

applications as gypsum in studies conducted under Oregon Agricultural Experiment Station Project No. 310 involved rates of zero and 100 pounds of sulfur per acre, while the rates for studies conducted under Project Nos. 73, 255, and 331 were zero, 40 and 160 pounds. Samples of the bulk soils used for the experiments were obtained. After the crops were harvested, samples of soil were taken from each replication of the zero and 100 pound sulfur pots of Project 310. The zero, 40 and 160 pounds sulfur applications for the other studies were part of a 3 x 3 factorial of sulfur and molybdenum. Since three levels of molybdenum were applied for each level of sulfur, composite soil samples from pots containing the different levels of molybdenum but only one level of sulfur were taken after the crops were harvested. Separate composite samples were taken for each replication and level of sulfur.

Results and Discussion

Two separate analyses of the soils obtained from field and greenhouse experiments were made to determine the amounts of sulfate sulfur extracted with sodium acetate solution (pH 4.8). The total levels of sulfate were determined after incubating the soils and the initial levels of sulfate were determined before incubating the soils. The amounts of sulfate sulfur actually released by incubation were derived by subtracting the initial amounts (before incubation) from the total amounts. Thus, soil sulfate sulfur was divided into sulfate released before incubation, sulfate released by incubation, and total sulfate.

The levels of total sulfate, sulfate released before incubation and sulfate released by incubation of soils prior to cropping were each correlated with the yield responses obtained in the field or greenhouse and with the yield of check pots in the greenhouse.[1] The soil series were grouped according to their parent material because of lack of experiments on a specific soil series. The correlations were done separately for the field and greenhouse samples. The greenhouse samples were further divided according to the treatment levels.

Field Samples Before Cropping

The ranges in total sulfate extracted, sulfate prior to incubation and sulfate released by incubation were generally quite low for the three general groups of soils studied; old alluvium, recent alluvium, and residium (Table 6). Total sulfate sulfur ranged from 15.6 to 22.4 ppm, sulfate sulfur released before incubation ranged from 8.6 to 17.2 ppm, and that released by incubation ranged

1 Appreciation is expressed to Dr. R. G. Peterson, Department of Statistics, Oregon State College for the correlations.

from 0.8 to 8.3 ppm. The additional amounts of sulfate released by incubation were smaller than the amounts prior to incubation. The data indicated some tendency for the sulfate released before incubation to be slightly lower for the recent alluvium than for the old alluvium or residual soils. There was also an indication of a relationship between sulfate released before incubation and sulfate released by incubation. (This will be discussed in a later section.)

The yield responses to sulfur in the field experiments on grains for which samples were available were generally negligible (Table 6). The responses associated with the individual samples ranged from 84 to 153 percent.[1] However, only 8 out of 37 of the response values were 110 percent or greater. Only two locations were known to have given statistically significant responses to sulfur applications on grains. It is not known from the data available if the 202 percent response to sulfur of clover grown on residium was statistically significant, although it is likely that it was. In general, the yield increases on residual soils were higher than on the alluvial soils.

Correlation coefficients were calculated on the basis of differences in soil regardless of crops and also for

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[1] Percent yield response is defined as: yield with sulfur treatment x 100 / yield without sulfur treatment.

crops regardless of soils. The correlation coefficients between soil sulfate and crop response were generally low. Only sulfate extracted before incubation of samples from residual soils gave a significant correlation with crop response (-0.868).

From an examination of the data it would appear that the general lack of correlation may have been due to lack of response at most locations, to a generally narrow range of yield response and/or possibly to the narrow range of sulfate soil test values. It is also possible that soil populations need to be defined within more narrow limits than used here. The question of variability between soil series and within these broad groups may be important in this type of correlation study. It is rather difficult to obtain adequate correlation curves without a reasonable range in both response and soil test values. As previously mentioned, this study is exploratory and existing yield data and soil samples were utilized.

Greenhouse Samples Before Cropping

The general orders of magnitude and ranges of the three categories of sulfate extracted from the soils used in greenhouse experiments (Table 7) were approximately the same as for field samples (Table 6). The data do indicate, however, that the range of sulfates extracted from the

Table 6

Responses versus sulfates in soils before cropping, grouped according to parent material of soils regardless of crop type. Field experiments with applications of 20 lbs. S/acre. Soil analysis-averages of duplicate samples.

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response $\left(\frac{\text{S plot}}{\text{check}} \times 100\right)$	ppm Soil Sulfate Sulfur		
					Total	Before Incubation	Released by Incubation
Alluvium, old	16 C[1]	Willamette	Barley	109*	18.9	13.8	5.1
	17 C	Willamette	Barley	98	16.9	14.1	2.8
	18 C	Willamette	Barley	92	16.9	13.9	3.0
	19 R1	Willamette	Barley	85	16.9	12.3	4.6
	20 R2	Willamette	Barley	104	16.3	12.3	4.0
	21 R3	Willamette	Barley	110	16.1	12.9	3.2
	22-24 C	Willamette	Barley	101	16.7	14.6	2.1
	25 R1	Willamette	Barley	99	18.6	10.5	8.1
	26 R2	Willamette	Barley	102	16.9	8.6	8.3
	27 R3	Willamette	Barley	95	16.9	12.3	4.6
	28-30 C	Willamette	Barley	84	17.5	12.8	4.7
	31 R1	Willamette-Amity	Barley	102	16.9	12.3	4.6
	32 R2	Willamette-Amity	Barley	100	18.6	12.3	6.3
	33 R3	Willamette-Amity	Barley	100	16.5	13.6	2.9
	1 R1	Amity	Barley	153	19.8	15.9	3.9
	2 R2	Amity	Barley	94	19.7	16.7	3.0
	3 R1	Amity	Barley	99	20.6	14.4	6.2
	4 R2	Amity	Barley	98	20.7	14.1	6.6
	5 R3	Amity	Barley	100	21.1	14.5	6.6

Table 6 (Continued)

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response $\left(\frac{\text{S plot}}{\text{check}} \times 100\right)$	ppm Soil Sulfate Sulfur Total	Before Incubation	Released by Incubation
Alluvium,	6-8 C	Amity	Oats	124	17.9	15.3	2.6
old	9 C	Carlton	Barley	104	19.1	11.8	7.3
	10 R1	Carlton	Barley	102	19.1	12.2	6.9
	11 R2	Carlton	Barley	99	18.9	13.5	5.4
	12 R3	Carlton	Barley	101	18.6	13.5	5.1
	13-15 C	Dayton	Oats	95	19.1	17.2	1.9
	Correlation Coefficients with Percent Response				0.175	0.239	-0.098
Alluvium,	34 R1	Newberg	Barley	98	16.0	10.5	5.5
recent	35 R2	Newberg	Barley	91	17.3	10.5	6.8
	36 R3	Newberg	Barley	81	16.6	10.2	6.4
	37 R2	Newberg	Barley	95	17.4	14.3	3.1
	38 R3	Newberg	Barley	104	15.7	13.0	2.7
	39-41 C	Wapato	Oats	101	16.6	15.2	1.4
	Correlation Coefficients with Percent Response				-0.455	0.612	-0.749
Residium	42 R1	Aiken	Clover	98	15.9	15.0	0.9
	44-45 C	Laughlin	Clover	107	17.1	13.0	4.1
	46-47 C	Laughlin	Clover	202	15.6	11.0	4.6
	48 R1	Melbourne	Barley	115	15.7	14.9	0.8
	49 R2	Melbourne	Barley	116	15.7	14.3	1.4
	50 R3	Melbourne	Barley	138	15.8	13.7	2.1

Table 6 (Continued)

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response $\left(\frac{\text{S plot}}{\text{check}} \times 100\right)$	ppm Soil Sulfate Sulfur Total	Before Incubation	Released by Incubation
Residium	51-53 C	Melbourne	Barley	136*	19.7	12.6	7.1
	54-56 C	Olympic	Barley	126	22.3	14.1	8.2
	57-59 C	Olympic	Oats	87	22.4	15.1	7.3
	Correlation Coefficients with Percent Response				-0.313	-0.868**	0.095
Grains only:	Correlation Coefficients with Percent Response				0.080	0.252	-0.145
All soils and crops:	Correlation Coefficients with Percent Response				-0.086	0.009	-0.082

1 C, R1, R2, R3. Composite or individual replication samples respectively.

* Significant (5%) response.

** Significant (1%) correlation.

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samples of loess was smaller than in the other types of parent material. Total release of sulfate and sulfate released before incubation were quite low in the Deschutes soil. This is interesting in view of the known responses to sulfur on this soil series.

There were indications of a direct correlation between organic matter content and the levels of total sulfate and sulfate released before incubation in these soils (Table 8). In general, the higher the organic matter content, the greater the amount of sulfates (total release and release before incubation). However, the amount of sulfate released by incubation did not seem to be dependent on the organic matter content. Knappa, Tillamook and Astoria soils, which had relatively high amounts of organic matter (12-18%) had greater amounts of total sulfates and sulfate released before incubation than did Baker and Deschutes soils, which contained about 1-3 percent organic matter. Sulfate released by incubation, however, was less for the Knappa and Tillamook soils than for Deschutes soil.

Correlations of check yields with soil sulfates extracted before cropping in the greenhouse were made. The two soils cropped to clover, although included in Table 7, were not used in these correlation analyses. The correlations of sulfate test values with yield of alfalfa (grams/

Table 7

Yields per check pot versus sulfates in soils before cropping with legumes grouped according to parent material of soils. Greenhouse experiments. Soil analysis-averages of duplicate samples.

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Check Yield (g/pot)	ppm Soil Sulfate Sulfur		
					Total	Before Incubation	Released by Incubation
Alluvium, old	207	Baker	Alfalfa	8.75	13.8	9.9	3.9
	207	Baker	Alfalfa	8.63	13.8	9.9	3.9
	207	Baker	Alfalfa	7.67	13.8	9.9	3.9
	214	Barron	Alfalfa	15.82	15.0	14.3	0.7
	214	Barron	Alfalfa	17.32	15.0	14.3	0.7
	214	Barron	Alfalfa	17.83	15.0	14.3	0.7
	221	Coker	Alfalfa	15.91	14.7	12.0	2.7
	221	Coker	Alfalfa	14.11	14.7	12.0	2.7
	221	Coker	Alfalfa	16.28	14.7	12.0	2.7
	228	Knappa	Alfalfa	10.50	20.9	19.1	1.8
	228	Knappa	Alfalfa	10.05	20.9	19.1	1.8
	228	Knappa	Alfalfa	10.73	20.9	19.1	1.8
	235	Medford	Alfalfa	15.03	16.1	12.8	3.3
	235	Medford	Alfalfa	16.01	16.1	12.8	3.3
	235	Medford	Alfalfa	13.00	16.1	12.8	3.3
	242	Tillamook	Alfalfa	8.10	20.2	18.4	1.8
	242	Tillamook	Alfalfa	6.50	20.2	18.4	1.8
	242	Tillamook	Alfalfa	5.88	20.2	18.4	1.8
	249	Willamette	Alfalfa	19.87	19.0	11.1	7.9
	249	Willamette	Alfalfa	20.48	19.0	11.1	7.9
	249	Willamette	Alfalfa	18.29	19.0	11.1	7.9
	Correlation Coefficients with Check Yields				-0.205	-0.446*	0.425

Table 7 (Continued)

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Check Yield (g/pot)	ppm Soil Sulfate Sulfur		
					Total	Before Incubation	Released by Incubation
Alluvium, recent	256	Chehalis	Alfalfa	11.33	16.5	10.5	6.0
	256	Chehalis	Alfalfa	11.56	16.5	10.5	6.0
	256	Chehalis	Alfalfa	10.97	16.5	10.5	6.0
	263	Powder	Alfalfa	33.77	17.6	9.3	8.3
	263	Powder	Alfalfa	31.39	17.6	9.3	8.3
	263	Powder	Alfalfa	31.72	17.6	9.3	8.3
	270	Sams	Alfalfa	11.23	16.4	10.6	5.8
	270	Sams	Alfalfa	10.48	16.4	10.6	5.8
	270	Sams	Alfalfa	10.28	16.4	10.6	5.8
	277	Wingville	Alfalfa	28.37	25.5	19.4	6.1
	277	Wingville	Alfalfa	22.43	25.5	19.4	6.1
	277	Wingville	Alfalfa	17.99	25.5	19.4	6.1
	284	Clatsop	Clover	25.20	24.4	18.3	6.1
	284	Clatsop	Clover	25.50	24.4	18.3	6.1
	Correlation Coefficients with Check Yields				0.343	0.107	0.853**
Loess	354	Athena	Alfalfa	9.69	14.9	7.4	7.5
	354	Athena	Alfalfa	8.83	14.9	7.4	7.5
	354	Athena	Alfalfa	7.38	14.9	7.4	7.5
	375	Walla Walla	Alfalfa	12.40	17.1	10.0	7.1
	375	Walla Walla	Alfalfa	15.28	17.1	10.0	7.1
	375	Walla Walla	Alfalfa	16.53	17.1	10.0	7.1
	361	Cascade	Alfalfa	16.48	17.1	11.2	5.9
	361	Cascade	Alfalfa	16.43	17.1	11.2	5.9

Table 7 (Continued)

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Check Yield (g/pot)	ppm Soil Sulfate Sulfur		
					Total	Before Incubation	Released by Incubation
Loess	368	Cascade	Alfalfa	14.61	17.7	9.9	7.8
	368	Cascade	Alfalfa	15.77	17.7	9.9	7.8
	Correlation Coefficients with Check Yields				0.900**	0.929**	-0.478
Residium	305	Aiken	Alfalfa	10.86	15.4	8.9	6.5
	305	Aiken	Alfalfa	11.04	15.4	8.9	6.5
	305	Aiken	Alfalfa	11.17	15.4	8.9	6.5
	319	Aiken	Alfalfa	13.60	15.3	12.7	2.6
	319	Aiken	Alfalfa	11.60	15.3	12.7	2.6
	326	Aiken	Alfalfa	24.00	17.8	12.6	5.2
	326	Aiken	Alfalfa	24.50	17.8	12.6	5.2
	333	Aiken	Alfalfa	25.30	17.7	11.0	6.7
	333	Aiken	Alfalfa	30.90	17.7	11.0	6.7
	340	Aiken	Alfalfa	12.90	17.0	11.1	5.9
	340	Aiken	Alfalfa	11.40	17.0	11.1	5.9
	298	Astoria	Alfalfa	15.69	22.1	18.2	3.9
	298	Astoria	Alfalfa	15.52	22.1	18.2	3.9
	298	Astoria	Alfalfa	14.86	22.1	18.2	3.9
	291	Astoria	Clover	22.90	20.2	14.7	5.5
	291	Astoria	Clover	22.30	20.2	14.7	5.5
	Correlation Coefficients with Check Yields				0.221	0.058	0.250

Table 7 (Continued)

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Check Yield (g/pot)	ppm Soil Sulfate Sulfur		
					Total	Before Incubation	Released by Incubation
Pumice	382	Deschutes	Alfalfa	5.66	11.5	7.4	4.1
	382	Deschutes	Alfalfa	6.15	11.5	7.4	4.1
	382	Deschutes	Alfalfa	6.33	11.5	7.4	4.1

** Significant (1%) correlation

* Significant (5%) correlation

Table 8

Amounts of organic matter and sulfate sulfur in soils analyzed before cropping in greenhouse.

Soil Series	% Organic Matter in Soils[1]	ppm Soil Sulfate Sulfur		
		Total	Before Incubation	Released by Incubation
Knappa	18.32	20.9	19.1	1.8
Tillamook	17.64	20.2	18.4	1.8
Astoria	12.80	22.1	18.2	3.9
Wingville	7.54	25.5	19.4	6.1
Aiken	6.83	15.4	8.9	6.5
Barron	5.40	15.0	14.3	0.7
Athena	4.99	14.9	7.4	7.5
Willamette	4.01	19.0	11.1	7.9
Medford	3.33	16.1	12.8	3.3
Sams	2.86	16.4	10.6	5.8
Coker	2.82	14.7	12.0	2.7
Baker	2.80	13.8	9.9	3.9
Walla Walla	2.24	17.1	10.0	7.1
Chehalis	2.19	16.5	10.5	6.0
Powder	2.08	17.6	9.3	8.3
Deschutes	1.57	11.5	7.4	4.1

1 Percent organic matter in soils obtained through the courtesy of T. T. Chao, Department of Soils, Oregon State College.

pot) were generally poor with the exception of total sulfates before incubation on loess soils and sulfates released by incubation on recent alluvium (Table 7). It is somewhat distressing to find that under one condition a particular test appears to be superior but under a different set of conditions or soils the results are opposite. The generally poor correlations for the old alluvium, residium and in some cases the recent alluvium again raises the question of possible variability between series within these broad groupings. Also, it is realized that correlations of a soil test with absolute yield is generally unsatisfactory due to the many variabilities between locations, series and broader soil groupings.

Relative yields or responses are generally more useful for correlation purposes than check yields. The relative yields or responses to sulfur under greenhouse are shown in Tables 9, 10, and 11. These have been correlated with the levels of soil sulfates. Since different rates of sulfur were used in the different experiments, it was necessary to separate the responses on the basis of amounts of sulfur applied.

The correlations of the soil tests with responses in experiments conducted under Projects 73, 255 and 331 were very low for both the 40 (Table 9) and the

160 (Table 10) pound treatments. The low correlations are undoubtedly influenced by the small ranges in response and/or soil test values. Although the responses to 40 and 160 pounds of sulfur on loess soils were significant, the responses to 40 pounds were quite similar for the two soils, while the range in response to 160 pounds was larger. The larger range in response to 160 pounds sulfur may have contributed to the higher correlations. However, it is felt that the data for the loess soils are not too useful in view of the small number of soils in the group. Only one residual soil gave a significant response to sulfur applications, and only at the 40 pound rate. The ranges in response were also quite small for both rates.

The yield data from the experiment conducted under Project 310 were much more useful for correlation purposes. The number of soils in this experiment was larger and the ranges in response to sulfur for the different groups of soils were considerably larger (Table 11). Significant responses were observed on 11 of the 16 soils tested. The responses for sulfur treated pots relative to the check pots ranged from 90 to better than 400 percent. Relatively high correlation coefficients were obtained for sulfate soil tests versus response for these soils. Generally, the correlations for total sulfate and sulfate before incubation were better than that for sulfate released by incubation. Three of the coefficients for sulfate released by incubation were positive which indicates more response to sulfur applications when the sulfate release values are higher. This is difficult to interpret. The other two soil tests, however, gave quite encouraging results. For the old alluvial soils (21 samples representing seven different soils with three replications each) the correlations for total sulfate and sulfate extracted before incubation were -0.725 and -0.712 respectively. For soil testing purposes this is generally regarded as fairly good. For the recent alluvium, the correlations were -0.672 and -0.631 for total sulfate and sulfate released by incubation, respectively. The correlation coefficient (-0.470) for sulfate prior to incubation was not significant. The correlations for residium was best with a value of -0.999. However, the number of soils or samples in this group was less than what would be desired for more critical correlation analyses. Although the correlation coefficients were not determined for the pumice category since only one soil (Deschutes) was available, the data show that response was greatest on this soil while total sulfate and

Table 9

Responses of legumes versus sulfates in soils before cropping, grouped according to parent material of soils. Greenhouse experiments with application of 40 lbs. S/acre.

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response $\left(\frac{\text{S pot}}{\text{check}} \times 100\right)$	ppm Soil Sulfate Sulfur		
					Total	Before Incubation	Released by Incubation
Alluvium, Recent Loess	284	Clatsop	Clover	104	24.4	18.3	6.1
	284	Clatsop	Clover	111	24.4	18.3	6.1
	361	Cascade	Alfalfa	116 } **	17.1	11.2	5.9
	361	Cascade	Alfalfa	123 }	17.1	11.2	5.9
	368	Cascade	Alfalfa	116 } **	17.7	9.9	7.8
	368	Cascade	Alfalfa	124 }	17.7	9.9	7.8
	Correlation Coefficients with Percent Response				0.066	-0.066	0.066
Residium	319	Aiken	Alfalfa	112 } *	15.3	12.7	2.6
	319	Aiken	Alfalfa	127 }	15.3	12.7	2.6
	326	Aiken	Alfalfa	105	17.8	12.6	5.2
	326	Aiken	Alfalfa	104	17.8	12.6	5.2
	333	Aiken	Alfalfa	114	17.7	11.0	6.7
	333	Aiken	Alfalfa	97	17.7	11.0	6.7
	340	Aiken	Alfalfa	96	17.0	11.1	5.9
	340	Aiken	Alfalfa	80	17.0	11.1	5.9
	291	Astoria	Clover	100	20.2	14.7	5.5
	291	Astoria	Clover	103	20.2	14.7	5.5
	Correlation Coefficients with Percent Response				-0.346	0.222	-0.611

* Significant (5%) response

** Significant (1%) response

Table 10

Responses of legumes versus sulfates in soils before cropping, grouped according to parent material. Greenhouse experiments with application of 160 lbs. S/ acre.

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response ($\frac{\text{S pot}}{\text{Check}}$ x 100)	ppm Soil Sulfate Sulfur		
					Total	Before Incubation	Released by Incubation
Alluvium,	284	Clatsop	Clover	106	24.4	18.3	6.1
Recent	284	Clatsop	Clover	105	24.4	18.3	6.1
Loess	361	Cascade	Alfalfa	131 **	17.1	11.2	5.9
	361	Cascade	Alfalfa	131	17.1	11.2	5.9
	368	Cascade	Alfalfa	118 *	17.7	9.9	7.8
	368	Cascade	Alfalfa	114	17.7	9.9	7.8
	Correlation Coefficients with Percent Response				-0.983**	0.983**	-0.983**
Residium	319	Aiken	Alfalfa	100	15.3	12.7	2.6
	319	Aiken	Alfalfa	104	15.3	12.7	2.6
	326	Aiken	Alfalfa	110	17.8	12.6	5.2
	326	Aiken	Alfalfa	104	17.8	12.6	5.2
	333	Aiken	Alfalfa	108	17.7	11.0	6.7
	333	Aiken	Alfalfa	102	17.7	11.0	6.7
	340	Aiken	Alfalfa	91	17.0	11.1	5.9
	340	Aiken	Alfalfa	86	17.0	11.1	5.9
	291	Astoria	Clover	97	20.2	14.7	5.5
	291	Astoria	Clover	97	20.2	14.7	5.5
	Correlation Coefficients with Percent Response				-0.065	0.065	-0.137

* Significant (5%) response

** Significant (1%) response or correlation

Table 11

Responses of legumes versus sulfates in soil before cropping, grouped according to parent material of soils. Greenhouse experiments with application of 100 lbs. S/acre.

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response $\left(\frac{\text{S pot}}{\text{Check}} \times 100\right)$	ppm Soil Sulfate Sulfur: Total	ppm Soil Sulfate Sulfur: Before Incubation	ppm Soil Sulfate Sulfur: Released by Incubation
Alluvium, Old	207	Baker	Alfalfa	386	13.8	9.9	3.9
	207	Baker	Alfalfa	419 **	13.8	9.9	3.9
	207	Baker	Alfalfa	421	13.8	9.9	3.9
	214	Barron	Alfalfa	189	15.0	14.3	0.7
	214	Barron	Alfalfa	183 **	15.0	14.3	0.7
	214	Barron	Alfalfa	167	15.0	14.3	0.7
	221	Coker	Alfalfa	149	14.7	12.0	2.7
	221	Coker	Alfalfa	185 **	14.7	12.0	2.7
	221	Coker	Alfalfa	153	14.7	12.0	2.7
	228	Knappa	Alfalfa	113	20.9	19.1	1.8
	228	Knappa	Alfalfa	109	20.9	19.1	1.8
	228	Knappa	Alfalfa	105	20.9	19.1	1.8
	235	Medford	Alfalfa	249	16.1	12.8	3.3
	235	Medford	Alfalfa	201 **	16.1	12.8	3.3
	235	Medford	Alfalfa	259	16.1	12.8	3.3
	242	Tillamook	Alfalfa	90	20.2	18.4	1.8
	242	Tillamook	Alfalfa	94	20.2	18.4	1.8
	242	Tillamook	Alfalfa	109	20.2	18.4	1.8
	249	Willamette	Alfalfa	158	19.0	11.1	7.9
	249	Willamette	Alfalfa	160 **	19.0	11.1	7.9
	249	Willamette	Alfalfa	154	19.0	11.1	7.9
	Correlation Coefficients with Percent Response				-0.725**	-0.712**	0.192

Table 11 (Continued)

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response $\left(\frac{S\ pot}{Check} \times 100\right)$	ppm Soil Sulfate Sulfur Total	ppm Soil Sulfate Sulfur Before Incubation	ppm Soil Sulfate Sulfur Released by Incubation
Alluvium, Recent	256	Chehalis	Alfalfa	281)	16.5	10.5	6.0
	256	Chehalis	Alfalfa	259)**	16.5	10.5	6.0
	256	Chehalis	Alfalfa	285)	16.5	10.5	6.0
	263	Powder	Alfalfa	96	17.6	9.3	8.3
	263	Powder	Alfalfa	117	17.6	9.3	8.3
	263	Powder	Alfalfa	108	17.6	9.3	8.3
	270	Sams	Alfalfa	285)	16.4	10.6	5.8
	270	Sams	Alfalfa	317)**	16.4	10.6	5.8
	270	Sams	Alfalfa	324)	16.4	10.6	5.8
	277	Wingville	Alfalfa	98	25.5	19.4	6.1
	277	Wingville	Alfalfa	101	25.5	19.4	6.1
	277	Wingville	Alfalfa	114	25.5	19.4	6.1
	Correlation Coefficients with Percent Response				-0.672*	-0.470	-0.631
Loess	354	Athena	Alfalfa	240)	14.9	7.4	7.5
	354	Athena	Alfalfa	281)**	14.9	7.4	7.5
	354	Athena	Alfalfa	347)	14.9	7.4	7.5
	375	Walla Walla	Alfalfa	248)	17.1	10.0	7.1
	375	Walla Walla	Alfalfa	218)**	17.1	10.0	7.1
	375	Walla Walla	Alfalfa	141)	17.1	10.0	7.1
	Correlation Coefficients with Percent Response				-0.698	-0.698	0.698

Table 11 (Continued)

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Percent Yield Response $\left(\frac{S\ pot}{Check} \times 100\right)$	ppm Soil Sulfate Sulfur Total	ppm Soil Sulfate Sulfur Before Incubation	ppm Soil Sulfate Sulfur Released by Incubation
Residium	305	Aiken	Alfalfa	164 } **	15.4	8.9	6.5
	305	Aiken	Alfalfa	160 } **	15.4	8.9	6.5
	305	Aiken	Alfalfa	160 } **	15.4	8.9	6.5
	298	Astoria	Alfalfa	105	22.1	18.2	3.9
	298	Astoria	Alfalfa	104	22.1	18.2	3.9
	298	Astoria	Alfalfa	106	22.1	18.2	3.9
	Correlation Coefficients with Percent Response				-0.999**	-0.999**	0.999**
Pumice	382	Deschutes	Alfalfa	464 } **	11.5	7.4	4.1
	382	Deschutes	Alfalfa	416 } **	11.5	7.4	4.1
	382	Deschutes	Alfalfa	445 } **	11.5	7.4	4.1
All Soils:	Correlation Coefficients with Percent Response				-0.736**	-0.651**	0.053

* Significant (5%) correlation

** Significant (1%) correlation or response sulfate before incubation was lowest. When all of the 16 soils were grouped together, correlation coefficients of -0.736 for total sulfate and -0.651 for sulfate extracted before incubation were obtained. The relationship between response and total sulfate and sulfate released before incubation are shown in Figures 2 and 3. After plotting the points, visual examination of the graphs indicated that the relations may be expressed better by curvilinear regression rather than linear regression, although the latter was significant. Significant correlation coefficients (0.814 and 0.661) for the curvilinear relationship of response to total sulfate and sulfate before incubation were obtained. Although the soils were quite variable within this broad grouping the large ranges in response and soil test values undoubtedly contributed to fairly good correlation studies. It would appear from these data that if proper response data and groups of soil are available, a soil test for sulfate may offer some promise.

Greenhouse Samples After Cropping

Initially, it was decided to sample experiments conducted in the greenhouse after treatments were applied and several harvests were made. It was felt that this would provide an opportunity to test whether the sulfate soil tests would detect differences in previous sulfur

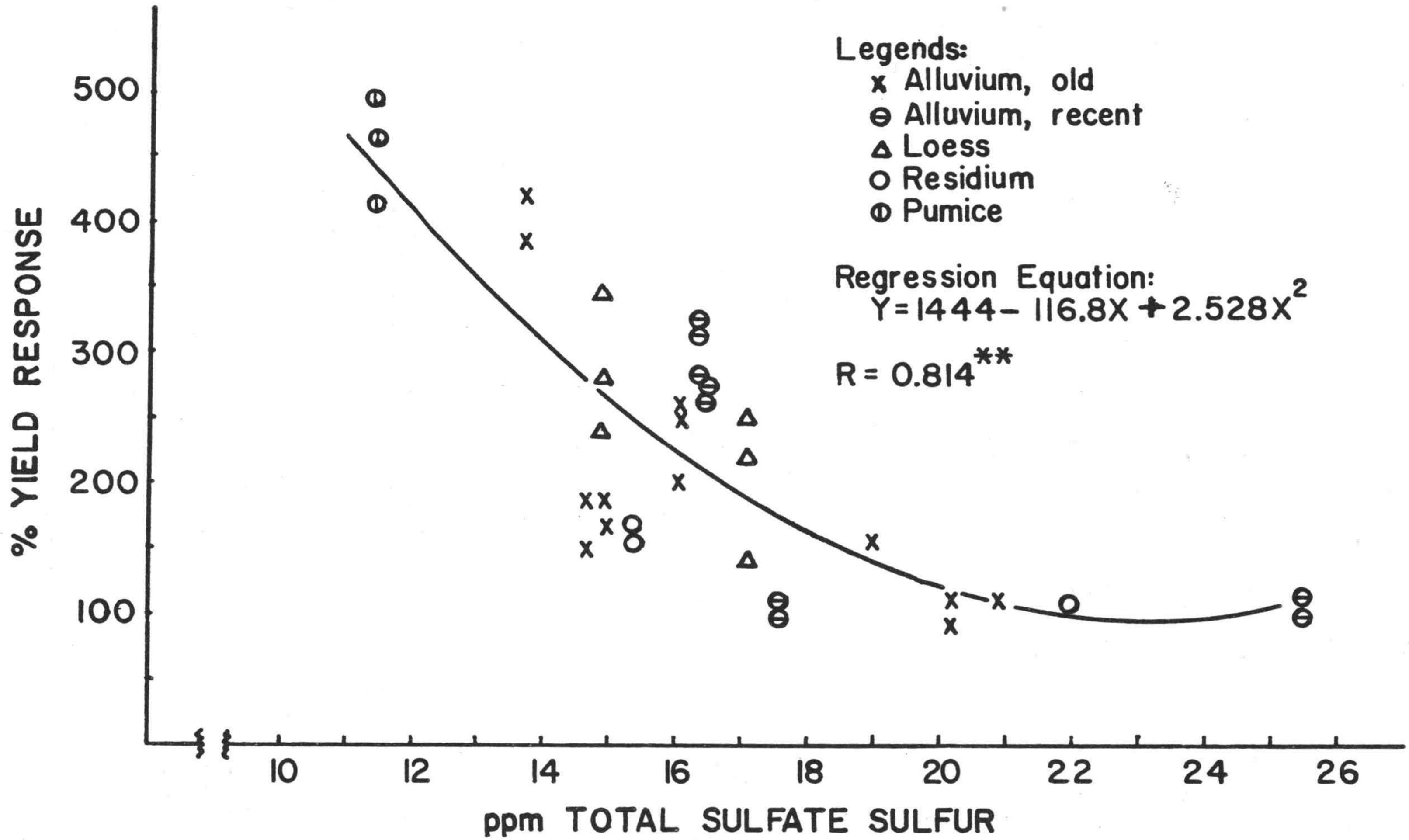

Figure 2. Relation of alfalfa responses from 100 lbs. sulfur per acre to total sulfate sulfur in soils. Samples from greenhouse experiments prior to cropping.

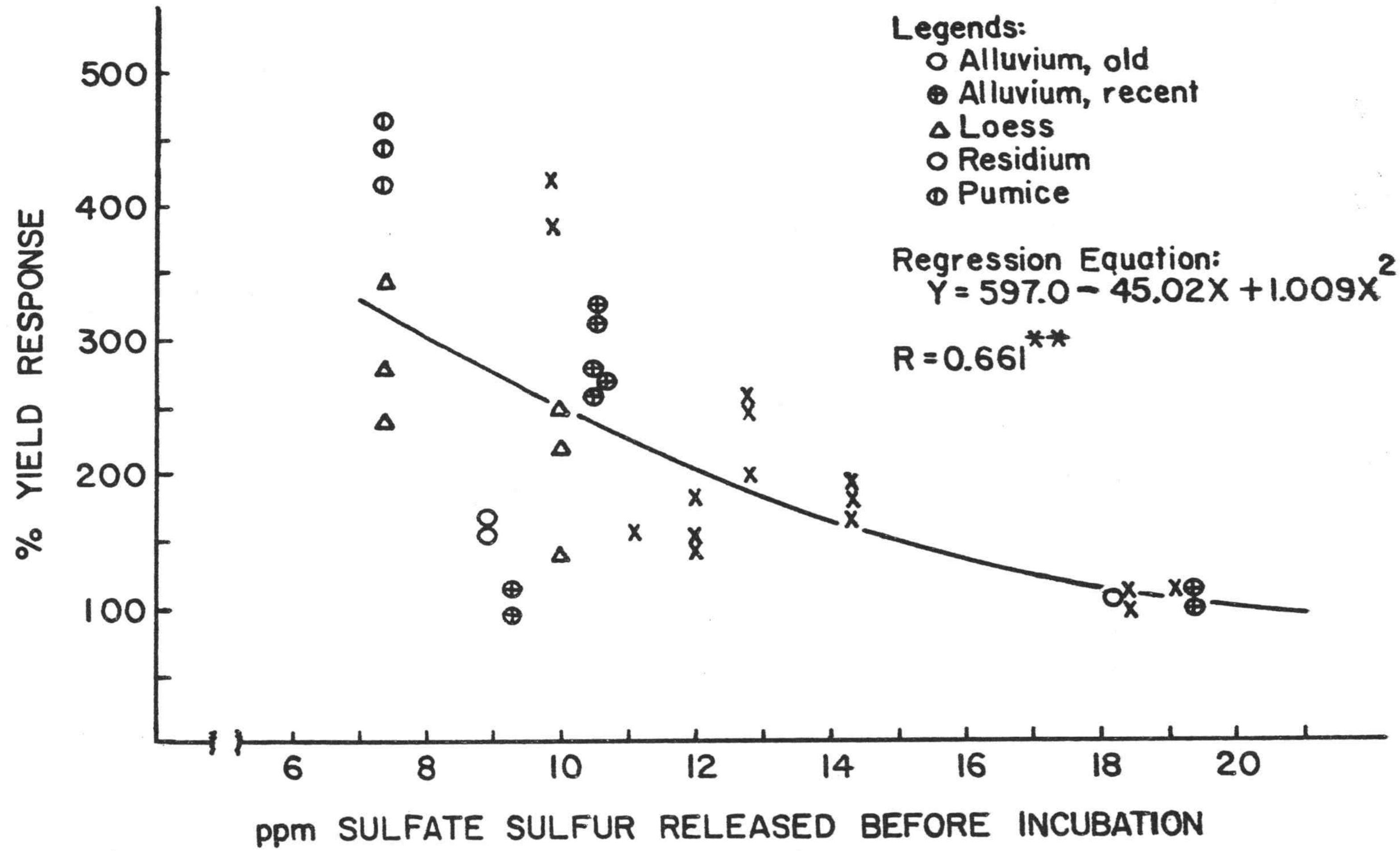

Figure 3. Relation of alfalfa responses from 100 lbs. sulfur per acre to sulfate sulfur released before incubation of soils. Samples from greenhouse experiments prior to cropping.

applications or management. The data in Table 12 show that there was very little relationship between soil test values for the pots which received sulfur applications as compared to those which did not receive any sulfur. It would appear that successive cropping in the greenhouse was sufficient to remove the influence of previous sulfur treatments. This is consistent with the data of T. T. Chao[1] who has observed that the sulfur contents of alfalfa for some of these treatments decreased markedly with successive cuttings and by the fourth cutting, the sulfur contents of the plants were all quite low.

Sulfate Released Before Incubation Versus Sulfate Released by Incubation

Examination of the soil test values seemed to indicate that sulfate released by incubation may be inversely related to sulfate released before incubation. Therefore, sulfates released before incubation were correlated with sulfates released by incubation, with the soils grouped according to their parent material. Separate correlations were calculated for field samples, greenhouse samples before cropping and greenhouse samples after cropping. The greenhouse samples after cropping were further divided according to the levels of sulfur treatments. The results

[1] Personal communication. Unpublished data of T. T. Chao, Soils Department, Oregon State College, Corvallis, Oregon.

(Table 13) show that fairly good correlations were obtained. The negative value of the correlation coefficient means that the two forms of sulfate were inversely related; that is, soil sulfates released by incubation decreased as the sulfates extracted before incubation increased. The relationship for samples from greenhouse experiments prior to cropping are shown in Figure 4. Unlike the correlations between response and soil sulfates shown in Figure 2 and 3, the regression in Figure 4 was not curvilinear.

Since the available sulfates present in the soil were not extracted before the soils were incubated, the sulfates in the soil may have depressed microbial activities during the incubation. The depression may have been greater when the level of available sulfate was higher. On the other hand, preliminary studies on time of incubation did not suggest that there was any depressive effect of amounts of sulfate released in four weeks as compared to three weeks. At that stage of the investigation it was assumed that if there were any depressive effects of sulfate accumulation, they would be manifested in the studies on time of incubation. This problem may be worthy of further investigation.

Table 12

Yields per pot versus sulfates in soils after cropping with legumes, grouped according to parent material of soils. Greenhouse experiments with applications of 0, 40, 100 and 160 lbs. S/acre. Sulfate analysis-averages of duplicate samples.

Parent Material of Soil	Sample Number	Soil Series	Specific Crop	Pounds S Applied	Yield (g/pot)	ppm Soil Sulfate Sulfur		
						Total	Before Incubation	Released by Incubation
Alluvium, Old	201	Baker	Alfalfa	0	8.75	11.7	7.4	4.3
	02	Baker	Alfalfa	0	8.63	11.7	7.4	4.3
	03	Baker	Alfalfa	0	7.67	11.8	7.4	4.4
	04	Baker	Alfalfa	100	33.73	11.8	7.4	4.4
	05	Baker	Alfalfa	100	36.13	13.8	7.4	6.4
	06	Baker	Alfalfa	100	32.28	13.7	9.8	3.9
	208	Barron	Alfalfa	0	15.82	14.9	13.6	1.3
	09	Barron	Alfalfa	0	17.32	14.9	14.5	0.4
	10	Barron	Alfalfa	0	17.83	14.9	12.3	2.6
	11	Barron	Alfalfa	100	29.85	14.9	14.2	0.7
	12	Barron	Alfalfa	100	31.64	14.9	13.6	1.3
	13	Barron	Alfalfa	100	29.84	14.9	14.2	0.7
	215	Coker	Alfalfa	0	15.91	14.7	12.1	2.6
	16	Coker	Alfalfa	0	14.11	14.7	12.1	2.6
	17	Coker	Alfalfa	0	16.28	14.7	12.1	2.6
	18	Coker	Alfalfa	100	23.69	14.8	12.1	2.7
	19	Coker	Alfalfa	100	26.14	14.8	12.1	2.7
	20	Coker	Alfalfa	100	24.83	14.9	12.2	2.7
	222	Knappa	Alfalfa	0	10.50	20.8	19.0	1.8
	23	Knappa	Alfalfa	0	10.05	20.8	19.0	1.8
	24	Knappa	Alfalfa	0	10.73	21.0	19.2	1.8
	25	Knappa	Alfalfa	100	11.90	21.1	18.6	2.5
	26	Knappa	Alfalfa	100	10.99	20.5	18.0	2.5
	27	Knappa	Alfalfa	100	11.29	20.8	19.0	1.8

Table 12 (Continued)

Parent Material of Soils	Sample Number	Soil Series	Specific Crop	Pounds S Applied	Yield (g/pot)	ppm Soil Sulfate Sulfur Total	ppm Soil Sulfate Sulfur Before Incubation	ppm Soil Sulfate Sulfur Released by Incubation
Alluvium, Old	229	Medford	Alfalfa	0	15.03	14.7	12.2	2.5
	30	Medford	Alfalfa	0	16.01	14.7	12.8	1.7
	31	Medford	Alfalfa	0	13.00	16.0	14.6	1.4
	32	Medford	Alfalfa	100	37.35	16.1	12.2	3.8
	33	Medford	Alfalfa	100	32.16	16.1	11.0	5.1
	34	Medford	Alfalfa	100	33.70	16.1	12.0	4.1
	236	Tillamook	Alfalfa	0	8.10	19.9	15.6	4.3
	37	Tillamook	Alfalfa	0	6.50	20.0	16.3	3.7
	38	Tillamook	Alfalfa	0	5.88	20.5	15.7	4.8
	39	Tillamook	Alfalfa	100	7.27	20.9	17.0	3.9
	40	Tillamook	Alfalfa	100	6.10	21.3	17.1	4.2
	41	Tillamook	Alfalfa	100	6.42	20.8	17.0	3.8
	243	Willamette	Alfalfa	0	19.87	18.9	8.6	10.3
	44	Willamette	Alfalfa	0	20.48	19.5	8.6	10.9
	45	Willamette	Alfalfa	0	18.29	19.0	11.1	7.9
	46	Willamette	Alfalfa	100	31.46	18.8	10.4	8.4
	47	Willamette	Alfalfa	100	32.70	18.9	11.0	7.9
	48	Willamette	Alfalfa	100	28.08	17.9	10.4	7.5
Alluvium, Recent	250	Chehalis	Alfalfa	0	11.33	16.9	7.4	9.5
	51	Chehalis	Alfalfa	0	11.56	16.9	8.6	8.3
	52	Chehalis	Alfalfa	0	10.97	16.9	8.6	8.3
	53	Chehalis	Alfalfa	100	31.82	16.9	10.5	6.4
	54	Chehalis	Alfalfa	100	29.89	17.7	8.6	9.1
	55	Chehalis	Alfalfa	100	31.26	18.6	9.9	8.7
	257	Powder	Alfalfa	0	33.77	17.2	9.8	7.4
	58	Powder	Alfalfa	0	31.39	17.2	9.2	8.0
	59	Powder	Alfalfa	0	31.72	17.6	8.0	9.6

Table 12 (Continued)

Parent Material of Soils	Sample Number	Soil Series	Specific Crop	Pounds S Applied	Yield (g/pot)	ppm Soil Sulfate Sulfur Total	ppm Soil Sulfate Sulfur Before Incubation	ppm Soil Sulfate Sulfur Released by Incubation
Alluvium, Recent	260	Powder	Alfalfa	100	32.51	16.8	7.4	9.4
	61	Powder	Alfalfa	100	36.88	18.8	8.6	10.2
	62	Powder	Alfalfa	100	34.23	17.3	7.4	9.9
	264	Sams	Alfalfa	0	11.23	17.5	7.5	10.0
	65	Sams	Alfalfa	0	10.48	17.1	7.5	9.6
	66	Sams	Alfalfa	0	10.28	16.2	8.0	8.2
	67	Sams	Alfalfa	100	31.95	16.3	4.9	11.4
	68	Sams	Alfalfa	100	33.17	16.0	9.3	6.7
	69	Sams	Alfalfa	100	33.34	16.0	12.4	3.6
	271	Wingville	Alfalfa	0	28.37	23.9	18.3	5.6
	72	Wingville	Alfalfa	0	22.43	23.9	19.9	4.0
	73	Wingville	Alfalfa	0	17.99	24.8	20.0	4.8
	74	Wingville	Alfalfa	100	27.85	24.8	18.1	6.7
	75	Wingville	Alfalfa	100	22.61	24.8	19.4	5.0
	76	Wingville	Alfalfa	100	20.52	25.4	19.9	5.5
	278	Clatsop	Clover	0	25.20	17.1	10.9	6.2
	79	Clatsop	Clover	0	25.50	17.5	10.3	7.2
	80	Clatsop	Clover	40	26.20	19.9	13.9	6.0
	81	Clatsop	Clover	40	28.30	19.2	12.8	6.4
	82	Clatsop	Clover	160	26.60	19.7	17.0	2.7
	83	Clatsop	Clover	160	26.90	22.6	15.5	7.1
Loess	348	Athena	Alfalfa	0	9.69	13.9	7.4	6.5
	49	Athena	Alfalfa	0	8.83	13.7	9.3	4.3
	50	Athena	Alfalfa	0	7.38	14.8	7.5	6.3
	51	Athena	Alfalfa	100	23.22	14.7	9.3	5.4
	52	Athena	Alfalfa	100	24.80	14.9	7.4	7.5
	53	Athena	Alfalfa	100	25.64	16.2	7.4	8.8

Table 12 (Continued

Parent Material of Soils	Sample Number	Soil Series	Specific Crop	Pounds S Applied	Yield (g/pot)	ppm Soil Sulfate Sulfur		
						Total	Before Incubation	Released by Incubation
Loess	355	Cascade	Alfalfa	0	16.48	16.3	9.9	6.4
	56	Cascade	Alfalfa	0	16.43	16.3	10.5	5.8
	57	Cascade	Alfalfa	40	17.96	16.2	10.5	5.7
	58	Cascade	Alfalfa	40	20.22	16.2	9.2	7.0
	59	Cascade	Alfalfa	160	21.59	17.0	13.9	3.1
	60	Cascade	Alfalfa	160	21.48	16.6	7.4	9.2
	362	Cascade	Alfalfa	0	14.61	15.8	6.7	9.1
	63	Cascade	Alfalfa	0	15.77	16.2	8.3	7.9
	64	Cascade	Alfalfa	40	16.97	16.3	6.8	9.5
	65	Cascade	Alfalfa	40	19.50	16.8	12.3	4.5
	66	Cascade	Alfalfa	160	17.28	17.8	13.0	4.8
	67	Cascade	Alfalfa	160	18.04	18.8	9.9	8.9
	369	Walla Walla	Alfalfa	0	12.40	16.7	0.0	16.7
	70	Walla Walla	Alfalfa	0	15.28	16.2	0.0	16.2
	71	Walla Walla	Alfalfa	0	16.53	16.7	0.0	16.7
	72	Walla Walla	Alfalfa	100	30.69	17.0	6.8	10.2
	73	Walla Walla	Alfalfa	100	33.32	17.1	6.8	10.3
	74	Walla Walla	Alfalfa	100	23.27	19.4	9.9	9.5
Residium	286	Astoria	Clover	0	22.3	20.5	14.4	6.1
	87	Astoria	Clover	40	22.9	20.6	16.4	4.2
	88	Astoria	Clover	40	23.0	19.8	15.7	4.1
	90	Astoria	Clover	160	21.7	22.5	17.5	5.0
	292	Astoria	Alfalfa	0	15.69	19.5	14.9	4.6
	93	Astoria	Alfalfa	0	15.52	19.6	17.6	2.0
	94	Astoria	Alfalfa	0	14.86	19.7	15.0	4.7

Table 12 (Continued)

Parent Material of Soils	Sample Number	Soil Series	Specific Crop	Pounds S Applied	Yield (g/pot)	ppm Soil Sulfate Sulfur Total	ppm Soil Sulfate Sulfur Before Incubation	ppm Soil Sulfate Sulfur Released by Incubation
Residium	295	Astoria	Alfalfa	100	16.46	20.3	17.2	3.1
	96	Astoria	Alfalfa	100	16.16	20.4	17.6	2.8
	97	Astoria	Alfalfa	100	15.74	20.5	17.4	3.1
	299	Aiken	Alfalfa	0	10.86	21.5	10.0	11.5
	300	Aiken	Alfalfa	0	11.04	19.9	9.8	10.1
	01	Aiken	Alfalfa	0	11.17	17.4	10.8	6.6
	02	Aiken	Alfalfa	100	17.83	17.5	8.9	8.6
	03	Aiken	Alfalfa	100	17.71	17.3	8.9	8.4
	04	Aiken	Alfalfa	100	17.85	17.7	6.4	11.3
	306	Aiken	Alfalfa	0	16.6	15.2	6.3	8.9
	07	Aiken	Alfalfa	0	17.8	16.6	8.8	7.8
	08	Aiken	Alfalfa	40	23.1	14.2	6.3	7.9
	09	Aiken	Alfalfa	40	20.9	16.7	8.9	7.8
	10	Aiken	Alfalfa	160	24.6	13.6	9.8	3.8
	11	Aiken	Alfalfa	160	20.8	13.6	8.4	5.2
	313	Aiken	Alfalfa	0	13.6	16.0	8.8	7.2
	14	Aiken	Alfalfa	0	11.6	16.7	10.8	5.9
	15	Aiken	Alfalfa	40	15.2	16.7	12.7	4.0
	16	Aiken	Alfalfa	40	14.7	16.7	13.3	3.4
	17	Aiken	Alfalfa	160	13.6	50.0	14.5	35.5
	18	Aiken	Alfalfa	160	12.1	46.5	14.0	32.5
	320	Aiken	Alfalfa	0	24.0	16.9	12.8	4.1
	21	Aiken	Alfalfa	0	24.5	16.5	12.6	3.9
	22	Aiken	Alfalfa	40	25.1	17.5	14.6	2.9
	23	Aiken	Alfalfa	40	25.5	17.5	14.6	2.9
	24	Aiken	Alfalfa	160	26.4	18.3	10.8	7.5
	25	Aiken	Alfalfa	160	25.6	19.7	12.7	7.0

Table 12 (Continued)

Parent Material of Soils	Sample Number	Soil Series	Specific Crop	Pounds S Applied	Yield (g/pot)	ppm Soil Sulfate Sulfur		
						Total	Before Incubation	Released by Incubation
Residium	327	Aiken	Alfalfa	0	25.3	15.1	12.5	2.6
	28	Aiken	Alfalfa	0	30.9	16.5	13.8	2.7
	29	Aiken	Alfalfa	40	28.9	17.0	9.4	7.6
	30	Aiken	Alfalfa	40	29.9	19.7	10.6	9.1
	31	Aiken	Alfalfa	160	27.2	19.4	12.6	6.8
	32	Aiken	Alfalfa	160	31.6	18.0	10.7	7.3
	334	Aiken	Alfalfa	0	12.9	15.2	6.2	9.0
	35	Aiken	Alfalfa	0	11.4	15.2	8.6	6.4
	36	Aiken	Alfalfa	40	12.4	15.9	8.6	7.3
	37	Aiken	Alfalfa	40	9.1	16.5	8.6	7.9
	38	Aiken	Alfalfa	160	11.8	17.8	15.2	2.6
	39	Aiken	Alfalfa	160	9.8	20.0	15.7	4.3
	341	Aiken	Alfalfa	0	17.1	15.9	11.1	4.8
	42	Aiken	Alfalfa	0	16.4	15.9	9.8	6.1
	43	Aiken	Alfalfa	40	17.4	17.7	9.9	7.8
	44	Aiken	Alfalfa	40	16.5	17.6	11.7	5.9
	45	Aiken	Alfalfa	160	17.5	27.4	15.4	12.0
	46	Aiken	Alfalfa	160	17.3	37.3	16.0	21.3
Pumice	376	Deschutes	Alfalfa	0	5.66	16.7	6.7	10.0
	77	Deschutes	Alfalfa	0	6.15	16.2	6.8	9.4
	78	Deschutes	Alfalfa	0	6.33	16.1	6.7	9.4
	79	Deschutes	Alfalfa	100	26.27	16.8	8.6	8.2
	80	Deschutes	Alfalfa	100	25.61	16.8	9.5	7.3
	81	Deschutes	Alfalfa	100	28.14	16.9	8.6	8.3

Table 13

Regression correlation coefficients between sulfate released before incubation and sulfate released by incubation.

Groups	Location of Corresponding Data	Pounds S Applied	Sample Size, n	Correlation Coefficient, r
Field Experiments, before cropping				
Alluvium, old	Table 6	0	25	-0.681**
Alluvium, recent	Table 6	0	6	-0.952**
Residium	Table 6	0	9	-0.261
Greenhouse Experiments, before cropping				
Alluvium, old	Table 7	0	7	-0.592
Alluvium, recent	Table 7	0	5	-0.415
Loess	Table 7	0	4	-0.648
Residium	Table 7	0	7	-0.572
All Soils	Table 7	0	24	-0.472*
Greenhouse Experiments, after cropping				
Alluvium, old	Table 12	0	21	-0.542*
Alluvium, recent	Table 12	0	14	-0.920**
Loess	Table 12	0	10	-0.974**
Residium	Table 12	0	19	-0.739**
Loess	Table 12	40	4	-0.995**
Residium	Table 12	40	14	-0.851**
Loess	Table 12	160	4	-0.948**
Residium	Table 12	160	13	0.266

Table 13 (Continued)

Groups	Location of Corresponding Data	Pounds S Applied	Sample Size, n	Correlation Coefficient, r
Greenhouse Experiments, after cropping				
Alluvium, old	Table 12	100	21	-0.528*
Alluvium, recent	Table 12	100	12	-0.743**
Loess	Table 12	100	6	-0.467
Residium	Table 12	100	6	-0.994**

* Significant (5%) correlation

** Significant (1%) correlation

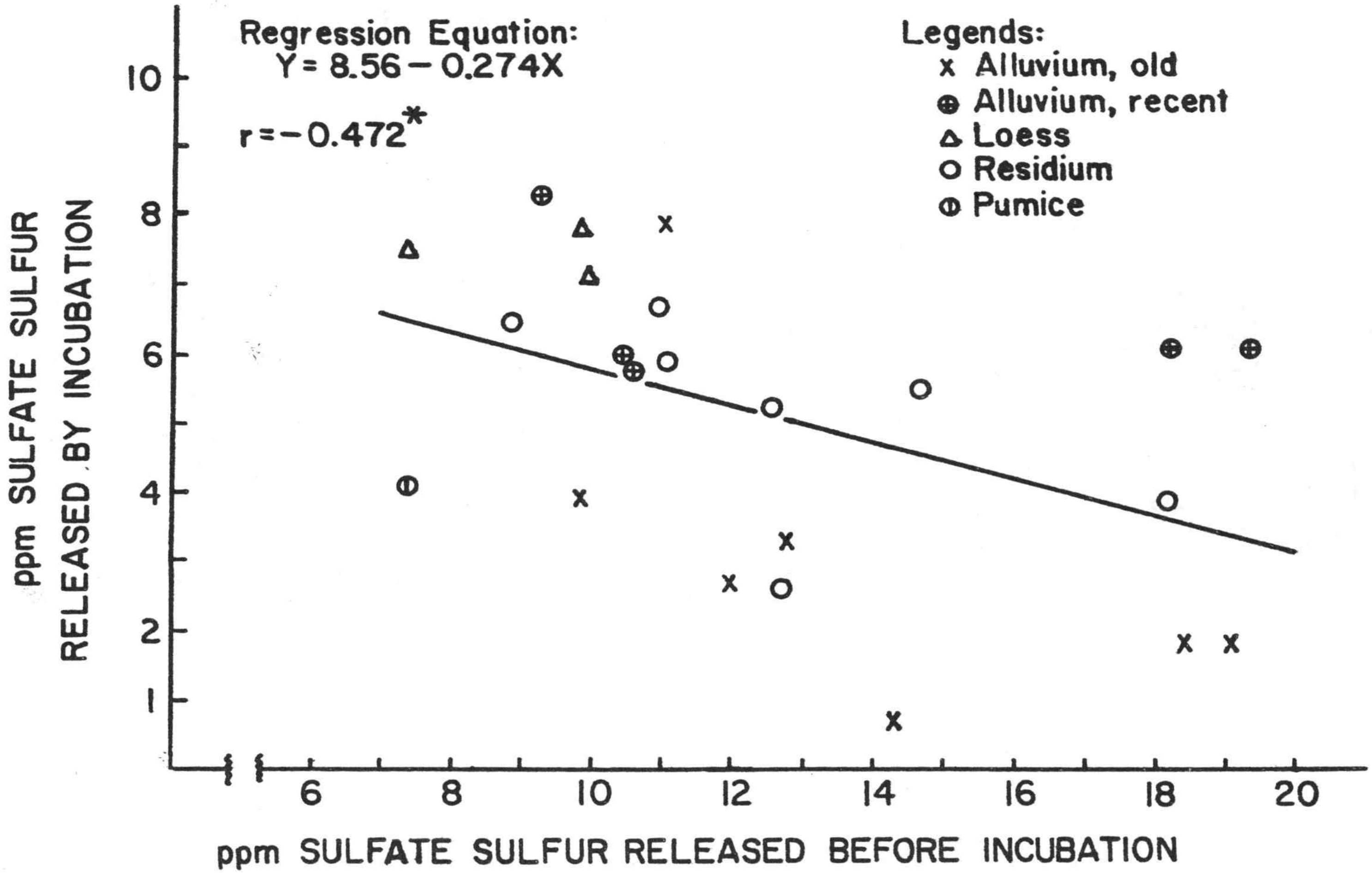

Figure 4. Relation of sulfate sulfur released by incubation to sulfate sulfur released before incubation of soils. Samples from greenhouse experiments prior to cropping.

SUMMARY AND CONCLUSIONS

Although numerous crops in many areas of Oregon and the rest of this country are known to respond to sulfur fertilization, there is no universally accepted method of soil analysis to detect sulfur deficiencies. However, nitrifiable nitrogen has been used successfully in Iowa (64) to predict the nitrogen needs of corn. Since sulfur and nitrogen are similarly related to soil organic matter, a preliminary study was conducted to determine the relationship of sulfate released before incubation and sulfate released by incubation with crop responses to sulfur and to get an indication of the possibility of using such soil-tests to predict the sulfur requirements of crops.

The Iowa method (64) of nitrifying soil organic nitrogen was initially investigated to determine whether a similar method could be employed for the mineralization of organic sulfur in soils. The investigations indicated that the vermicullite employed to increase the moisture holding capacity and porosity of the soils during incubation should be treated to remove sulfates before using. Activated charcoal should also be treated to remove sulfates before using.

Numerous soil samples obtained from field and greenhouse experiments where sulfur was a variable were analyzed for sulfate sulfur by a method similar to that proposed by

Chesnin and Yien (13). Another portion of the samples were incubated at 35° C. After four weeks of incubation, the samples were analyzed for total release of sulfate. The actual release of sulfate due to mineralization was derived by subtracting the "available" sulfate (sulfate before incubation) from the total sulfate. The analyses indicated that the amount of sulfate produced by mineralization was generally lower than that extracted prior to incubation.

There were indications of a direct correlation between organic matter content and the levels of total sulfate and sulfate released before incubation for some of the soils used in greenhouse studies. However, the amount of sulfate released by incubation was not dependent on the organic matter content.

Correlations of the three forms of soil sulfate with crop yields or responses were generally poor for the samples derived from field experiments. However, the correlations were generally better for the greenhouse samples tested before they were cropped to legumes. Most of the poor correlations occurred where the ranges in crop response were narrow, where the number of significant response was limited and/or where the soils grouped together were highly variable. The small range in soil sulfates may also have affected the correlations. Correlations of total sulfate and sulfate before incubation with responses of alfalfa in a greenhouse study involving 100 pounds of sulfur per acre were fairly good. These soils had a wide range of response, many of which were highly significant. The ranges in soil tests for these soils also were wider.

Correlation of sulfate released by incubation versus sulfate released before incubations revealed that they were inversely related; that is, sulfate released by incubation decreased as sulfate released before incubation increased.

For the methods employed and the samples utilized, the studies indicated that analysis of soil samples before cropping for total sulfate offers the best promise for making recommendations for sulfur fertilization. Determination of available sulfate also offers some promise for use as a soil-testing procedure. However, for more critical correlation studies, samples of similar soils known to give large variation of responses should be employed. Furthermore, future field and greenhouse studies should control the plant species and soil series, which was not possible for these studies because only yield data and soil samples that were available were utilized. Only the greenhouse experiment on alfalfa treated with 100 lbs. of sulfur was near optimum in its usefulness for correlation studies.

In order to obtain a better understanding of the relations of soil sulfates to crop responses the present method of incubation and soil analysis should be further investigated. Improvements to release higher amounts of mineralized sulfates may result in better correlations of sulfate released by incubation, as well as total sulfates, with yield response to sulfur applications.

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