STUDIES OF SULFUR IN RELATION TO THE SOIL SOLUTION

BY

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INTRODUCTION

Sulfur has given excellent results as a fertilizer for the past twelve years when applied to many of the nearly neutral, semi-arid, basaltic soils of the northwestern United States, especially when used on legumes. More than 120,000 acres in Oregon can be expected, with sulfur, to yield an additional ton an acre of alfalfa a year. One-third of this area has been sulfured.

The reason for the marked increase in yield is not well understood, even though an extensive literature on the agrotechnic use of sulfur has accumulated. Possible deficiency of sulfur in soils has not been seriously considered until recently when better methods of analyses revealed much larger quantities of sulfur in plant tissue than was reported by earlier investigators.[23] Assuming that the quantity of sulfur in plant tissue is an indication of the amount required for normal growth processes, it is readily seen why sulfur is given more consideration than formerly in studies of crop production. The supply of sulfur found in soils by modern methods of analysis[51, 54] emphasizes the relative importance of this element. Elemental sulfur has been extensively studied during the past dozen years in relation to its effect on soils, soil micro-organisms, and plants. The adequacy of the supply of sulphate in the soil solution has been questioned and the study of gains and losses in soil sulfur has been given much attention.

The supply of sulfur in soils is in many instances less than that of phosphorous.[50] The total sulfur content of normal soils can be expected to range from 300 to 1200 pounds in two million.[46, 51] Many of the surface soils of the northwest contain less than 500 pounds of sulfur in 2,000,000, and often run as low as 100 to 300 pounds in the plowed surface of an acre in leached basaltic land. A six-ton alfalfa crop may remove 30 pounds of sulfur an acre. Certain soils that have been cropped for a generation appear to have lost 20 to 40 per cent of the initial sulfur content. The sulfur content of soils seems to vary with the organic matter supply and is usually largest in the surface soil.

Analyses of percolate from lysimeters, where the amount drained out annually is not large, may indicate the nature of the soil solution.[53] The percolate from the Cornell lysimeters contains sulfur lost at the rate of 30 to 44 pounds an acre each year;[46] in Iowa,[16] 67 pounds;

at Rothamsted,[46] 71.6 pounds; at Bromberg, Germany,[19] about 100 pounds; and in Wisconsin[23] and at Oregon Experiment Station the loss in drainage has been 15 to 40 pounds an acre a year, or about four times the amount received in precipitation.[50]

The concentration of sulfate ion in water extracts of soil has often been 100 parts per million or more and often approximates half the concentration of sulfate in the displaced soil solution. Burd[9] reports concentrations of sulfate in displaced solutions from California soils of from 148 to 655 parts per million. Sulfate was found to increase in fallow and to help hold cations in solution, especially when nitrates were depleted by crops or reduced by anaerobic bacteria.

The soil solution is not diluted with sulfur-free rain water. Ames and Boltz[3] present data showing 9 pounds of sulfur per acre in the country and 72 pounds in town received annually from rainfall. Different investigators[50] report determinations showing wide variations between amounts of sulfur thus gained or lost in different sections of the country.

Sulfur is often added to land in barnyard manure, potassium sulfate, ammonium sulfate, commercial "superphosphate," or as calcium sulfate. Especially in southeastern United States, where much commercial fertilizer is used, the practice tends to overcome any possible crop depression from lack of a sufficient concentration of this nutrient in the soil solution. Sulfur seems to be more abundant in soils originating from granitic rock than in soils of basaltic origin. Sulfates may accumulate with alkali, as in the Great Basin region, owing to absence of drainage for its removal.

Hart and Peterson in 1911[23] announced new figures for the sulfur content of crops, showing that Leguminosae and Cruciferae are especially heavy users of sulfur. Reimer and Tartar[51] found that a six-ton crop of alfalfa removed about 30 pounds of sulfur an acre. Recent data by Jones[32] tends to reduce this amount slightly.

One well established function of sulfur is that of increasing the protein content of alfalfa.[51, 45] It is said to be present in the protein cystine.[47, 53] Evidence has been found that the SH group in cystine plays a catalytic rôle in synthesis of vegetable fats in plant cells.[44, 58] Increased root and nodule development[22, 51] and a richer green color have commonly resulted from use of sulfur on alfalfa. Stiffer straw and heavier seed has been noted from sulfur or sulfate applications to grain land[15, 34] Harris et al[24] reported a higher concentration of sulfate in the leaf-tissue fluids of Upland than of Egyptian cotton and suggest that this difference may be related to drought resistance, alkali resistance, and the concentration of sulfate ions in the soil solution.

Adequate evidence does not seem to have been found to establish any close relation between the total sulfur in soils and the sulfate content of the soil solution or sulfate requirement of crops. It seems probable that plants may contain more sulfur than required perhaps both in organic and inorganic form where the sulfate concentration of the soil solution with which they are grown is high.

Numerous investigators have found[50] that with heavy applications of sulfur to soils there is an increase in concentration of free hydrogen ions somewhat proportional to the amount of sulfur applied and oxidized to sulfates. Adams[1] demonstrated that sulfate formed may be leached out, while the acidity is not removed. Hydrogen ion seems to participate in an exchange for absorbed cations, such as calcium ion, permitting the hydrogen ion to remain with the soil as an acid silicate, while the calcium ion is leached out in association with sulfate. The amount of increase in concentration of hydrogen ions in the soil solution as a result of sulfur applications may depend on the amount of readily soluble bases present and general buffer effects of the soil.

Lipman,[36] Kelley,[33] and others have suggested sulfur for correcting the reaction of "black alkali" soil by increasing the hydrogen ion concentration upon oxidation and combination with water. This acid may then dissolve calcium compounds and bring about exchange of such multivalent bases for sodium in the solid phase and thus improve permeability and reaction of "black alkali" land. Johnson and Powers[30] found sulfur an effective chemical treatment for such land under eastern Oregon conditions, especially when used in combination with gypsum or manure. Sulfur may improve the reaction of an alkaline soil, flocculate colloids so as to permit better drainage, and tend to dissolve calcium from its compounds, all of which may improve soil conditions for legume crops.

The Lipman[37, 38] process of rendering rock phosphate soluble depends upon the production of acid by oxidation of sulfur to produce soluble phosphate. Lipman and his associates have studied the most economical proportion of soil, sulfur, and "floats," best suited to moisture and climatic conditions, for economical production of available phosphates. Good results have been secured in western Oregon by applying rock phosphate in combination with sulfur and manure alternated with lime.[50] A reciprocal relation between phosphates and calcium in solution in the soil has been shown by Burd[8] and by Stephenson and Powers.[56] A moderate increase in acidity may tend to increase phosphate in the soil solution while higher acidity may increase calcium ions and aluminum ions and precipitate phosphate to a relatively insoluble form.

There has been some controversy as to whether calcium sulfate or sulfur liberates potassium in the soil. According to Lipman and Gericke[35] this depends upon the particular soil. Different investigators have reported the potassium content of soil water extracts to be somewhat increased as a result of sulfur or sulfate applications.[16, 41, 46]

MacIntire[39] has pointed out a relation of sulfur applications to increased loss of calcium in percolate from lysimeters. This relation has been found to hold true with two Oregon soils employed in lysimeters at Oregon Experiment Station. Adams[1] held that it was difficult to find much calcium in solution in acid soils with a hydrogen ion concentration as great as pH 5.0. Stephenson and Powers[56] found that the most striking effect of sulfur on water extracts of three soils tested was the increase in calcium ion in solution. This effect would be expected to be less marked upon acid soils which have been rather thoroughly leached of soluble calcium compounds.

Nitrification and sulfofication largely result from biological activities. A little sulfur may stimulate ammonification.[49] Sulfur may oxidize and unite with ammonia as sulfate of ammonia.[2] McCool[43] finds that sulfur aids decomposition of organic matter and formation of nitrates. A little sulfur appears to aid nodule development[49, 51] and nitrification[45, 46] in arid soils, while larger applications may result in increasing the hydrogen ion concentration sufficiently to depress nitrogen fixation and nitrification. Rudolfs[54] observed five times more bacteria in alkaline soil that was neutralized by sulfur. Burd and Martin[11] have noted a reciprocal relation between the amount of nitrates and sulfates obtained in the soil solution. Whenever sulfur stimulates growth of legumes an increase may be expected in nitrogen supply in the soil and of nitrate in the soil solution.

Recent investigations lead to the conclusion that the supply of available sulfur, like the supply of available nitrogen, follows a fairly definite cycle. Joffe[29] concludes that making acid phosphate by the Lipman process is chiefly a problem of providing favorable conditions for sulfur oxidation. Brown and Kellogg[7] find that soils have a fairly definite sulfur oxidizing power.[6] Lipman and McLean[38] report that temperature, aeration, moisture content, and proportion of materials affeet sulfur oxidation, and they find no advantage in starting sulfur oxidation with a soil of high aeidity. Halversen and Bollen[21] report that sulfur application inereases the sulfur oxidizing power of soils; they find little need for inoculated sulfur for many Oregon soils. It appears that heavy textured soil is unfavorable and good organie supply is favorable to rapid sulfur oxidation in soils. Brown and Gwinn[5] note that phosphorus and manure increase sulfofication in loam soils. Stephenson[59] has reeently demonstrated that the rate of sulfur oxidation is related to the surfaee area and that sulfur, ground to pass a forty-mesh sieve, should oxidize at a rate adequate to meet plant needs. Boullanger and Dugardin[4] suggest that eertain sulfur eompounds are oxidation eatalysts. The possibility that sulfur oxidation inereases anion eoneentration, thus holding eations in the soil solution and bringing about eonditions favorable to base exehange reaetions, will be developed later.

A review of the laterature emphasizes the need of further investigation as to the rôle of sulfur in the soil solution.

The writer wishes to acknowledge his indebtedncss to Dr. W. F. Gerieke for helpful eounsel during the eourse of these investigations.

SCOPE OF THE EXPERIMENTS

The primary purposc of experiments reported herein has been to determine the effects of sulfur on soil solutions and their relation to sustained erop produetion. The study has ineluded the effeets of sulfur on soil reaction, liberation of bases, and eoneentration of sulfate and other anions, especially as related to the nutritive requirements of alfalfa at different growth periods and to sustained produetiveness of soils.

The main study has been chemieal, supported by some physiologieal experiments and eonfirming field trials, and has ineluded four lines of attaek, as follows: (1) effect of sulfur and sulfates on the soil solution; (2) effect of sulfur on the solutions of different soils; (3) determination of the minimum optimum eoncentration of sulfate for alfalfa by the water culture method; (4) eonfirmation of field-plat trials.

The soils employed and some of their eharaeteristics are given in table 1.

RELATION OF REPLACEABLE BASES AND SULFOFICATION TO THE SOIL SOLUTION

The soil characteristics presented in table 1 indicate the amount of soil solution these soils can retain, their total sulfur content, sulfur oxidizing power, the sulfate content of their displaced soil solutions, the nature and amount of replaceable bases contained, and the response of these soils to sulfur treatment.

TABLE 1

SOME CHARACTERISTICS OF THE SOILS USED

Soil series and type	Usable water capacity (approximate), acre-ins. per acre-ft.	Total sulfur, lbs. to 2,000,000	Sulfur oxidized in 14 days, per cent	Sulfates displaced soil solution, p.p.m.	Replaceable bases, per cent of soil Ca, Mg, Na, K	Replaceable calcium per cent of soil	Response to sulfur in field
1. Madera sand	¾″	821	....	140	.0613	.0400	Good
2. Umatilla med. sand	⅔″	240	36	32	.1734	.1248	Slight
3. Catherine loam	2½″	......	17	464	.7941	.6512	Fair
4. Yakima sandy loam	1½″	...	15	64	.2322	.1419	Good
5. Deschutes sandy loam	1½″	403	18	168	.1967	.1383	Very good
6. Willamette silty clay loam	2″	680	10	14*	4153	.3654	Slight
7. Carlton silt loam	1¾″	280	4	12*	.2876	.2600	Fair
8. Antelope clay adobe	2½″	400	3	117†	1 0816	.8624	Very marked

* Growing crop May 7. † 2:1 extract.

The sulfofying power of a soil seems to have a closer relation to the sulfate content of the soil than does the total sulfur supply. Halversen and Bollen[21] have shown the relation of sulfur oxidizing power of soils to sulfate content. As sulfofication is largely a biological process, providing conditions most favorable for the organisms should aid in maintaining a favorable sulfate concentration in the soil solution. Application of manure with sulfur has appeared to be an effective aid to sulfofication in treated alkali land.[30] Burd has recently reported data[8] emphasizing the importance of biological activities in keeping up a favorable concentration of nutrient anions and the importance of supplying sufficient total anion concentration to hold favorable amounts of cations in the soil solution. Johnson[31] seems to show that growing alfalfa may increase the sulfate-supplying power of a soil. This may be due to plant removal of sulfate formed. The forms of sulfur in a soil may affect rate of sulfofication.

The total supply of replaceable bases and the proportion of univalent to multivalent bases adsorbed may indicate the power of recovery of a soil solution after exhausting crops and the properties likely to be imparted due to base exchange reactions. Sulfur may oxidize and then unite with water to cause an increase in hydrogen ion concentration in the soil solution. The presence of readily soluble compounds, such as calcium carbonate, under such a condition will favor solution, the rate of which will depend on the concentration of acid present. Twentieth normal hydrochloric acid in large quantity has been found to be capable of replacing about all the replaceable base held by the soil adsorbing complex where drainage is provided.[17] When the concentration of hydrogen ion or other cation is increased as a result of sulfur oxidation, base exchange may occur. This is sufficient to indicate the close and important relation of sulfur oxidation, solubility effects, and base exchange reactions in the soil system to changes in its liquid phase. Where the supply of replaceable calcium in table 1 is low and response from sulfur applications marked, it would seem to indicate that sulfur oxidation results in solubility effects.

Soils 2 and 6 (table 1) give little response to sulfur applications, and these soils oxidize sulfur rapidly. Soil 2 is irrigated with water containing two or three pounds of sulfate sulfur per acre-foot. Soil 8 oxidizes sulfur slowly and gives marked response to sulfur applications.

In order to learn the chemical effects of sulfur and sulfates on the soil solution twenty-eight two-gallon stoneware jars that had been coated with valspar were filled with screened surface soil of Madera sand type which was known to give typical response to sulfur applications. These were divided into groups of four and treated with different salts (table 2) at a rate sufficient to supply one hundred pounds of sulfur an acre. Three jars of each group were planted to Grimm alfalfa while the fourth was fallowed. Before the seedlings were one inch high, their number was reduced to ten uniform sized plants in each jar. The soil was maintained at about optimum moisture content by frequent additions of distilled water and the fallows periodically sampled and screened and their solutions displaced and analyzed, using methods described by Burd.[11] Displacement of the soil solution is accomplished by packing the soil in brass tubes, adding distilled water above as a displacing medium, and then air pressure from the top.

Methods of analysis employed were, for the most part, those in use in the plant nutrition laboratories of the University of California and recently made available by Hibbard.[25] The water culture technique employed has been described by Hoagland,[28] Gericke,[18] and Davis.[14] Hydrogen ion concentration determinations of solution cultures were colorimetric and of displaced soil solutions were electrometric. Successive portions of displaced solutions were found to show fairly uniform electrical conductivity or specific resistance until dilution by the displacing medium began, at which point displacement was terminated, the dilute solution discarded, and the uniform solution saved and analyzed.

CHEMICAL EXPERIMENTS

SULFUR AND SULFATES AND THE SOIL SOLUTION

TABLE 2

The treatments given to portions of soil are indicated in column 1, table 2. Sulfur was added at the rate of one hundred pounds per acre, while calcium oxide and sulfates were added in quantities contained in gypsum equivalent to one hundred pounds sulfur an acre.

The composition of the soil solutions displaced from treated and untreated fallow jars of Madera sand as determined in parts per million, after 6 weeks' and again after 12 weeks' incubation, is presented in table 2. Analyses are also given for the solution displaced from the original soil and for the two lots receiving heavy sulfur applications after 15 months.

The reaction of this soil on the untreated field plats was slightly alkaline and gave a pH value of 7.3. Soil from sulfured field plats was found to be exactly neutral. After 6 weeks' incubation in the green house a very slight acidity had been developed in untreated, fallow jars, as shown later (table 6), perhaps owing to formation of carbonic acid from decomposition of organic matter under the moist, warm conditions in the greenhouse. After 6 weeks' time the development of a slightly higher concentration of hydrogen ions was observed in soils that had been treated with sulfur or with certain sulfates. The concentration of sulfate ion in the solutions displaced in such cases was found to have increased. After 12 weeks' incubation the hydrogen ion concentration had increased with certain sulfate treatments, and the heavy sulfur application resulted in hydrogen ion concentrations that were unfavorably high for growth. This high acidity still prevailed after 15 months. In the cropped series definite acidity developed and growth appeared to maintain a hydrogen ion concentration of about pH 6.0. Reaction might be modified by decomposition of organic matter, excretions by roots, formation of sulfuric acid in sulfur treated pots, as by hydrolysis, and selective absorption of cations, added in certain salts such as sulfate of ammonia.

TABLE 2

EFFECT OF SULFUR AND SULFATE ON THE SOIL SOLUTION

Analysis reduced to 10 per cent moisture (wet weight).

Initial soils	Dec.	Parts per million soil solution					
Treatment, pounds per acre	pH	NO_3	SO_4	PO_4	Ca	K	Mg
Untreated plot	7.3	47	140	4.5	119	57	129
Sulfured plot	7.0	25	194	3.5	146	83	78
Analysis of fallows (after six weeks incubation)							
X untreated	6.2	116	253	4.0	144	98	119
Sulfur, 100 lb.	6.1	161	369	3.5	203	125	221
CaO	6.5	127	195	6.0	142	88	95
S and CaO	6.3	156	256	5.0	196	105	156
K_2SO_4	6.1	157	294	3.0	116	172	113
$CaSO_4$	6.3	120	247	3.5	135	112	90
X	6.7	70	161	4.0	172	60	91
$(NH_4)_2SO_4$	5.4	150	205	4.0	227	63	121
$MgSO_4$	5.9	53	235	4.5	294	63	196
Displaced soil	6.5	30	153	4.0	160	27	98
S-200	5.9	38	284	5.0	327	125	119
S-500	5.9	21	509	3.5	349	133	102
Second analysis (after twelve weeks)							
X	6.1	124	261	3.0	207	107	160
S	6.3	100	341	5.0	382	72	131
CaO	6.7	102	204	5.0	324	127	118
CaO and S	6.7	77	322	5.0	356	120	144
K_2SO_4	6.5	84	282	4.0	361	165	165
$CaSO_4$	6.6	82	224	4.0	351	84	79
X	5.8	87	190	4.0	139	47	139
$(NH_4)_2SO_4$	5.6	176	258	5.0	326	57	128
$MgSO_4$	5.6	79	241	4.0	268	64	183
Displaced soil	6.4	88	219	3.5	124	32	148
S-200	4.8	47	376	24.0	376	135	157
S-500	4.4	37	..	52.0	....	156	171
Third analysis (after fifteen months)							
X	7.7	44	94	2.0	79	70	....
S-200	3.8	24	672	1.0	152	174	....
S-500	3.6	1	1510	15.0	171	319	....

The nitrate in field plats, treated with 200 pounds of sulfur per acre about a year previous to sampling, was lower than in the adjacent untreated plat. After incubation for 6 weeks there was a tendency toward accumulation of nitrates except where unfavorable acidity had developed. After 12 weeks, high acidity developed in heavily sulfured jars, which made conditions unfavorable for nitrification and depressed the supply of nitrate to or below that of the untreated soil. A large portion of the nitrogen in the ammonium sulfate applied appeared later as nitrate.

An initial supply of 140 parts per million of sulfate was found in the soil solution. Since this soil will retain about 10 per cent useable moisture between the wilting point and the excess point, this represents 14 parts per million sulfate for the whole system. Sulfur in the field trial increased the sulfate content to 194 parts per million. Madera sand appeared to have good sulfofying power, even without sulfur additions. Addition of sulfur and sulfate substantially increased the sulfate content of the soil solution in six weeks. Adding calcium oxide with sulfur appeared to retard, rather than to encourage, sulfofication. Sulfofication appeared to be somewhat proportional to the amount of sulfur applied, for in subsequent determinations gains in sulfate concentration were found. Small differences were found from the application of various sulfates.

The amount of soluble phosphate first seems to increase, then to decrease, after sulfur treatment. There appears to be a little depression in the amount of phosphate in the solution after sulfur applied has brought considerable calcium into solution. With heavy sulfur applications, marked increase in acidity and increase in the amount of phosphate were found in the soil solution after 12 weeks' incubation. Increase in solubility of bases such as calcium, iron, or aluminum appears to have resulted in precipitation of phosphate before 15 months passed.

Calcium was found to come into solution strikingly as a result of sulfur or sulfate applications. Nearly three times as much calcium was found in the soil solution following heavy applications of sulfur. Sulfur appears to have modified the soil solution with respect to calcium more than with any other ion.

Potassium was brought into solution in the soil to the extent that the supply in solution was almost doubled by heavy applications of sulfur. Part of the potassium, when applied as potassium sulfate, appeared to be fixed by exchange reaction with other bases which were brought into solution. Heavy application of sulfur resulted in a large increase in potassium ion in the soil solution after 15 months. Either potassium-bearing compounds were slowly dissolved or the calcium had participated in a base exchange with the potassium in the solid phase.

Magnesium ion has a tendency to increase in concentration in the soil solution owing to sulfur additions and possibly also to base exchange reactions following an increase in total concentration of the soil solution.

The analyses in general indicate that a very important function of sulfate is that of bringing in and holding bases in solution. It also appears to increase the soil acidity with a resulting increase in availability of phosphate. The phosphate, however, may tend to disappear if the soil is well supplied with bases, such as calcium, which may react on the calcium and cause precipitation of phosphate from the soil solution. This reciprocal relation of soluble calcium relative to phosphate has been noted by Burd.[9]

SULFUR AND SOIL SOLUTIONS OF DIFFERENT SOILS

Table 3

Samples of typical Oregon soils were collected from old sulfur experiment fields, including soil from both sulfured and unsulfured plats, that had been in experiments for as long as ten years and had received sulfur generally at the rate of one hundred pounds per acre every three or four years. These samples were screened and brought to optimum moisture content, allowed to stand so that equilibrium would be established between the solid and liquid phases of the system, and the displaced soil solutions recovered and analyzed.

Results are given in table 3 and show that sulfur applications increase acidity in all cases, and usually more than 0.5 pH. There was a noticeable difference in the buffer value of various soils used. The Catherine loam did not resist change in reaction well and became unfavorably acid. The nitrate content of these solutions was not greatly modified, but gave evidence of being depressed in certain cases from sulfur applications on acid soils, as shown in the case of Catherine loam.

The sulfate content of Willamette and Carlton soils was found to be very low. Samples were taken from these soils on May 7 following a cool spring, which would be unfavorable to sulfur oxidation, and from plats supporting winter grain that had attained 5 to 9 inches height. Both these humid soils are subject to leaching in winter. Sulfofication is rapid under laboratory conditions in the Willamette soil and little crop increase results from sulfur applications thereto, while Carlton soil is lower in total sulfur and sulfate and responds to sulfur applications. Sulfur substantially increased the sulfate content of all soil solutions. There was a tendency for sulfur to increase the calcium content of the soil solution, and also the potassium content.

TABLE 3

EFFECT OF SULFUR ON SOIL SOLUTIONS

Soils displaced May, 1925, reduced to comparative moisture basis.

Soil	Treatment pounds per acre	H_2O	pH	N	SO_4	PO_4	Ca	K	Mg
Madera sand field sample	200 S. Ac.	10% (wet basis)	....	24	672	....	152	174	....
1. Madera sand	200 S. Ac.	10	7.00	25	194	3.5	146	83	78
2. Madera sand	Untreated	10	7.30	47	140	4.5	119	57	129
3. Deschutes sandy loam	3 x 100 S.	15	7.47	51	316	1.0	123	96	....
4. Deschutes sandy loam	Untreated	15	7.84	63	153	10.0	100	29	....
5. Umatilla medium sand	3 x 100 S.	10	7.00	54	58	3.0	86	31	....
6. Umatilla medium sand	Untreated	10	7.76	36	37	3.0	86	25	....
7. Yakima sandy loam	100 S.	15	6.83	20	103	2.0	211	86	....
8. Yakima sandy loam	Untreated	15	7.67	40	75	4.5	164	75	....
9. Carlton silt loam	2 x 100 S.	20	6.15	18	207	2.0	95	75	....
10. Carlton silt loam	Untreated	20	6.83	15	13	1.0	41	58	....
Wheat 5″ May 7.									
11. Willamette silty clay loam	3 x 320 S.	20	5.81	trace	46	1.5	42	14	....
12. Willamette silty clay loam	Untreated	20	6.14	none	17	4.0	45	11	....
Barley 9″ May 7.									
13. Catherine loam	100 S.	30	5.47	22	555	3.0	328	318	....
14. Catherine loam	Untreated	30	6.57	18	114	2.0	142	256	....
Water extract, two to one.									
		Fe							
15. Antelope clay adobe	Untreated	.04	....		117	3.8	100	20.3	....
16. Antelope clay adobe	3 x 200 S.	.08	....		100	3.6	105	17.3	....
17. Antelope clay adobe	3 x 400 S.	.07			74	4.6	152	14.8	....

Willamette silty clay loam and Carlton silt loam are acid soils, the latter occurring in the low foothills of the Willamette Valley. The Willamette soil from the old valley filling contains a fair total amount of sulfur, and this soil has a high sulfofying power and good supply of organic matter. It gives only slight response to sulfur applications. Carlton and other "redhill" soils are low in total sulfur and in soluble sulfate and give moderate response to sulfur treatment. They are also low in soluble calcium, and in some instances the soluble potassium is low. Sulfur may help to bring bases into solution in these acid soils, but calcium sulfate may be more safely used.

Umatilla medium sand receives from two to three pounds of sulfur in each acre foot of irrigation water and requires at least five acre-feet of water a season. This soil is rather low in replaceable bases and does not afford much opportunity for base exchange. It gives slight response to sulfur applications. On the finer soils in that region a moderate increase in alfalfa yield is secured from sulfur applications.

Catherine loam has given more profitable returns from calcium sulfate than from sulfur. The reaction of this soil is already slightly acid and sulfur may develop an unfavorably acid condition. This was formerly wild meadow land.

Yakima sandy loam and Deschutes sandy loam are arid soils of nearly neutral reaction with large total supplies of calcium and having only moderate amounts of potassium ion in their soil solutions. The former is typical of the main soil area of Klamath Project. Potassium salts pay when applied to Deschutes sandy loam in which potatoes are growing, and sulfur appears to bring treble the amount of potassium into the soil solution in this soil. The alfalfa crop in this section when soil-treated with sulfur develops an especially rich green color.

The samples of Antelope clay adobe used in these experiments come from the sulfur fertilized plats established by F. C. Reimer north of Medford and are too heavy for displacement, so water extracts were made. The amount of iron in solution was doubled by sulfur applications and there was some increase in calcium ion in solution. Iron pyrite on this land has given as good increase in alfalfa yield as sulfur, after time was allowed for oxidation of the pyrite. Ferrous sulfate has given the best yields in plat trials at this experiment field.[50]

After the analyses above given were completed, ferric chloride was sprayed on two plats of alfalfa previously unfertilized and a vigorous growth resulted, showing all the visible results commonly secured from sulfur on this field. There is a possibility of iron participating in a base exchange but that does not seem to have been an important factor here. Iron sulfate has been successfully used to overcome chlorosis in fruit trees where applied at the tree roots in this heavy soil.

The chief effect of sulfur may depend considerably on the characteristics of the particular soil at hand and its reaction, physical condition, chemical composition, or micro-organisms present. On arid soils of slightly alkaline reaction sulfur may improve the reaction of the soil solution for alfalfa. This may increase the solubility of iron in the soil solution and favor the development of chlorophyll. It may also favor the absorption of anions, such as nitrates, by plants. A better supply of calcium, as well as sulfate, is often provided by an application of sulfur.

SULFUR AND ALFALFA YIELD WITH SOILS IN JARS

Four portions of each of these soils were arranged in one-gallon jars, two being treated with sulfur at the rate of one hundred pounds an acre, the others untreated. One treated and one untreated jar containing soil of each type were then planted to alfalfa for culture tests and for displacement and analysis, if needed, to check against field plat samples.

The increase in yield of alfalfa secured in these jars as a result of sulfur treatment ranged from 7 per cent to 54 per cent. Umatilla sand appeared to give greater response to this treatment, in the jars and irrigated with distilled water, than under field conditions where large amounts of irrigation water contributed a substantial part of sulfur needed by the alfalfa. Ferric chloride was found to be about as effective as sulfur on Antelope clay adobe, for increasing alfalfa yield in jars, which is further evidence that an important effect of sulfur on this soil is to improve availability of iron and overcome chlorosis in alfalfa grown in it.

A preliminary experiment was conducted with young alfalfa transplanted from Madera sand soil from near Delhi, California. These plants had grown through the summer season on soil receiving no sulfur. Three two-gallon jars were untreated, a second lot of three received sulfur, a third lot, reprecipitated calcium carbonate and sulfur, a fourth received, sulfur as calcium sulfate, and the fifth lot received potassium sulfate at a rate that would supply one hundred pounds of sulfur to the acre. The plants were set out December 29, 1923. Cuttings were made when the growth bloomed freely, February 29, May 5, and May 27. One jar of each lot was not harvested at the second cutting but was left for seed. The effect of sulfur was to favor seed formation. Sulfur or sulfates increased the height, vigor, and yield of alfalfa in this trial 20 to 40 per cent.

Sulfur reduced the water requirement about one-third, as expressed in units of water per unit of dry matter.

Thirty-six two-gallon stoneware pots, paralleling the fallow jars used for analyses, were planted to Grimm alfalfa, thinned to ten plants per jar and four months' growth harvested in one cutting May 27, 1924. All the sulfates were applied in amounts needed to provide one hundred pounds per acre of sulfur and gave similar increases in yield. The jars receiving potassium sulfate attained the maximum height. Moderate increases over the untreated pots were secured with calcium and with sulfur used singly or combined. The increased efficiency of water consumed by this growth in treated jars was reflected in a lower water requirement per unit dry matter. A more favorable concentration of soil nutrients or better balance in the solution should lead to a lower transpiration and therefore a lower water requirement, unless other conditions cause small yield of dry matter.

HYDROGEN ION CONCENTRATION IN CROPPED AND FALLOW SOIL POTS

The hydrogen ion determinations were made about every two weeks for all soil in jars. Colorimetric tests were made promptly, after sampling, with fresh color standards checked with the hydrogen electrode.

Decomposition of organic matter on untreated fallow pots seems to have brought the reaction down from pH 6.9 to about 6.2. Calcium carbonate tended to maintain a more nearly neutral reaction, while heavy application of sulfate developed an unfavorably acid condition.

Cropped pots when untreated developed a slight acidity and maintained a pH of about 6.0. There was a somewhat more uniform reaction in cropped pots due perhaps to the tendency of plants to maintain a favorable reaction. Results strongly indicate that a slightly acid reaction is brought about by CO_2 evolved by growing roots in cropped pots or from decomposition of organic matter in fallows forming HCO_3 as suggested by Hoagland.[28] They also indicate that a slightly acid reaction is most favorable for alfalfa growth in soil and that moderate applications of sulfur may improve the reaction of basic or slightly alkaline arid soils for alfalfa nutrition.

PHYSIOLOGICAL EXPERIMENTS

In order to test the adequacy, for alfalfa growth, of sulfate concentrations found in soil solutions, some supplementary water culture experiments were undertaken. It was hoped that this would shed further light on the sulfate need of alfalfa, the form in which sulfate is best obtained from the soil solution, the part of the growth period when sulfate is most needed, the crop-producing power of limited amounts of sulfate, and especially the concentration of sulfate needed for best growth. For this work fruit jars were used and the seedlings supported on flat perforated corks. Molal solutions were prepared of di-potassium acid phosphate, mono-potassium acid phosphate, calcium nitrate, and magnesium sulfate. From these stock solutions a culture solution was made up to an osmotic concentration equal to approximately 1 atmosphere pressure and a pH value of about 6.0. Sulfur-free culture solutions were provided by substituting calcium nitrate for magnesium sulfate, and limited amounts of sulfate were added in certain experiments from a saturated solution of calcium sulfate to provide the number of parts per million of sulfate desired. A fresh solution of iron-tartrate was used for supplying soluble iron. In these experiments records were kept of the height and vigor of plants, the amount of transpiration, and the reaction to culture solutions. The reaction tests were made almost daily where there was a tendency to deviate from the optimum range.

Preliminary Studies

Tables 4 to 8

Two-year-old alfalfa plants were secured from sulfured and unsulfured field plats near Delhi, where crops grown on Madera sand had shown typical response to sulfur. The crowns of plants grown on sulfured fields were much larger than those of the same age from the unsulfured plats. The color of foliage on sulfured land was a dark green, the plants presenting a marked contrast to the rather chlorotic, unthrifty plants from the unsulfured plats. Sulfur in this field trial had doubled the yield of alfalfa. The plants were washed free of soil, dried on a blotter, and their individual weights determined. There had been some root pruning so the tops were clipped back close to the crowns. The plants recovered promptly from transplanting when placed in the nutrient solution.

TABLE 4

ALFALFA TRANSPLANTED FROM SULFUR-TREATED LAND

Mean yield in grams. Dry matter (3 cuttings) from single two-year-old plants.

Treatment	Mean yield tops, grams	Water requirement tops
A—Four-quart jars. Plants from sulfured field.		
Complete solution	8.4 $\pm$ .10	533
Solution lacking S	4.5 $\pm$ .16	801
B—Three-quart jars. Plants from unsulfured field.		
Complete solution	5.1 $\pm$ .24	589
No S. Two nitrates	1.9 $\pm$ .08	1377
C—Two-quart jars.		
Complete solution	4.5 $\pm$ .24	612
No S. Two phosphates	2.1 $\pm$ .07	1285
D. I—Reaction adjusted April 1, with N/10 H_2SO_4.		
Complete solution	2.4 $\pm$ .12	780
No S. Two nitrates	1.6 $\pm$ .05	1067
No S. Two phosphates	1.5 $\pm$ .02	1089
D. II—Reaction adjusted April 1, with N/10 HCl. (Whole plant.)		
Complete solution	1.9 $\pm$ .06	598
No S. Two nitrates	1.4 $\pm$ .06	754
No S. Two phosphates	1.5 $\pm$ .08	751

A trial was made with twelve plants from the unsulfured field plat, the plants being divided evenly into two lots according to weight. Six plants were placed on four-quart jars containing complete culture solutions and six others on solutions lacking sulfur. In two weeks a difference in the appearance of the plants was noticeable. The plants provided with sulfate developed a better green color and made over twice the growth during the first two months compared with those grown in the sulfur-free solution. The plants came into bloom and were cut January 29, April 23, and May 27, 1924. The yields are presented in table 4, section *A*. Approximately twice the yield of dry matter was secured from plants grown on solutions provided with sulfate. These plants made nearly twice as efficient use of water consumed for each unit of dry matter produced as did the sulfur-free series.

A second lot of plants were set on three-quart jars and treated as in the above experiment, except that the sulfur-free solution included two nitrate salts instead of two phosphate salts as in the above

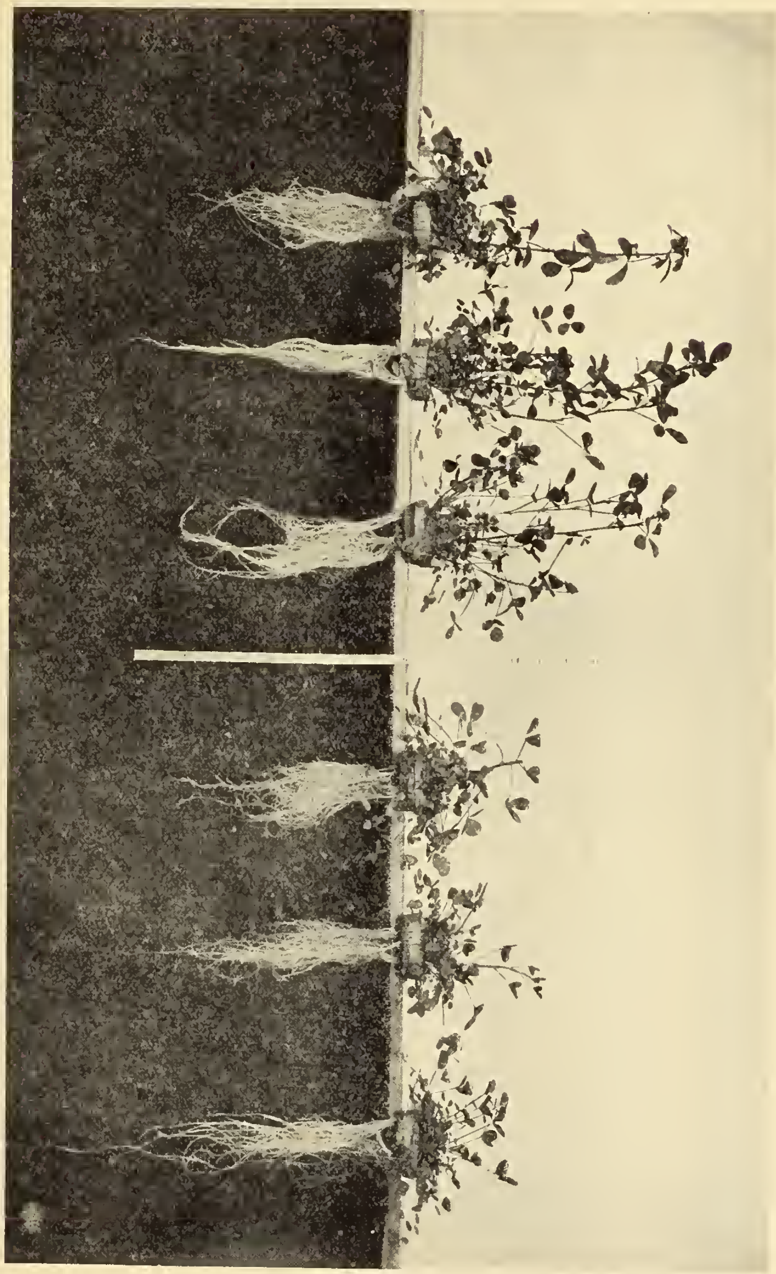


Fig. 1. Alfalfa seedlings grown in partial versus "complete" nutrient solutions. Seedlings at the right were grown in cultural solutions lacking sulfur.

trial. The presence of two nitrate salts, or a larger supply of nitrates, was associated with greater yields relative to untreated plants than was secured in the above experiment (table 4, *B*). These transplants were one year old and sulfur about trebled the yield, resulting in a transpiration requirement of about three-sevenths of that from the plants grown in sulfate-free solutions.

A third lot of transplants from the unsulfured alfalfa plats were divided into lots of six and placed on two-quart jars, one-half being provided with a complete culture solution and the others with a solution lacking sulfate. The yields of these plats are presented in section *C* of table 4. Where sulfate was provided the plants outgrew their chlorotic appearance in two to three weeks' time, indicating that lack of sulfate was the cause of their devitalized condition. The total yield for plants provided with sulfate was more than twice that from the sulfur-free series for three cuttings, and consumed less than half the amount of water per unit of dry matter produced.

A fourth lot of transplants two months old was secured without root injury and set on three dozen, one-quart culture jars. One dozen of these jars were provided with a complete nutrient solution; a second dozen were provided with a water culture solution lacking sulfate and containing two nitrate salts; and the third dozen were provided with nutrients using two phosphate salts. When these plants were six weeks old they developed a chlorotic appearance, and at that time half of the dozen plants in the sulfur-free solution were brought to a favorable reaction by the use of N/10 sulfuric acid. To six other cultures N/10 hydrochloric was applied to produce a favorable reaction. Results of this trial, table 4, section *D*, show a marked increase in yield where sulfate was included. Applying a limited quantity of sulfate, when plants had been grown for six weeks on culture solutions, produced more improvement than can be credited to improved reaction alone. In all these trials the water consumption was greatly increased where sulfur was lacking. In three days' time the sulfate added in acid caused a dark green color of the foliage, which was noticeable until the plants were harvested a month later. This difference is indicated in figure 3.

Fig. 2. Effect of sulfur on size, pattern, and color of alfalfa leaves. Left, alfalfa from sulfured soil pot; right, from unsulfured soil pot.

Fig. 3. Plants grown on solutions where reaction was adjusted with N/10 H_2SO_4 versus N/10 HCl.

SULFUR AND CHLOROPHYLL

Chlorophyll determinations, following methods described by Willstätter, with the whole top growth from sulfured and unsulfured alfalfa in soil pot trials, yielded 12 per cent more chlorophyll where sulfur was applied. Alfalfa leaves collected from sulfured and unsulfured field plats showed an increase of 18 per cent in chlorophyll content. The effect of sulfur in increasing width, size, and color of alfalfa leaves is indicated in figure 2. Lack of sulfur resulted in a lack of rich green color and lack of vigor. Data in table 3 show that sulfur application increased the iron content of the water extract from Antelope clay adobe and this iron is known to play an important rôle in chlorophyll synthesis. Sulfuric acid was also found to restore color better than hydrochloric acid when used to adjust reaction in culture solutions.

WATER CULTURE EXPERIMENTS WITH SEEDLINGS

Table 5, *A*

To check the plan of providing sulfate to seedlings in partial culture solutions two days in six, three series of cultures were provided. In the first, plants were exposed to calcium sulfate one day in five; in the second, two days in six; and in the third, four days in eight. The remainder of the period the plants grew on culture solutions lacking sulfur. During the first two months of the experiment the best growth was obtained with plants exposed to calcium sulfate two days in six. In the latter part of the growth period the plants exposed to sulfate only one day in five forged ahead and gave definitely better total yields both of total and marketable dry matter, indicating that extra sulfur was most helpful in the early part of the growth period and perhaps undesirable later. Figure 6 shows typical plants of each series after three months' growth. There was little difference in the yields of the plants exposed two days, compared to those exposed four days to sulfate solution, as shown in section *A* of table 5. There was a marked difference in appearance of plants with and without sulfur in this and other trials, as shown in figure 7.

HOW DOES SULFUR GO INTO THE PLANT?

TABLE 5, *B*

Sixteen series of six cultures each containing three Grimm alfalfa seedlings per culture were employed in an experiment, which covered a growing period of 110 days. Plants were grown on culture solutions lacking sulfur four days in six and on various partial culture solutions containing different sulfates two days in six. Solutions were changed

TABLE 5

HOW DOES SULFUR GO INTO THE PLANT?

Alfalfa yield in grams. Dry matter. May 26, 1924.

(Grown on sulfur-free culture solutions and transferred to solutions containing sulfate at regular intervals.)

A. Treatment	Mean yield: Whole culture 3 plants	Mean yield: Tops	Water requirement tops
Complete culture solution, unchanged	3.1	2.2 ± .08	855
Complete culture solution, changed monthly	3.9	2.4 ± .15	867
S-free solution, unchanged	3.8	2.2 ± .16	851
S-free solution, changed monthly	4.1	2.6 ± .10	712
S-free solution 4 days; then $CaSO_4$ 1 day	5.7	3.9 ± .23	569
S-free solution 4 days; then $CaSO_4$ 2 days	4.2	2.7 ± .14	790
S-free solution 4 days; then $CaSO_4$ 4 days	4.5	2.9 ± .23	651
B.			
S-free solution 4 days; then $(NH_4)_2SO_4$ 2 days	3.6	2.7 ± .21	725
S-free solution 4 days; then $MgSO_4$ 2 days	1.8	1.2 ± .10	796
S-free solution 4 days; then K_2SO_4 2 days	5.3	3.6 ± .18	635
S-free solution 4 days; then Ca, NO_3, SO_4 and PO_4 2 days	5.5	3.6 ± .13	639
S-free solution 4 days; then Mg, NO_3, SO_4 and PO_4 2 days	.9	2.8 ± .69	1875
S-free solution 4 days; then K, NO_3, SO_4 and PO_4 2 days	4.6	3.3 ± .23	699
S-free solution 4 days; then Ca, NO_3 and SO_4 2 days	4.6	3.3 ± .06	691
S-free solution 4 days; then Mg, NO_3 and SO_4 2 days	1.3	1.0 ± .16	1149
S-free solution 4 days; then K, NO_3 and SO_4 2 days	3.6	3.3 ± .21	638
C. Solutions free of K and SO_4 vs. — (Cation supplied only 2 days in 6 and with SO_4)			
S-free solution 4 days; then $CaSO_4$ 2 days	4.3	2.9 ± .11	421
S-free solution 4 days; then K_2SO_4 2 days	2.9	1.7 ± .10	603
S-free solution 4 days; then $(NH_4)_2SO_4$ 2 days	.3	.2 ± .02	6666
Complete solution	3.5	2.5 ± .10	603
S-free solution 4 days; then $(NH_4)_2SO_4$ (grain) 2 days	6.7	5.5 ± .30	539
Displaced soil solution (6 weeks' growth)	.15	1.1 ± .11	750
Displaced soil solution plus K_2SO_4	.6	.2 ± .03	937

frequently during the latter part of the growth period to avoid contamination by moving the plants from the sulfur-free nutrient solution to the companion solutions containing different forms of sulfate. The plant roots were washed by standing them in two-gallon jars of tap water for twenty or thirty minutes and then rinsing in distilled water before transferring from one partial solution to the other. An extra series of stationary controls having sulfur, and one lacking sulfur,

Fig. 4. Effect of different sulfates on alfalfa.

were provided to test the effect of changing the nutrient solutions monthly. Changing the solution showed a little advantage, as did changing the plants. This was probably due to improved aeration.

Two days in six, series 8 to 12 received sulfate in single salt solution as calcium sulfate, potassium sulfate, ammonium sulfate, or magnesium sulfate, respectively. During the first two months of this experiment plants receiving sulfate in the form of calcium sulfate were definitely the better plants. During the last four or five weeks of the experiment these were overtaken by the potassium sulfate series, the yield of which was slightly greater. The yield with ammonium sulfate was about four-fifths of that with the calcium sulfate, while the magnesium sulfate series yielded only about two-fifths as much as the calcium sulfate series.

Series 12 to 14 were provided in which the sulfate-bearing solution received nitrate, phosphate, and sulfate salts of the base under consideration. Under this condition calcium salts produced one-eighth more total dry matter than the potassium salts. The yield with magnesium sulfate was very low.

In series 15 to 17 the cations were supplied in both nitrate and sulfate forms. There was no significant difference in the yield obtained with potassium from that obtained with calcium sulfate.

In all trials, solutions containing magnesium salt gave very poor results. The reaction was difficult to control in the case of magnesium salt solutions, and even with reaction controlled there appeared to be magnesium toxicity. During the first two months of the growth period, calcium sulfate appeared to be definitely the best form of sulfur for the alfalfa plants. For typical plants, the relative growth for different treatments is shown photographically in figure 4.

During the latter part of the growth period potassium sulfate showed advantage in certain cases.

CALCIUM SULFATE VERSUS POTASSIUM SULFATE

TABLE 5, *C*

An experiment was arranged to compare further the value of calcium sulfate with that of potassium sulfate. In this experiment the main solution was deprived of both sulfate and the cation concerned, so that it could be obtained only during the two days out of six when the roots were in the partial nutrient solution containing sulfate. Thus calcium, potassium, or magnesium was held out of the main solution and applied only as sulfates in the partial nutrient

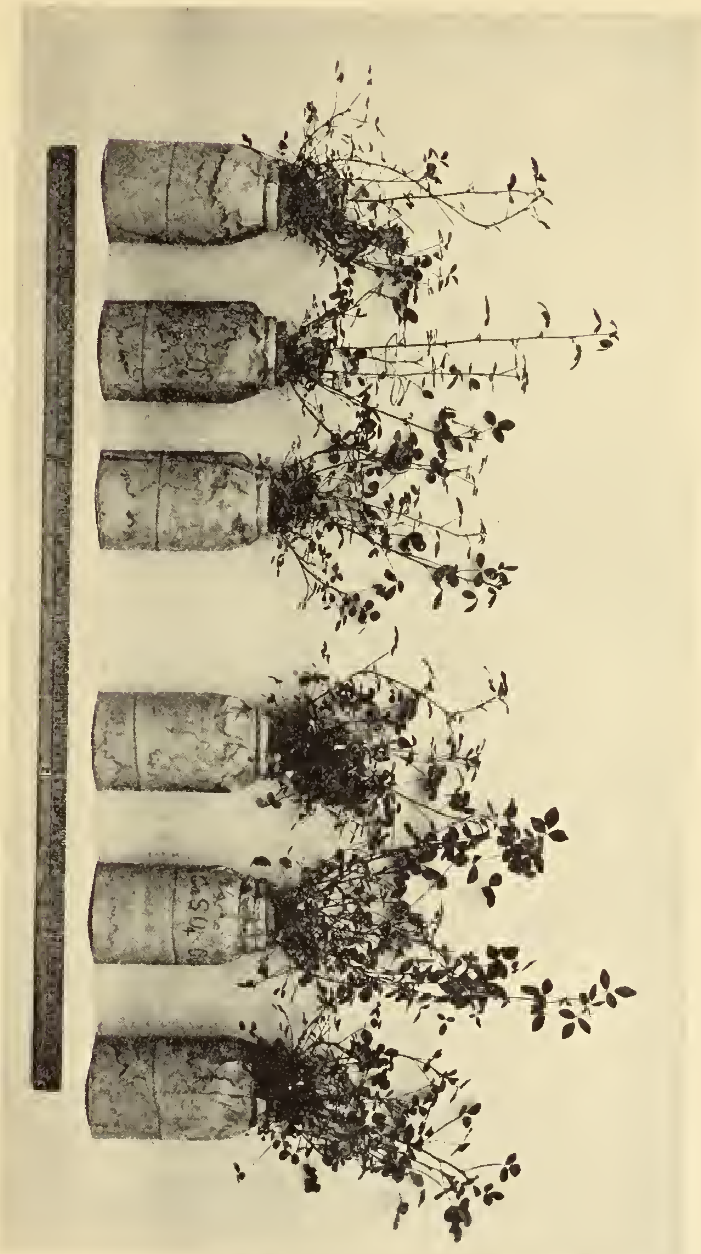


Fig. 5. Effect of providing K and S versus Ca and S only in single salt solutions.

solutions to which plants were transferred two days in six. Also nitrogen, as well as sulfur, was kept out of a certain main solution, being provided only two days in six as ammonium sulfate. In this experiment potassium sulfate gave only about three-fifths the yield of tops and of total dry matter as was secured with calcium sulfate. In this trial nitrogen and potassium were not present at one time, and the calcium sulfate excelled during nearly all of the growth period, as shown in figure 5. During the period when the plants were nine to ten weeks old, the potassium sulfate series and calcium sulfate series were nearly equal in size.

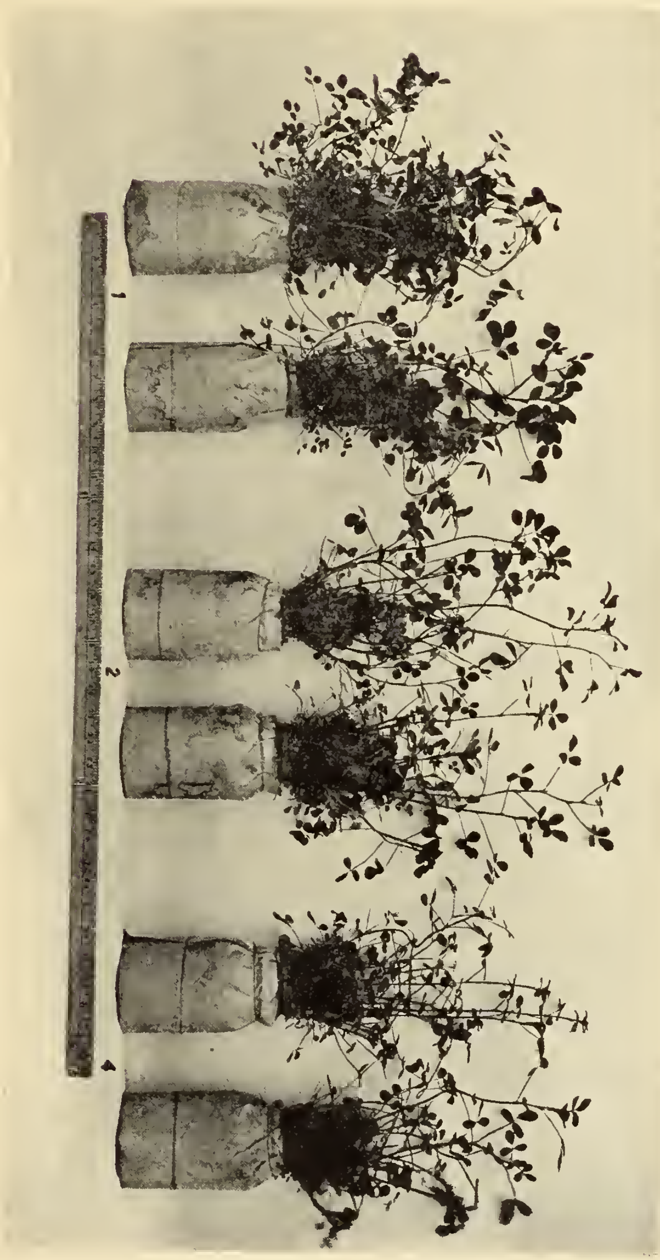
In connection with the value of potassium sulfate relative to calcium sulfate, it should be noted that calcium encouraged much branching of roots and a bushy top growth, or cell division; whereas potassium produced cell elongation and seemed more important at that period of growth when the alfalfa was making its maximum increase in height. The ash of alfalfa contains about 16.25 per cent calcium and 24.7 per cent potassium and the oven dry alfalfa, 1.26 per cent calcium as against 1.87 per cent for potassium.[32] As shown by Gericke[18] potassium is best supplied to plants in association with nitrate, and this was verified in the course of these experiments.

COMPLETE NUTRIENT SOLUTION VERSUS DISPLACED SOIL SOLUTION

Table 5, *C*

In connection with these experiments, three series of 400 cc. bottles were provided, set with two alfalfa seedlings to each culture. One set was filled with a complete nutrient solution, the second with the natural soil solution displaced from the Delhi soil, and the third series with displaced solution reinforced with sulfate applied as potassium sulfate. After the first few weeks the displaced solutions made little further progress. The series reinforced with sulfate yielded about three times as much dry matter as the natural soil solution, but only about one-fifth as much as the control. At Oregon Experiment Station sulfates have increased growth on lysimeter waters even where the untreated drainage water was changed frequently and where the sulfate concentration in the percolate was similar to that obtained from displacing the same soil type.[59]

A similar experiment, to be reported elsewhere, dealt with the best salt for supplying calcium ion for alfalfa in partial solution cultures.



One day in five. Two days in six. Four days in eight.

Fig. 6. Plants exposed to $CaSO_4$ for different intervals. Photographed after ninety days' growth.

The largest yields were secured with calcium supplied as sulfate. A slightly lower yield was secured with calcium nitrate, and much less with calcium phosphate. The mean weight of tops from six weeks' growth was .96 gm. with calcium sulfate; .80 gm. with calcium nitrate, and .20 gm. with calcium phosphate.

WHEN DOES ALFALFA MOST NEED SULFUR?

TABLE 6

Six series of cultures were arranged to determine more definitely the time in the growth period up to the blooming stage when alfalfa makes the maximum demand for sulfur. Three series were started in complete solutions while three companion series were in sulfur-free solutions. After three weeks the first pair of series of plants was reversed as to sulfur supply. The second pair was interchanged after six weeks, the third pair at nine weeks. These plants were harvested after twelve weeks' growth. The advantage of having sulfate supplied during the first six weeks of this growth period was still evident at harvest time, as shown by figure 7.

In all three cases the plants started in the sulfur-containing solution gave higher yields. There was some indication of benefit in the case of plants removed from sulfur after six weeks, and there was less recovery from lack of sulfur where this element was applied late in the growth period.

TABLE 6

AT WHAT STAGE IN GROWTH PERIOD OF ALFALFA IS SULFUR MOST NEEDED? Twelve weeks' growth period.

Culture series	Treatment	Mean yield in grams		Water requirement tops
		Whole culture	Tops	
28	S first 3 weeks	3.9	2.4±.16	430
28*a*	S after 3 weeks	3.3	2.3±.15	519
29	S first 6 weeks	3.6	2.4±.12	449
29*a*	S after 6 weeks	2.5	1.8±.07	714
30	S first 9 weeks	3.8	2.7±.23	411
30*a*	S after 9 weeks	3.2	2.1±.12	608

Solutions used in this trial were changed each month and analyzed. The sulfate content for solutions containing an initial concentration of 672 parts per million of sulfate at the end of the first month's growth showed a decrease in concentration to 584 parts per million. The solution being renewed, it was decreased in concentration to 450 parts per million the second month. A new solution was reduced

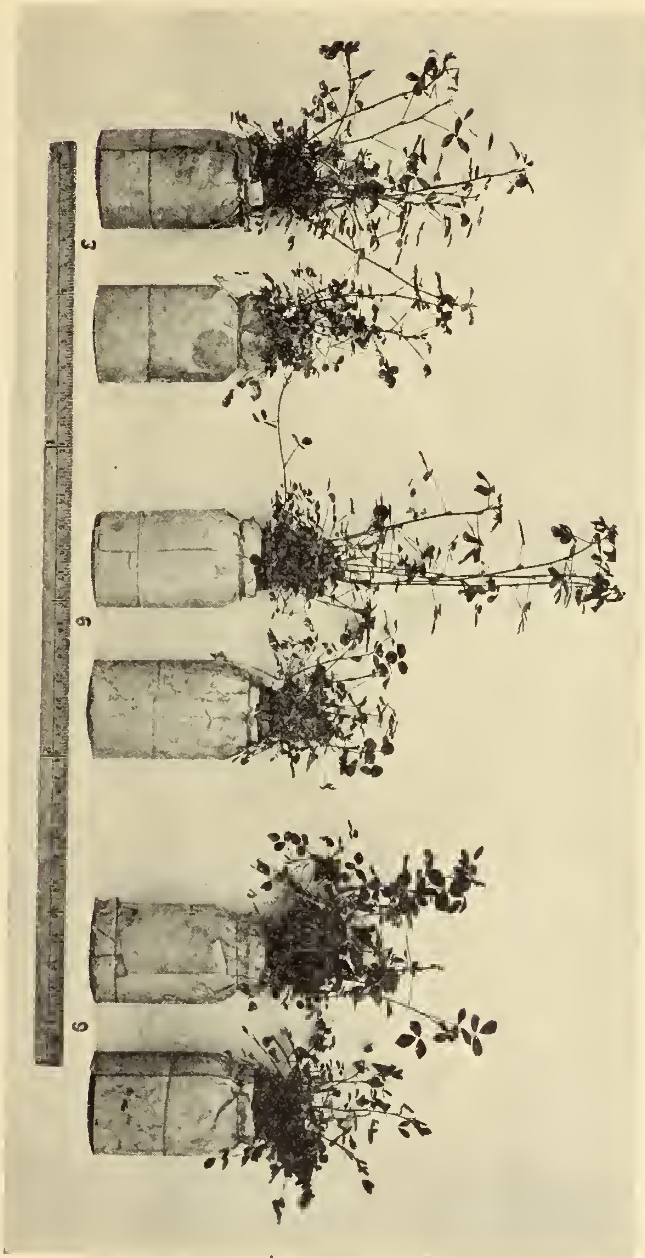


Fig. 7. Plants grown in water cultures containing sulfate at different times in the growth period. First culture at left had sulfate the first three weeks; third culture, the first six weeks; and the fifth culture, the first nine weeks. Cultures 2, 4, and 6 from left received the reverse treatment.

to 612 parts per million the third month. The maximum absorption of sulfur occurred in the second month and the minimum absorption in the third month. From all indications, plants seem to require sulfate largely during the earlier part of the growth period, presumably in building up their system after the seedlings have attained some size. Sulfur applications may increase the sulfofying power of a soil and result in a more favorable concentration of sulfate ion at critical periods of growth. In all these experiments, providing a suitable sulfate supply gave a lower water requirement.

CROP-PRODUCING POWER OF LIMITED AMOUNTS OF SULFUR WITH ALFALFA

Table 7

Eight series of solution cultures were prepared, including sulfur-free controls and solutions containing 5 to 30 parts per million of sulfur as calcium sulfate arranged in increments of 5 parts per million. One part per million is equivalent to 1 milligram per liter or practically the volume of cultures used. Maximum production per milligram sulfur secured was .18 gms. alfalfa and with the solution

TABLE 7

Crop Producing Power of Limited Amounts of Sulfur

Dry matter yield in grams, six weeks' growth.

Series No.	Treatment, approximate (mgm's S)	Mean yield whole plant	Yield tops	Water requirement	Inorganic S as per cent SO_4
42	No S	.55	43± 04	921	.101
36	5 p.p.m. S	.92	.72± 03	486	.086
37	10 p.p.m. S	1 02	.78±.01	449	.127
38	15 p.p.m. S	1.15	.86±.05	412	.223
39	20 p.p.m. S	1.06	82±.04	427	.320
40	25 p.p.m. S	.97	.82±.04	460	.238
41	30 p.p.m. S	1 06	.78± 02	475	.317

containing about 5 parts per million sulfur. Alfalfa plants were grown thereon and when a month old showed a general increase in growth up to the series containing 15 parts per million of sulfur. With greater sulfate treatments the amount of growth was practically uniform. When the plants were six weeks old, 10 parts per million of sulfur appeared to be sufficient, as shown by figure 8. The plants had attained 18 to 20 inches in height, and their requirement for sulfur appeared to be somewhat diminished. There was indication of a slight stimulating effect with 10 parts per million compared to

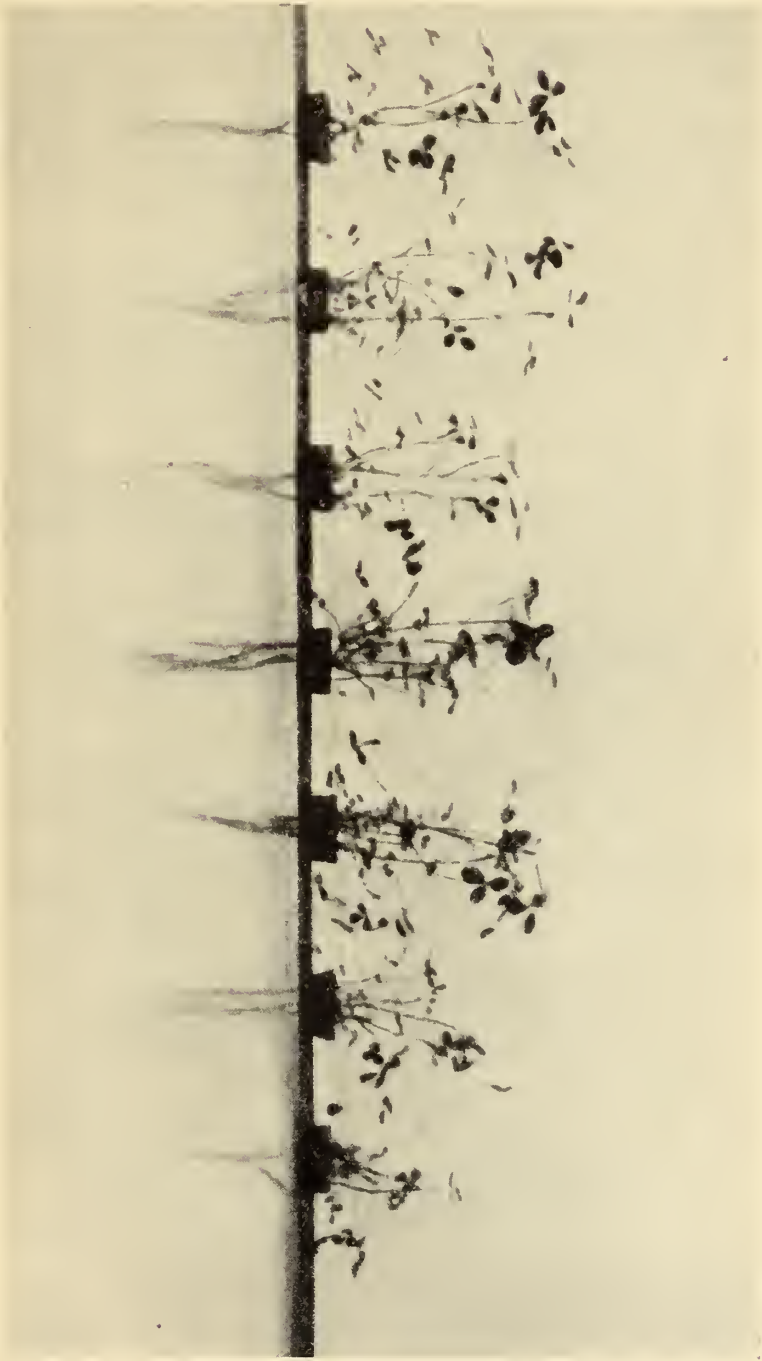


Fig. 8. Alfalfa grown on solutions containing limited quantities of sulfate after five weeks.

two or three times that amount at the later growth state, as shown by figure 9. It was necessary to harvest before the plants were two months old. The maximum yield was secured with 15 parts per million of sulfur in the solution. That there was no significant difference between this and the control or the series receiving larger amounts of sulfate is shown graphically by figure 12. The sulfur-free solution there used was prepared from C. P. chemicals, but it was found to yield 1.6 p.p.m. sulfur in the culture solutions as used.

Some soil solutions from Madera sand were found to contain 150 parts per million of sulfate. Their usable water capacity was less than 10 per cent. An acre-foot of solution would not be stored in less than 10 acre-feet of such soil, giving 14 parts of sulfate per million in the total mass. In the soil, diffusion may be slower than in the culture solution.

The last column in table 7 gives the sulfate recovered by hot water extraction of plants grown in this experiment. A much larger amount of unassimilated sulfate appears to have been present in plants grown on solutions containing 15 p.p.m. or more of sulfur.

CONCENTRATION OF SULFATE NEEDED FOR OPTIMUM GROWTH OF ALFALFA?

Evidence on the least probable concentration needed for optimum growth is scarce. The concentration of different ions necessary in solution cultures or soil solutions is not definitely known.

Cameron[12] matured wheat in tap water which was claimed to contain 0.5 parts per million of phosphorus as phosphate ion.

Burd[10] suggested that, as a result of crop removal, the concentration in a soil solution may fall below supplying the need of certain ions by plants. He reports a two-day nitrate supply as the lowest concentration found under growing barley, as judged by the rate of crop removal. It is further suggested that crop removal may hasten solution from the solid phase of the system.

McCall and Richards[42] found a higher concentration necessary in quartz cultures to give equal effect with water cultures.

Hoagland and Martin[27] point out that suitable concentration is affected by size of culture vessel, plants to be grown, rapidity of growth, reaction, and frequency of renewal of the solution.

In an experiment previously referred to, potassium sulfate greatly increased the yield of alfalfa grown on displaced soil solution, although

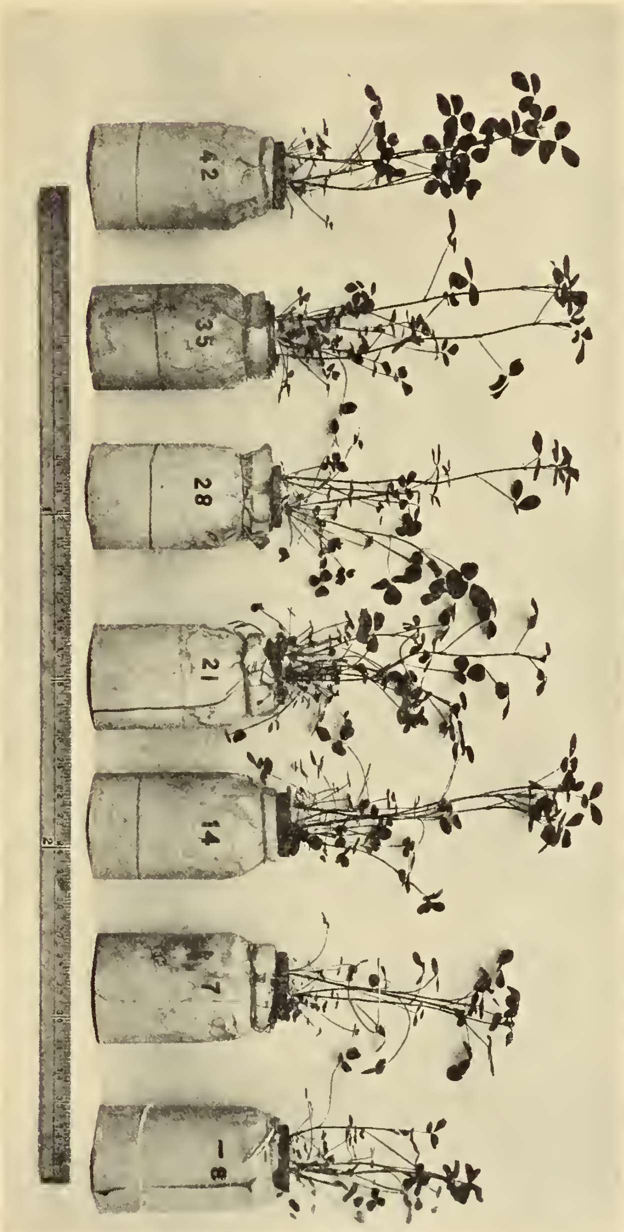


Fig. 9. Alfalfa grown on solutions containing limited quantities of sulfate after eight weeks.

there was already an apparent abundance of potassium present. Calcium sulfate or potassium nitrate increased the yield of grain grown in lysimeter water in recent experiments at the Oregon Experiment Station,[59] even though the soil water was frequently renewed. The study of crop-producing power of limited amounts of sulfate indicated that 10 to 15 parts per million of sulfur or 30 to 45 of sulfate might be needed for best growth of alfalfa. Two soil solutions studied contained 12 and 14 parts per million respectively. An experiment was planned to test the concentration of sulfate needed for alfalfa in culture solutions, and also in a solid medium, as an aid in interpretation of soil solution analyses.

SULFATE CONCENTRATION EXPERIMENT WITH CULTURE SOLUTIONS

To study further the concentration of sulfate needed for optimum growth of alfalfa, a new experiment was planned in which four series of alfalfa plants were set up and grown in each concentration of sulfate employed, so that one series from each lot could be harvested at intervals of 10 days and subjected to chemical tests. Control series were arranged without sulfur and with complete nutrient solutions. The concentrations of sulfur in other series were 2, 4, 8, 16, 32, and 64 parts per million, respectively. Within 10 days the plants without sulfur were making poorer growth than the remaining series. The plants had been set on the jars when the fourth leaf appeared on the seedlings. Up to 20 days' growth 16 parts per million of the element sulfur in sulfate form caused a more rapid growth than a lesser concentration. Before the 30-day period a concentration of 8 parts per million appeared to be sufficient and this condition obtained, so far as could be judged by height and appearance of plants, to the close of the experiment. The amount of growth for different concentrations is indicated in figure 10.

INORGANIC SULFUR

To arrive at a procedure for studying the unassimilated or inorganic sulfate in different lots of these plants at different stages of growth, samples of alfalfa grown on field plats at Delhi were digested for 8 hours, in one case with hot, and the other with cold water. The pulp was filtered out, washed, and the extract acidified, redigested, and filtered free from protein. The sulfate was then precipitated with barium chloride, ignited, and weighed. Further tests were made

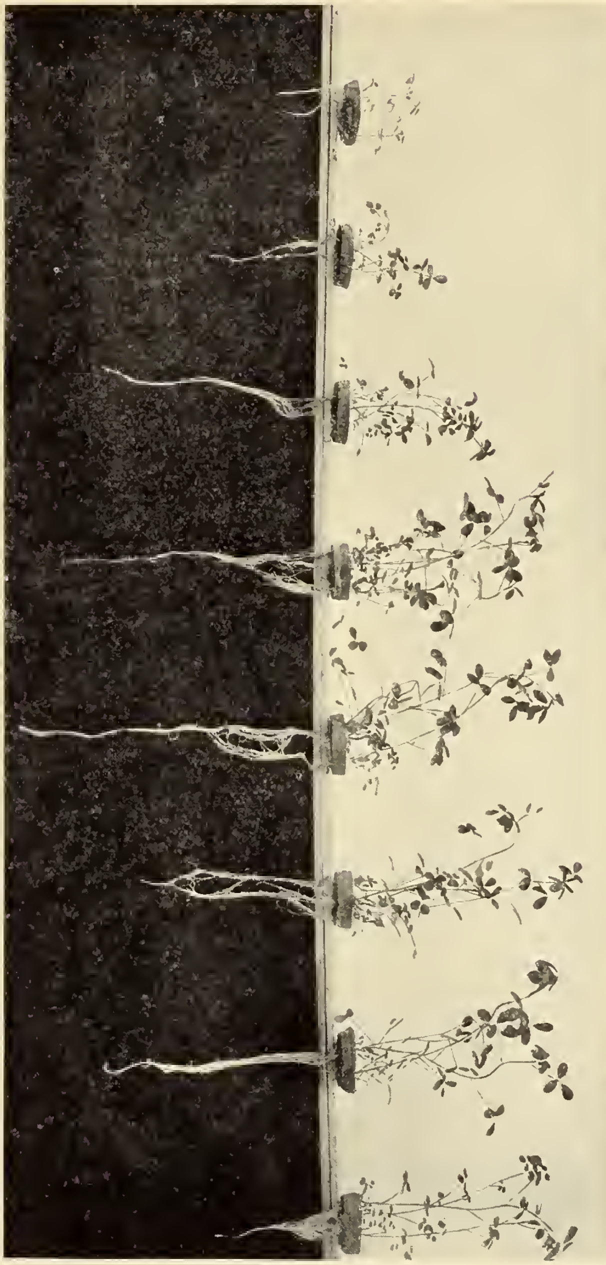


Fig. 10. Effect of various sulfate concentrations on alfalfa.

by an additional 4 hours' digestion of the pulp, which indicated that digestion with hot water for a period of 12 hours was desirable.

A second preliminary experiment was conducted on 1 gram portions of alfalfa meal diluted with water up to 10, 25, 50, and 100 times. Little advantage was found in diluting above 25 times and none above 50 times. Grimm alfalfa seed carried through this determination yielded 0.3 per cent of sulfate.

The alfalfa grown in water cultures with limited amounts of sulfate when analyzed by this method showed a rapid increase in sulfate content up to 15 parts per million, a further gradual increase up to 30 parts per million, and little or no increase thereafter (table 7). In other words, the supply of inorganic sulfates in these young plants increased with the growth curve. Peterson[48] and Hall[20] seem to find only a small portion of sulfur in alfalfa plants in inorganic form. Determinations of organic sulfur from the residue of 30-day-old plants were made and indicate a reciprocal relation to the inorganic sulfur content.

YIELD AND INORGANIC SULFATE CONTENT AS AFFECTED BY SULFATE CONCENTRATION?

Table 8

In table 8 are presented the yield and the hot water extractable sulfate from alfalfa plants grown in solutions with definite concentrations of sulfate, maintained by renewing the solution every three days. The yield of dry matter in plant tops was increased by supplying sulfur up to 8 to 16 parts per million as sulfate in the solution. During the earliest part of the experiment 16 parts per million of sulfur seemed to give better growth than 8 parts per million. Later in the experiment the lower concentration appeared to be fairly adequate. Sulfate determinations indicated that some ex-osmosis of sulfate occurred for higher concentrations the last 10 days of the trial. With these plants there was further evidence of some stimulation about the least optimum concentration, as shown graphically in figure 13.

The sulfate extractable in hot water increased rapidly up to the least concentration necessary for optimum growth. A greater supply of inorganic, or hot water extractable sulfate, was found in the plants at 30 days of age than at earlier or later growth periods. It appears that as fast as the plants gain some capacity a considerable amount

Fig. 11. Effect of hydrogen ion concentration on inoculated alfalfa in culture solutions with little nitrate.

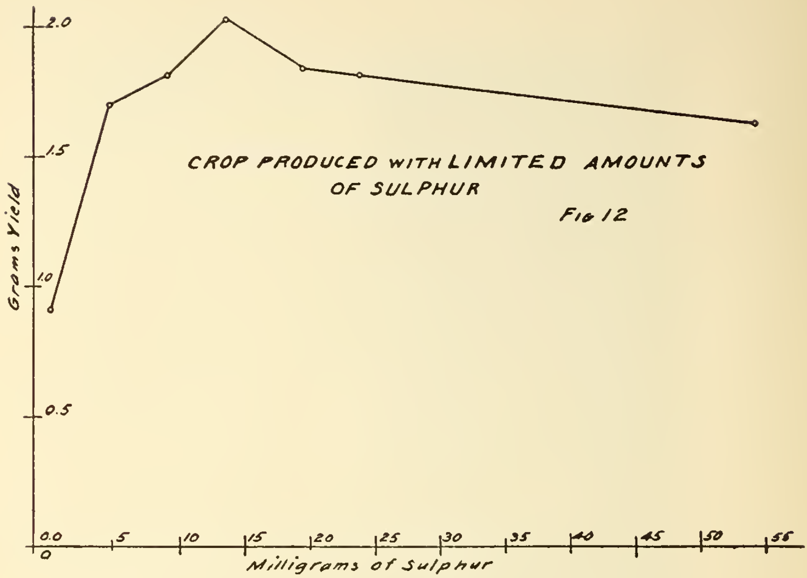


Fig. 12. Graph showing yield from limited amounts of sulfur.

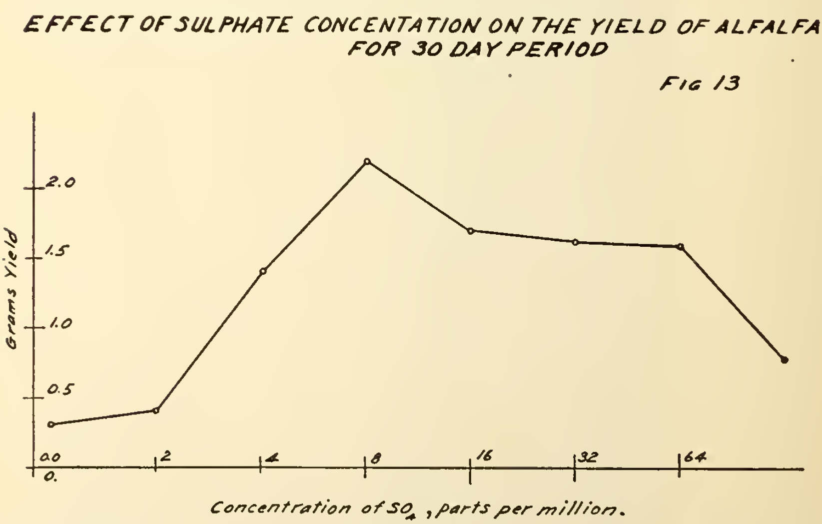


Fig. 13. Graph showing yield from culture solutions maintained with different concentrations of sulfur.

of sulfate is taken in, and later this sulfate is assimilated. Plants grown in the plant house at the University of California were benefited rather than injured by removing them from a complete solution after the first six weeks to a solution lacking sulfate, and they appeared to have sufficient sulfate to carry them up to the blooming period.

TABLE 8

EFFECT OF SULFATE CONCENTRATION OF CULTURE SOLUTION ON YIELD AND SULFATE CONTENT OF ALFALFA AT DIFFERENT STAGES OF GROWTH

Sulfur p.p.m. in culture solution (added as $CaSO_4$)	Mean yield in grams dry plant tops				Plant sulfate extractable with hot water, per cent		
	Growth period—days				Age of plants—days		
	10	20	30	40	10 and 20	30	40
None	.01± .001	.02± .002	.05± .006	.09± .002	.000	.150	.074
2	.01± .001	.03± .002	.07± .003	.23± .006	.068	.652	.291
4	.02± .001	.04± .002	.23± .005	.63±	.217	.750	.285
8	.02± .000	.09± .005	.37± .007	.79± .016	.440	.886	.255
16	.02± .000	.07± .003	.28± .005	.85± .027	.506	1.165	.259
32	.02± .001	.09± .004	.28± .007	.79± .018	.386	.896	.350
64	.02± .001	.10± .005	.27± .011	.76± .021	.929	.975	.448
Complete solution	.02± .004	.04± .002	.14± .003	.43± .012	.979	1.410	.317

From these studies it appears that 8 to 16 parts per million of sulfur or 24 to 48 parts per million expressed as sulfate, depending on age, is sufficient for best growth of alfalfa for these conditions. The sulfate contents of displaced soil solutions from a half-dozen representative Oregon soils (that have been included in sulfur experiments seven to ten years both with and without sulfur) reported above, throw further light on this problem.

SULFATE CONCENTRATION EXPERIMENT WITH SOLID CULTURE MEDIUM

A parallel experiment was conducted in pots of quartz sand to note the effect of a solid medium on concentration and diffusion. Duplicate one-gallon jars of washed Ottawa silica sand were arranged so that, with the aid of an aspirator, solutions could be removed for renewal. Five alfalfa plants were grown in each jar. The yield was not increased with solutions containing more than 8 parts per million of sulfur. It is possible that transpiration changed the concentration of sulfate during the 3-day intervals between solution changes, although a little free liquid was present in the bottom of each jar.

There may have been a concentration of sulfate at the surfaces of the quartz grains.

The rate of diffusion of sulfate ion may be expected to vary with a number of factors and a study is under way in quartz flour and quartz sand.

Two lots of quartz flour and two of quartz sand moistened to the moisture equivalent point were placed in trays $25 \times 25 \times 60$ cm. The two sides, one moistened with .01 N KCl, the other with .01 N H_2SO_4, were brought into contact and kept air tight at uniform temperature. In 45 days quantitative determinations of 1:1 extracts showed that sulfate had diffused 4 to 6 cm. in the quartz flour and 8–10 cm. in the sand. After 90 days a second test showed that diffusion had proceeded at a uniform rate. Apparently roots go to the nutrient more than nutrient goes to the roots. Concentration due to transpiration and surfaces may tend to compensate for slower diffusion in solid medium than in solution cultures. Results indicate that a suitable sulfate concentration in medium sand is not greatly different from that needed in water cultures.

REACTION STUDIES

The reaction of the solutions used in the water culture studies above described was corrected almost daily where necessary, to keep it between pH 5.5 and 6.0, which appears to be most favorable for alfalfa in solution cultures. At or above pH 6.5 a lighter green color developed in tops and a brown color on roots. Below pH 4.8 roots became dull in appearance and growth was retarded.

One culture series, table 5, *c*, was set with barley seedlings to compare the rate of change in reaction in a single salt solution of ammonium sulfate with grain to that induced by alfalfa. The reaction became unfavorably acid with grain in about half the time required with alfalfa. Alfalfa removes sulfate from solution at about the same rate as ammonium ion and affords less opportunity for the sulfate radicle to accumulate in the solution and, by combination with water, to increase the concentration of hydrogen-ions.

To learn more definitely the reaction best suited to inoculated alfalfa, water culture solutions were prepared containing only 20 parts per million nitrogen and with different pH values.* Eight

* This experiment was conducted in cooperation with Mr. Charles Hartmann, Jr., former Assistant in Soils, Oregon Agricultural Experiment Station.

stoneware jars, each of 4-gallon capacity, were provided with covers so as to support 25 plants per jar and were filled with nutrient solutions. Two jars were filled with "complete" nutrient solutions and adjusted to a pH value of 5.8. The other solutions were of similar salt proportions except that little nitrate was present. The reaction was stabilized by addition of potassium acid thalate in all cases equivalent to 200 parts per million. Two jars were made up to pH 5.2, a second pair to pH 6.0, and a third pair to pH 7.2. A fair growth was obtained in all but the alkaline solutions. The average yield for controls for 2 months' growth was .35 gram tops per plant. Cultures maintained at pH 7.2 yielded .13 gram of tops, and those kept at pH 5.2 yielded .21 gram tops per plant.

The greatest development of nodules occurred on roots of plants grown at pH 6.0. Nodules developed in the control the last week of the experiment. The range of pH in which alfalfa can grow appears to be about pH 4.8 to 7.0 and to be wider than for alfalfa bacteria, which will not tolerate such an acid solution as will alfalfa. Alfalfa appears to do best in a slightly acid culture medium. The equipment used and the comparative growth in different solutions after 5 weeks is shown in figure 11.

DISCUSSION

Is Sulfate Concentration in Soil Solutions Sometimes Too Low for Best Growth?

A study of gains and losses of soil sulfur, its oxidation, and the seasonal sulfate concentration in the soil solution, strongly indicates that certain soils at times have an unfavorably low supply of this nutrient. Sulfate concentration in the soil solution is apparently more closely related to the sulfofying power of a soil than to its total sulfur content. Many northwestern soils, however, with only 150 to 400 pounds total sulfur in the plowed surface of an acre, respond to sulfur applications, while relatively few having more than 500 or 600 pounds of sulfur in 2,000,000 of surface soil give much response in the way of increased yield from sulfur applications. In the 1916 series of Reimer plats on Antelope clay adobe, near Medford, any sulfur-bearing fertilizer has strikingly increased alfalfa yields. Sulfur-free fertilizers supplying soluble phosphates, potassium, or nitrates have not materially increased yields. Moreover, calcium compounds in these trials have not caused larger crops.[50] These facts led to the view that many basaltic soils in the northwest were in need of sulfur per se. Studies herein reported have developed good evidence that unfavorably low concentrations of sulfate are less general than formerly supposed, yet they may be found at times in certain soils.

Reviewing the evidence here presented, it is noted that certain soil solutions were found which in certain cases yielded but 14 and 16 parts per million of sulfate. For a given set of conditions, the crop-producing efficiency of a limited amount of sulfur decreases below a certain minimum. Sulfur in alfalfa seed seems to be insufficient to develop properly and to mature alfalfa plants. Further, the least optimum concentration of sulfate for alfalfa appears to range from 48 to 24 parts per million during the early weeks of the growth period. The maximum demand and perhaps the whole need of sulfate, moreover, is met during these early weeks of the growth cycle, when sulfur oxidation on certain soils has been slow and sulfates in the soil may have been depleted by leaching in the wet season. Diffusion in medium-textured soils may be much slower than in water culture solutions. Numerous factors will affect the concentration needed, as pointed out by Hoagland and Martin.[27] Johnson's work[31] seemed to indicate that reaction may affect sulfate absorption by plants from solution of low sulfate concentration; also, that acid or humid soil may supply sulfate needed from a lower concentration than that in neutral soils, and that sulfate production in soil is increased by cropping. Diffusion will vary with temperature, surface tension, texture, and moisture content. Sulfate additions to certain soil solutions and lysimeter waters[59] used as culture media for alfalfa and grain seedlings have increased the growth of these plants.

The majority of soils studied which respond to sulfur applications with alfalfa have 100 parts per million or more of sulfate, and other reasons for the marked increases in yields secured from this treatment must be found.

Does Sulfur Serve to Hold Calcium and Other Bases in Solution?

It has been noted in lysimeter studies at different experiment stations that there is a mutual effect of calcium and of sulfate on the composition of the percolate. Supplying either of these ions tends to increase the amount of the other found in a given amount of percolate from lysimeters.[59]

The most striking and general effect of sulfur on the soil solution encountered in these experiments is the increase in concentration of calcium, following application of sulfur or sulfate. This has been true not only for fallowed pot experiments but for samples from sulfured and unsulfured field soils. In certain cases, as with Deschutes sandy loam, potassium concentration in the soil solution was greatly increased following sulfur treatment. Increase in magnesium in the soil solution is also noticeable following sulfur application. The calcium in solution is frequently doubled or trebled as a result of sulfur application.

Water culture experiments show that where sulfates of calcium, potassium, or magnesium are supplied to alfalfa plants in partial nutrient solutions, 2 days in 6, calcium sulfate is the most favorable form and results in the largest amount of growth. Inversely, when calcium is supplied in partial solutions as sulfate, nitrate, or phosphate, the calcium sulfate is a very favorable source of calcium.

The amount of readily soluble and of replaceable calcium and other bases present in a soil, as indicated by Kelley,[33] is closely related to the composition of the soil solution. Madera sand, after 6 or 12 weeks of incubation with sulfur, was found to have released large amounts of calcium to the liquid phase. The replaceable calcium in this soil is low and some carbonates probably were dissolved. A year later the amount of calcium in solution had greatly decreased and the potassium in solution had markedly increased. Either there had been a slow solubility effect on relatively insoluble potassium-bearing compounds in the solid phase, or, what is more probable, the calcium ion brought into solution from carbonates had participated in a base exchange with the base-absorbing complex of the soil.

An important function of sulfur or other anions, as pointed out recently by Burd,[8] appears to be that of holding cations in solution and thus maintaining a favorable concentration of nutrients in the soil solution. He has suggested that when nitrate is largely removed by growing crops the sulfate operates to perform this function and to keep bases in available form. A 4-ton crop of alfalfa requires about 300 pounds of calcium.[32] The concentration of calcium ion in the soil solution for some of the soil types studied has been found to be as low as 20 or 30 parts per million at certain times. At present it appears that one of the leading effects of sulfur is to bring calcium and other bases into the soil solution and to hold them there.

Will the Average Application of Sulfur Hasten Soil Deterioration?

Recent studies by Gedroiz[17] and others indicate that the replaceable bases adsorbed by the soil-adsorbing complex of a fertile soil should be mainly calcium or the bivalent bases, calcium and magnesium. Further, that when this adsorbing complex becomes unsaturated with bases, as in aged acid soils, and contains much adsorbed hydrogen ion instead, these complex silicates may tend to become unstable and to deteriorate into the simpler oxides, namely, iron, silica, and aluminum oxides. The result may be a denser structure and a loss of base-adsorbing capacity. Perhaps a similar condition may come about with a soil impoverished of replaceable calcium in the case of a sodium-saturated, adsorbing complex with a ''black alkali'' condition, which may be a possible cause of ''slick spots.'' It is conceivable that heavy and continued applications of sulfur may hasten removal of calcium ion and ultimately lead to soil deterioration, especially under conditions favorable for leaching. After an initial application, subsequent sulfur treatments are often effective if applied at a lighter rate. Where fertilized alfalfa is consumed on the farm and over-irrigation is avoided, the increase in organic matter caused by moderate applications (that is, 80 to 100 pounds an acre) of sulfur every 3 or 4 years may have little effect on soil deterioration. It would seem that the use of sulfur as a fertilizer may be more safely practiced on basaltic soils that are liberally supplied with different forms of calcium.

Does Sulfur Improve Reaction of Arid Soils for Alfalfa?

Nitrate is known to be taken into the plant better under slightly acid conditions, and sulfur may improve reaction for nitrate adsorption by alfalfa. Johnston seems to find[31] that sulfate is taken up by alfalfa best when the pH value of the culture medium is about 5.8. Iron and phosphate are known to be relatively insoluble under alkaline conditions. Sulfur tends to increase the solubility of these nutrients up to a point where the calcium dissolved begins to react and cause reprecipitation. Sulfur doubled the amount of iron in solution in Antelope clay adobe. Ferric chloride applied by spraying on young growth on this soil resulted in the same improvement in color and yield that has been characteristic of sulfur-treated plats.

In nutrient solutions correction of reaction by addition of dilute sulfuric acid secured larger growth and better color of alfalfa than resulted from addition of hydrochloric acid. This sulfate may have affected the form of iron in the culture solution or it may have acted directly. Possibly the chlorine in the small amount added was injurious. Alfalfa grows best in a slightly acid medium, as indicated by Theron[57] and confirmed herein. The best reaction for the alfalfa-radicicola combination was found to be about pH 6.0.

Sulfur has been found effective in improving the reaction and structure of alkali soil at Kearney Park Experiment Field, California, and at Vale Experiment Field in Eastern Oregon.[30] Sulfur application may result in improved permeability in alkali land, and, by improvement in soil structure or possibly by modification of surface tension, may render soil more drought-resistant.

There are numerous other effects of sulfur on physical, chemical, and biological conditions related to plant nutrition. The three factors discussed above have stood out as being of chief importance in these studies with the soil solution. Which of these effects will be of major importance may depend on the particular soil and conditions at hand.

SUMMARY

1. Sulfur and sulfates applied to Madera sand soil in pot tests caused marked increase in calcium ion and a definite increase in other bases in the displaced soil solution. Calcium and sulfate ions go into the alfalfa plant especially well together.

2. Heavy applications of sulfur resulted in increased soil acidity, which caused an increase in phosphate and iron content of the soil solution up to a certain point, after which bases dissolved or replaced tended to precipitate these two ions from the soil solution.

3. Heavy application of sulfur tended to inhibit nitrification, though the normal application, or 100 pounds per acre, on arid soils may increase growth and nitrogen in the soil.

4. Evidence was found of base exchange as sulfur oxidation increased the concentration of hydrogen ion and then of other cations. Fixation and exchange of bases applied in sulfates, as in potassium sulfate, was noted.

5. Analyses of displaced soil solutions of several sulfured and unsulfured soils from fertilizer experiment fields tend to confirm results secured with Madera sand and further indicate that the sulfate content of some soils at certain seasons is very low. Further, that the effect of sulfur will depend much upon the particular soil at hand.

6. Sulfur is needed most by alfalfa during the early weeks of the growth period. Sulfur applications increase sulfofication and the sulfate content of the soil solution, and they may in turn serve to bring bases into solution, resulting in a more concentrated soil solution and decreased transpiration.

7. Water culture experiments indicate that a concentration of 48 to 24 parts per million of sulfate is most favorable for the growth of alfalfa under the conditions of the trial. The maximum production secured per milligram of sulfur was .18 gm. alfalfa and was produced with a solution having an initial sulfate content of 15 parts per million.

8. An average application of sulfur appears to improve the reaction of arid soils for alfalfa nutrition, resulting in increased growth and higher chlorophyll and sulfate content.

9. It is concluded (*a*) that some soils may have a sulfate content which is unfavorably low for best growth of alfalfa, especially early in the growth period; (*b*) that sulfur oxidizes to sulfate and brings additional calcium and other bases into solution; (*c*) that sulfur in moderate amounts improves the reaction of arid soils for alfalfa nutrition; (*d*) that the sulfur applications which are of greatest benefit will depend on the soil at hand; and (*e*) that ordinary applications of sulfur for alfalfa on the arid basaltic soils or soils liberally supplied with calcium compounds is probably good practice, especially where the growth secured is consumed on the farm.

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