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LITHIUM TOXICITY IN CITRUS[1]

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Among early reports concerning the possible cause of mottle-leaf in citrus (now known to be due to zinc deficiency) is one by Haas,[3] which describes a type of leaf-mottling that was artificially produced in citrus in sand and soil cultures as well as in the field by use of lithium salts. No confirmatory quantitative data on the Li content of the mottled leaves were reported, however, because the amount present was too small to be detected by the gravimetric procedure used. Haas noted that so far as he was aware, no report ever had been made of Li as a constituent of mottled citrus leaves in the field. No evidence of the occurrence of Li toxicity in other crops in the field could be found in a review of the literature on this element.

Experiments conducted by the authors, involving the application of Li salts to citrus seedlings grown in pots in the greenhouse and to mature citrus trees in the field, confirm the report by Haas on the toxicity of Li to citrus and the leaf symptoms that it produces. A spectrochemical method of analysis developed in this laboratory by Vanselow and Liebig[4] makes it possible to determine the Li content of plants showing toxicity symptoms.

On the basis of artificially produced leaf symptoms, the authors have recognized Li toxicity in citrus in the field and have confirmed the presence of this element by spectrochemical analysis. An account of these findings is presented in this paper.

METHODS OF ANALYSIS

Details of the spectrochemical method of analysis used in this study have been described by Vanselow and Liebig. A brief description follows:

The plant samples, when received, were washed by gently rubbing by hand under a small stream of tap water. They were rinsed with distilled water, wiped dry with clean cloth towels, and dried at 55°C. in a well-ventilated, electrically heated drying oven. After being thoroughly dried, the samples were crushed to suitable fineness by hand. To avoid the possibility of contamination by any metals, no mechanical grinder or disintegrator was used. A suitable amount of each sample was ashed in an electric muffle furnace at a temperature not ex-

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[2] The authors thank Arnold White, farm advisor, Santa Barbara County, California, and Benoist Goulben, senior laboratory assistant in the department of soils and plant nutrition, for their assistance in this investigation.

[3] Haas, A. R. C. Mottle-leaf in citrus artificially produced by lithium. *Bot. Gaz.* 87:630–641. 1929.

[4] Vanselow, A. P., and Liebig, G. F. Jr. Spectrochemical methods for the determination of minor elements in plants, waters, chemicals, and culture media. (Mimeo.) Calif. Agr. Exp. Sta. Berkeley. 1948.

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James Frank Breazeale
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James Frank Breazeale, widely known plant and soil scientist, died at his home in Tucson, Arizona, November 13, 1950.

Born in 1874 in Anderson County, South Carolina, Mr. Breazeale received his scientific training at Clemson College. The first student to register when the college opened its doors in 1893, he graduated with the B.S. degree in 1896.

His scientific career embraced a long period of service with the U. S. Department of Agriculture: as laboratory assistant in the Bureau of Soils, 1903–1907; as assistant chemist with the Bureau of Chemistry 1907–1917; as associate biochemist with the Bureau of Plant Industry, stationed at Chula Vista and Riverside, California, 1917–1921, and at the U. S. Field Station at Sacaton, Arizona, 1921–1923; and as research biochemist with the Bureau of Plant Industry in cooperation with the Arizona Agricultural Experiment Station, at Tucson, from 1923 to his retirement in 1938.

Mr. Breazeale was best known for his researches in soil fertility, plant nutrition, and plant-soil-moisture relationships. He was among the first to postulate that the absorption of nutrient ions by plants is an electrical phenomenon and to demonstrate that the action is independent of the movement of water into or out of the plant. Working with organic matter from plant and animal sources, he showed that manures contain vitaminlike substances which, even in minute amounts, stimulate growth of seedlings. He studied the salt problem of desert soils and methods of reclamation. He was perhaps the first to show that puddling in soils is a function of moisture content, reaching a maximum at or near the moisture equivalent and brought about by mechanical vibration or manipulation. Another of his striking discoveries was the transfer of moisture through the root system of plants from a moist to a dry soil.

Mr. Breazeale's publications numbering well above 60, reflect unusual clarity of reasoning, vividness of description, and thoroughness of experimental proof. He regarded the plant as the final arbiter that should be consulted in all soil fertility investigations. Absolute scientific integrity characterized all his scientific endeavors. He worked with literally thousands of seedlings and pot tests of different plants, demonstrating the correctness of his deductions with simple apparatus and without complicated theory or involved computations. He was truly a trail blazer. A man of rare originality and keen observation, he stimulated all who worked with him to think along new lines.

Mr. Breazeale had an unusually wide range of interests. In 1917 he wrote Farmers' Bulletin 359, "Canning vegetables in the Home," in which he ad-

ceeding 500°C. Ignition was carried out at this low temperature to avoid loss, by volatility, of any of the more readily vaporized minor elements. Ignition at this temperature does not usually yield a white ash, but leaves the ash mainly in the form of carbonates, with small amounts of unburned carbon which in no way interfere with the analysis by the spectrographic method.

Four parts of the sample ash were throughly mixed with one part of internal standard powder consisting of highly purified Na_2SO_4 medicated with the four internal standard elements Tl, Ge, Pd, and Be. Four internal standard elements were used because it has been found that a single internal standard cannot be depended on when all the minor metallic components are determined in one

FIG. 1. LITHIUM-TOXICITY SYMPTOMS ON SWEET ORANGE LEAVES

arcking. Li determinations were made by the method described by Vanselow and Liebig, with Ge as the internal standard.

The determinations were also carried out by a procedure found to be more sensitive and reliable. In this modification, Pd is the internal standard for Li. Craters of purified graphite were packed with 20-mgm. portions of the medicated ash and arcked for 40 seconds with the grating fully open, using a direct current of 12 amperes. The spectrograms, after processing, were measured on a photoelectric densitometer, and the results were computed on the graphic calculator described elsewhere.[5]

RESULTS AND DISCUSSION

Lithium-toxicity symptoms artificially induced

The Li-toxicity symptoms shown in figure 1 occurred on sweet orange seedlings grown in pots in the greenhouse about 6 months after 2 ppm. and 5 ppm. of Li

[5] Vanselow, A. P., and Liebig, G. F. Jr. A graphic calculator for spectrochemical analyses. *Jour. Opt. Soc. Amer.* 34:219–221. 1944.

as Li_2SO_4 were incorporated in the air-dried soil. Similar leaf symptoms were obtained in the field by applying to three series of 10-year-old Eureka lemon trees 12.5, 25.0, and 50.0 gm., respectively, of Li as LiCl. Since the soil area under the trees in the field to which the salt was applied measured 18 feet square, the rates of application of Li, based on the salt incorporated with a foot of soil, were 1.0, 2.0, and 4.0 ppm. The extent to which the Li-excess patterns occurred and the amount of defoliation that accompanied these symptoms in both greenhouse and field experiments were directly related to the amount of Li applied. The Li concentrations in various vegetative parts of the Li-treated sweet orange seedlings and in the leaves and fruit of the Li-treated Eureka lemon trees are

TABLE 1

Li content of vegetative parts of sweet orange seedlings treated with Li_2SO_4

Li applied to soil	Li content					
	Young leaves	Mature leaves	Top bark	Top wood	Root bark	Root wood
ppm.	*ppm.**	*ppm.**	*ppm.**	*ppm.**	*ppm.**	*ppm.**
2	25	220	25	15	20	5
5	35	140	10	5	8.5	4.5

* Dry matter.

TABLE 2

Li content of leaves and fruit from Eureka lemon trees treated with LiCl

Li applied to soil	Li content		
	Mature leaves	Fruit peel	Fruit juice
ppm.	*ppm.**	*ppm.**	*ppm.**
1.0	30	—	—
2.0	13.5	—	—
4.0	50.0	2.0	0.5

* Dry matter.

recorded in tables 1 and 2. All the leaves showed symptoms of Li toxicity. Normally, Li is not detected in citrus plant ash.

The Li content of the young leaves of the sweet orange seedlings appears to be related to the amount of Li applied to the soil (table 1). Such a relationship does not exist with respect to the other plant parts, however, for in each set of samples analyzed, less Li was found in the plants growing in the soil receiving the largest amount of salt. The more rapid and greater defoliation of the plants treated with the larger amounts of Li, may partly account for this condition, since defoliation serves to eliminate Li from the plant. It should be particularly noted that Li accumulates in the leaves as they mature and that concentrations of similar magnitude do not develop in other vegetative parts of the plant. As a matter of fact, 85 per cent of the Li content of the entire plant is in the leaves.

The analytical data reported in table 2 likewise show no direct relation between the Li content of leaves of Eureka lemon and the amount of Li applied to the soil. Inasmuch as Li-toxicity symptoms were present in all leaf samples reported in table 2, such symptoms may be expected when the amount of Li found in the leaves is 13.5 ppm. or more.

An analysis of lemon fruit taken from trees receiving the highest amount of Li reveals that a small amount of Li moves into the peel, but that a barely detectable amount can be found in the juice. Although detailed information is lacking concerning the toxicity of Li to humans, areas of Li concentration in citrus fruit will be an important consideration in any study concerned with the use, by humans, of citrus products obtained from trees irrigated with lithium waters. Further studies are now being planned concerning the effect on animals of plants containing Li.

TABLE 3

Lithium content of leaves of citrus varieties

CITRUS VARIETY	Li IN LEAVES
	*ppm.**
Lemon	12
Grapefruit	14
Navel orange	23
Valencia orange	14

* Dry matter.

Field evidence of lithium toxicity

Citrus leaves showing typical Li-toxicity symptoms (fig. 1) were recognized for the first time in the field in a grove in Santa Barbara County, California. Several citrus varieties were affected; namely, lemons, grapefruit, and Valencia and navel oranges. Leaf samples showing typical Li-toxicity patterns were taken from the several varieties for analysis.

Analyses of these samples showed that the macroelements were present in relatively normal amounts, although Ca was high. Spectrographic examination revealed that the content of all microelements except Li was normal. This element was present in amounts comparable to that found in leaves showing artificially produced Li-toxicity symptoms. The Li contents are given in table 3.

A survey of the cultural history of the grove and its general vegetative condition from year to year revealed that the Li-toxicity patterns apparently became more prevalent when the amount of irrigation water applied to the plantings was increased. An increase in the amount of irrigation water used was made possible by the recent development of new wells on the property.

Samples of water were taken from four wells used as sources of irrigation water. Spectrographic examination revealed Li contents of 0.075, 0.045, 0.055, and 0.080 ppm. Microelements other than Li are present in the waters from these four wells in amounts within the range commonly encountered in good irrigation

waters. In view of the fact that a Li determination had not been, and cannot be, made on the water source used in previous years to irrigate this grove, it is impossible from the information at hand to determine whether the Li symtoms now in evidence on the citrus are the result of an accumulation of Li in the soil over a long period, suddenly accentuated by the increased use of water, or whether these symptoms have been produced in a relatively short period by the small amount of Li known to be present in the well waters recently developed. Experiments are now being conducted to determine the relative toxicity to citrus and other plants of small amounts of Li (0.05 to 0.10 ppm.) present in irrigation waters.

SUMMARY

Lithium-toxicity symptoms have been artificially produced in citrus, and the presence of lithium in the affected plants has been confirmed by a recently developed spectrochemical method.

On the basis of the artificially produced symptoms, lithium toxicity in citrus in the field has been recognized and confirmed by analysis.

Concentrations of lithium in various parts of citrus plants showing toxicity symptoms are reported.