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A Lysimeter Study of Sulfur Fertilization of an Annual-Range Soil¹

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Deficiency of the plant nutrient sulfur is widespread on California soils. The majority of 242 responding sites recorded in a recent sulfur deficiency survey are on range or dry-farmed land (Martin, 1958). The Leguminosae are the group of plants that respond to sulfur fertilization most frequently. Several authors have pointed out the benefits from supplying additional sulfur where deficiencies occur (Conrad, 1950; Bentley and Green, 1954; Arkley, *et al.*, 1955 and Walker, 1957). The economics of the practice appear promising, because carriers of the sulfur are relatively inexpensive. However, choosing a rate and frequency of sulfur fertilization and source of sulfur that will maximize returns is a problem. The solution depends on detailed knowledge of such factors as the availability of sulfur in the soil, sulfur supplied by precipitation and air contact, leaching losses, erosion losses, and the differential uptake by various plant

species. The first of a series of lysimeter investigations to study these factors in relation to the nutrition and production of a range legume was initiated at the San Joaquin Experimental Range². Lysimetry was the technique chosen for studying these problems because sulfur leaches readily as the sulfate ion (Stauffer and Rust, 1954) and, therefore, study of the percolate was of prime interest. Several workers (Kohnke, *et al.*, 1940; Harrold and Dreibelbis, 1951; Stauffer and Rust, 1954, and Dreibelbis and McGuinness, 1957) reviewed the literature on lysimeter construction and discussed the advantages and problems of lysimetry. The radioisotope S³⁵ was incorporated in the gypsum fertilizer so that the fate of applied sulfur could be distinguished from that of sulfur from natural sources.

Methods

The lysimeters used for this study are 74 inches in diameter with side walls 25.5 inches deep. Each contains an 8-inch deep conical bottom drained by a polyethylene pipe leading to a 5-gallon glass carboy (Fig. 1). Lysimeter interiors were painted with asphaltum paint. During February 1957 the lysimeters

were installed on a hillside terrace with rims extending 2 inches above the ground surface.

A soil profile was reconstituted in the tanks by stockpiling soil from 0 to 1 inch, 1 to 6 inches, 6 to 12, and 12 to 24 inches, and then placing this soil in the proper sequence in the lysimeters. The soil settled approximately 2 inches during the first spring and summer with essentially no later subsidence. The soil used in this study is Vista sandy loam, an upland soil derived from granitic parent material. The soil contains 75 percent sand, 17 percent silt and

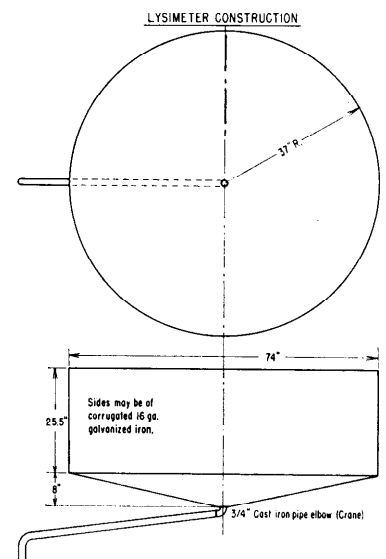


FIGURE 1. Diagram of lysimeter construction.

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8 percent clay and has a mean bulk density of 1.43 gms/cc in the lysimeters. It was characterized by analysis of the 6-12 inch depth in the lysimeters: pH 6.2; organic matter 0.61 percent; P 12.1 ppm; K 0.20 ppm; Ca 2.8 ppm; Na 0.50 ppm; Mg 0.35 ppm; total N 0.029 percent; conductivity 0.40 mmhos/cm, and cation exchange capacity 4.16 me/100 g. The Vista series is a permeable soil with depth of 21 to 36 inches and occurs extensively in the 10- to 20-inch rainfall zone of the Sierra Nevada foothills.

In October 28, 1957 the following treatments were randomized among the lysimeters: Check, 100, 200, and 300 pounds gypsum per acre. There were three check lysimeters, and two of each gypsum rate. The gypsum used at the 100- and 300-pound rates was labeled with approximately 2.5 mc. S^{35} activity per lysimeter and was broadcast as fine powder. All the lysimeters and the adjacent area were seeded with inoculated rose clover (*Trifolium hirtum* All.) at a rate of 50 pounds per acre and covered with $\frac{1}{4}$ inch of soil previously removed from the surface. A high rate of seeding was used to insure a complete stand of plants. As a precautionary measure against rodent and bird damage and contamination of the surrounding area with the radioisotope, each lysimeter was provided with a wire enclosure.

Percolate and rain water were collected during each storm period, and sulfur was precipitated as barium sulfate and determined gravimetrically (A.O.A.C., 1955). Rain was caught in glazed pots which were covered with aluminum foil during dry periods. The clover was harvested on May 9, 1958, at the full-bloom stage for yield determination. Clover samples were oxidized by the magnesium nitrate method (A.O.A.C., 1955) and sulfur determined (Johnson and Nishita,

1952). Radio-sulfur activity was determined on infinitely thick samples of barium sulfate (Hendricks, *et al.*) in a windowless gas flow counter. Smaller samples were corrected to infinite thickness from an appropriately determined calibration curve. Lead peroxide candles were exposed at the location to determine the sulfur dioxide content of the atmosphere (Alway, *et al.*, 1937).

Soil samples were obtained from four depths (0-1, 1-6, 6-12 and 12-24 inches) at the end of the growing season. These samples were extracted with Morgan's reagent (sodium acetate in acetic acid, pH 4.8) and analyzed by the method described by Johnson and Nishita (1952) for the microestimation of sulfur³.

Results and Discussion

As might be anticipated from knowledge of the solubility of calcium sulfate in the soil solution (Vanoni and Conrad, 1942), the sulfur in gypsum is very susceptible to leaching loss when applied to a coarse-textured soil. In this experiment sulfate sulfur⁴ was lost from all treated tanks at a rapid rate in the initial percolates from early-winter rains (Fig. 2). The magnitude of

the loss was proportional to the amount of gypsum applied. As the rainy season progressed the rate of sulfur loss per unit of percolate gradually declined and towards the end of the season approached asymptotic values for all treatments. The curves in Figure 2 also show that roughly comparable amounts of sulfur between adjacent treatment levels were leached by the end of the 1957-58 season: 15.0 pounds per acre for the first 100 pound increment of gypsum applied per acre, 18.5 pounds for the second increment, and 11.1 pounds for the third increment.

In view of this observation and because of the similarity in shape of the cumulative leached sulfur curves, Figure 3 was drawn. The cumulative amount

³ The authors wish to express sincere appreciation to J. E. Ruckman, S. S. Winans, and D. P. Ormrod, who helped in the collection of samples and the performance of chemical and radiological analyses. Preparation of radioactive fertilizer by Fertilizer Investigations Research, ARS, USDA is also appreciated.

⁴ For simplicity subsequent references to sulfate sulfur in percolate will be designated as sulfur.

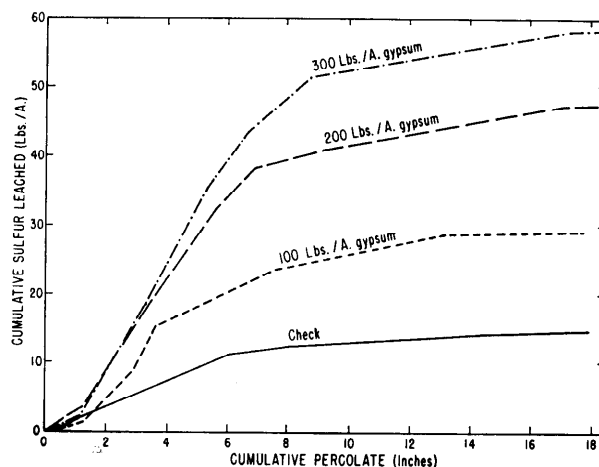


FIGURE 2. Cumulative amount of sulfur leached as a function of the amount of percolate from lysimeters receiving several rates of gypsum.

of sulfur lost by leaching was plotted as a percent of the total against the cumulative percolate expressed as a percent of the total percolate. The near equivalence of the curves for the three rates of gypsum and also the check treatment is striking. Each treatment, including the check, lost essentially the same percentage of the total leached sulfur with each increment of percolate. The curves of Figure 3 indicate that the amount of water passing through the soil was sufficient to maintain maximum solubility. Figure 3 also illustrates that the first 50 percent of the percolate carried down an average of 89.4 percent of the total leachable sulfur for all treatments. In 1957-58, a year of exceedingly heavy rainfall (31.8 inches), the first 50 percent of the percolate resulted from an amount of precipitation very nearly equal to the annual mean for the site (19.4 inches).

A large proportion of the sulfur applied in the gypsum was lost in the percolate as indicated by recovery of the radioisotope S^{35} . 77.0 and 77.9 percent of the sulfur applied in the 100- and 300-pound rates, respectively, were accounted for in the perco-

late collections (Fig. 4). The gypsum applied in the 200-pound treatment was not labeled, but there is no reason to expect that its fate would differ appreciably from the 100- and 300-pound treatments.

The sulfur brought down in rainfall was 21.4 pounds per acre during the 1957-58 season, a rather appreciable amount for an agricultural area (Jordan, *et al.*, 1959). There was considerable variation in the sulfur content of rain from the season's storms (Table 1). Concentration of sulfur in rain water ranged from a low of 0.50 ppm to a high of 4.70 pp. It was expected that the sulfur concentration would be high in the first fall rains and then would decline as sulfur in the air was washed out by additional storms (Seay, 1957). However, the data indicate that sulfur content of rain was as high in the last storms of the season as it was in the first storms, and no particular trend was evident.

Lead peroxide candles at the site did not show any appreciable amounts of sulfur in the local atmosphere; 100 sq. cm. of the exposed surface of the candles absorbed $0.50 + 0.11$ mg. sulfur.

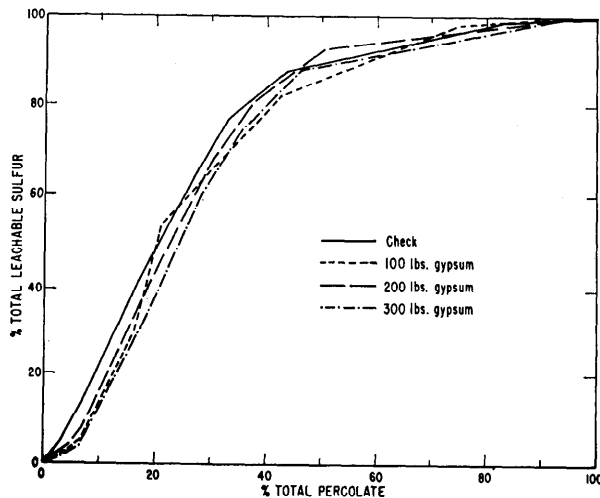


FIGURE 3. Relative rate of loss of sulfur by leaching as influenced by the rate of gypsum application.

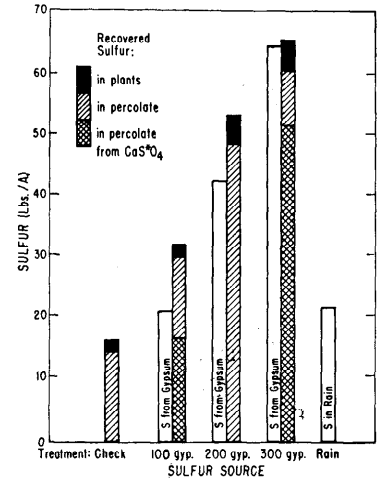


FIGURE 4. Sulfur added in gypsum and rainwater and recovered in clover plants and percolate.

Using a conversion value of 22 percent (average of data from Alway, *et al.*, 1937) to relate the surface absorption of the candles to soil absorption, the amount of sulfur absorbed from the atmosphere by the soil surface was almost negligible, 0.1 pound per acre. Apparently the sulfur brought down in the rain was

Table 1. Sulfur content of rain-water collected at the San Joaquin Experimental Range lysimeter site July 1, 1957 to June 30, 1958.

| Collection interval | Rain (in.) | Sulfur content (ppm) |
|---------------------|------------|----------------------|
| 9/17 - 12/3 | 2.99 | 2.75 |
| 12/4 - 5 | .54 | .50 |
| 12/6 - 16 | 1.73 | 3.61 |
| 12/17 - 18 | .54 | 3.25 |
| 12/19 - 22 | .18 | 4.70 |
| 12/23 - 1/9 | .27 | 2.32 |
| 1/10 - 24 | 2.10 | 3.88 |
| 1/25 - 27 | 1.66 | 2.35 |
| 1/27 - 2/5 | 2.73 | 3.45 |
| 2/6 - 13 | .68 | 1.32 |
| 2/14 - 19 | 1.30 | .85 |
| 2/20 - 25 | 2.57 | 2.50 |
| 2/26 - 3/17 | 6.14 | 2.73 |
| 3/18 - 24 | 3.20 | 3.47 |
| 3/25 - 4/7 | 5.14 | 3.61 |

Season total 31.77 W'td. mean 2.97

picked up in air masses some distance from the site.

Clover growth was stimulated by the gypsum applications. Yields of clover were significantly greater on the 200- and 300-pound treatments than on the check and 100-pound treatments (Table 2). The increases in sulfur content of the plant tissues were not significant. Often an increase in sulfur occurs when gypsum is applied to legumes growing on sulfur-deficient soils (Arkley, *et al.*, 1955; Bentley, *et al.*, 1955, and Walker, 1957). However, most of the leachable sulfur had been lost by the first week of March in this experiment. Rapid spring growth was initiated by the clover at approximately this time, and as a result, high concentrations of sulfur were not available for luxury consumption during the period of rapid plant growth.

Radioassay indicated that clover grown on the 100- and 300-pound treated lysimeters obtained an average of 30.8 and 57.4 percent of the sulfur, respectively, from gypsum, the proportion increasing at the higher level of application. Recovery of sulfur from applied gypsum by the clover amounted to a 2.8 and 6.7 percent, respectively.

A sulfur-balance sheet was constructed for each treatment using the data for additions to and losses from the soil (Table 3). Additions to the soil were

Table 3. Additions, losses, and apparent adsorption of sulfur by Vista sandy loam treated with gypsum.

| Item | Sulfur per acre (lbs.) from lysimeters treated with indicated gypsum per acre | | | |
|------------------------------------|---|-----------|-----------|-----------|
| | 0 | 100 (lbs) | 200 (lbs) | 300 (lbs) |
| Sulfur added from: | | | | |
| Gypsum | 0.0 | 21.3 | 42.7 | 74.3 |
| Rain | 21.4 | 21.4 | 21.4 | 21.4 |
| Air | 0.1 | 0.1 | 0.1 | 0.1 |
| Seed | 0.1 | 0.1 | 0.1 | 0.1 |
| Total | 21.6 | 42.9 | 64.3 | 85.9 |
| Sulfur lost in: | | | | |
| Percolate (gypsum) | | -16.4 | -47.7* | -50.1 |
| Percolate (rain, air and seed) | -14.2 | -12.8 | | - 8.7 |
| Crop removal (gypsum) | | - 0.6 | | - 4.3 |
| Crop removal (rain, air and seed) | - 2.5 | - 1.3 | - 6.0* | - 3.2 |
| Total | -16.7 | -31.1 | -53.7 | -66.3 |
| Calculated sulfur absorption from: | | | | |
| Gypsum | | 4.3 | 10.6* | 9.9 |
| Rain, air and seed | 4.9 | 7.5 | | 9.7 |
| Total | 4.9 | 11.8 | 10.6 | 19.6 |

* Non-labeled gypsum used; thus source of sulfur not distinguishable.

from gypsum, rain water, air contact, and seed. The losses resulted from leaching and crop removal, the former being the greater in magnitude, by far. Calculation of the net change shows that the soil adsorbed more sulfur than it released in all treatments. The net adsorption varied from 4.9 pounds per acre for the check up to 19.6 pounds per acre for the lysimeters receiving the 300-pound

rate of gypsum. Ensminger (1954) demonstrated sulfur adsorption capacity up to as high as 411 ppm in a sandy loam under laboratory conditions. The sulfur adsorbed by the soil from the label gypsum amounted to 20.2 and 15.4 percent of the sulfur applied in the 100- and 300-pound rates, respectively. These data lend support to the conclusion of Kramprath, *et al.* (1956) that the amount of sulfate adsorbed by soil is directly related to the concentration of sulfate in the applied solution. Because of the overriding adsorption effect there is no way to determine whether any sulfur was released from the native soil sulfur content.

Based on the net adsorption of sulfur by the soil of the check lysimeters it is apparent that the heavy rainfall did not leach all available native soil sulfur, but added to it. However, it is ex-

Table 2. Yield and sulfur content of rose clover grown in lysimeters treated with various amounts of gypsum.

| Gypsum treatment (lbs/A.) | Clover yield (lbs/A.) | Clover sulfur content (%) | Sulfur in clover obtained from gypsum (%) | Clover recovery of S from gypsum (%) | Area ¹ of clover (%) |
|---------------------------|-----------------------|---------------------------|---|--------------------------------------|---------------------------------|
| 0 | 2,480 | 0.10 | | | 62 |
| 100 | 1,351 | .14 | 30.8 | 2.8 | 50 |
| 200 | 4,433 | .11 | | | 98 |
| 300 | 5,357 | .14 | 57.4 | 6.7 | 96 |
| LSD (5%) | 3,025 | N. S. | | | |

Table 4. Effect of rate of gypsum application on soil adsorption of sulfur—a balance sheet check.

| Sulfur source | Sulfur per acre (lbs.) from lysimeters treated with indicated gypsum per acre | | | | Gypsum mean |
|---|---|----------|----------|----------|-------------|
| | 0 (Check) | 100 lbs. | 200 lbs. | 300 lbs. | |
| Total S adsorbed | | | | | |
| 1958-59 (Table 3) | 4.9 | 11.8 | 10.6 | 19.6 | |
| Gypsum S adsorbed | | | | | |
| 1958-59* | | 6.9 | 5.7 | 14.7 | 9.1 |
| Total S extracted by Morgan's reagent | 37.5 | 49.7 | 41.9 | 48.8 | |
| Gypsum S extracted by Morgan's reagent* | | 12.2 | 4.4 | 11.3 | 9.3 |

* Values obtained by subtracting sulphur absorbed in check lysimeters from amounts of sulphur absorbed in treated lysimeters.

pected that the contribution of sulfur from rainfall might be less in normal or subnormal rainfall years.

Chemical analysis of the soil at the end of the season showed an average increase of 9.3 pounds per acre of extractable sulfur in the treated lysimeters relative to the checks (Table 4). The increase can be attributed to the adsorption of added gypsum. The above value does not differ significantly from the average increase of 9.1 pounds per acre of sulfur adsorbed from the applied gypsum, as calculated from the balance-sheet data by comparing the amount of sulfur adsorbed in the treated lysimeters with that in the checks. The lack of close agreement among rates of applied gypsum is not surprising since the differences in extractable sulfur represent concentration differences in the neighborhood of 1 ppm extractable sulfur, which stretches the sensitive chemical method used to the lower limit of accuracy. However, these data serve as a worthwhile check on the balance sheet results given in Table 3.

It may be concluded from the preceding discussion that in a wet year gypsum applied to correct a sulfur deficiency may be subject to considerable leaching loss. A high rate of sulfur appli-

cation intended to last for several years could be lost as easily as a lower sulfur application rate intended for one year. Further study under less intense rainfall conditions is desirable.

Summary

A lysimeter study was initiated in the annual-range type to study the fate of sulfur applied in gypsum to an annual-legume, rose clover, on Vista sandy loam. The gypsum leached rapidly in a season of heavy rainfall. Sulfur contributed by rainfall amounted to 21.4 pounds per acre, and sulfur adsorbed from the atmosphere contributed approximately 0.1 pound per acre.

Use of the radioisotope S^{35} permitted identification of fertilizer sulfur in the percolate. In the growing season following fertilization 77.0 percent of the sulfur applied in the 100-pound gypsum rate and 77.9 percent of the sulfur applied in the 300-pound gypsum rate were accounted for in percolate collections.

Rose clover yield responded significantly to the higher levels of gypsum. The clover took up 30.8 and 57.4 percent of its tissue sulfur content from 100 and 300 pound applications, respectively.

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