ment caused by additions of toxic amounts of copper to the soil. This indicates that prominent iron chlorosis symptoms are not always associated with copper toxicity.

LITERATURE CITED

1. Ford, H. W. The influence of rootstock and tree age on the root distribution of citrus. Proc. Amer. Soc. Hort. Sci. (In Press) 1954.
2. Peech, M. and T. W. Young. Chemical studies on soils from Florida citrus groves. Fla. Agr. Exp. Sta. Bull. 448. 1948.
3. Reuther, W., F. E. Gardner, P. F. Smith and W. R. Roy. A progress report on phosphate fertilizer trials with oranges in Florida. Proc. Fla. State Hort. Soc. 61: 1-17. 1948.
4. Reuther, W., P. F. Smith and A. W. Specht. Accumulation of the major bases and heavy metals in Florida citrus soils in relation to phosphate fertilization. Soil Sci. 73: 375-381. 1952.
5. Reuther, W. and P. F. Smith. Iron Chlorosis in Florida citrus groves in relation to certain soil constituents. Proc. Fla. State Hort. Soc. 65: 62-69. 1952.
6. Reuther, W. and P. F. Smith. Effects of high copper content of sandy soil on growth of citrus seedlings. Soil Sci. 75: 219-224. 1953.
7. Smith, P. F. and A. W. Specht. Heavy metal nutrition in relation to iron chlorosis of citrus seedlings. Proc. Fla. State Hort. Soc. 65: 101-108. 1952.

MINERAL CONTENT OF ORANGES IN RELATION TO FRUIT AGE AND SOME FERTILIZATION PRACTICES

Paul F. Smith and Walter Reuther[1]
U. S. Horticultural Station
Orlando, Florida

There is little published information on the mineral content of Florida oranges. Certain minerals in oranges (unpublished) and in grapefruit (3) were determined by Fudge several years ago. In 1940 Bryan (1) published a rather complete mineral analysis of citrus fruit including both orange and grapefruit values from all available sources. Fertilization practices have changed considerably since that time and most groves today receive more nitrogen and less phosphorous and potassium, in relation to yield, than was customary a few years ago. The present report is concerned with the mineral content of whole fruit and of juice samples from two orange groves used for fertilizer studies the past few years. In one case, fruit samples at different stages of development were taken in order to obtain information as to when the minerals entered the fruit. The samples taken in relation to fertilization, however, were of mature fruit.

Experimental Procedure

For each sample 20 to 24 random-sized fruit were taken from several trees. None of the orchards received nutritional sprays during the particular year that samples were taken. Dust was removed by detergent washing, but this was not of any apparent benefit since the main contaminating chemical element removed is iron (10) and recontamination by this element resulted from the grinding process. The fruit was cut into several pieces, dried at 65° C. and ground through a large Wiley mill. After mixing, a portion of the ground material was saved for chemical analysis.

Some analyses for potassium in expressed juice were made by allowing fermentation to remove the sugar, acidifying with hydrochloric acid, boiling and filtering. Determinations were then made on the filtrate. The various elements were determined by the methods used for leaf analysis (7, 9). The micro-elements were determined partly by spectrographic methods and partly by standard chemical methods.

Results and Discussion

Mineral content in relation to age of fruit. In June 1948, 5 samples of 24 Valencia oranges were collected from plots of 12 trees each. These fruits were about 1 inch in diameter and less than 3 months of age. Five additional samplings from the same plots were made on the dates indicated in table 1. The trees were about 5 years of age and on Rough lemon stock. The soil was about average Lakeland fine sand. Conventional complete starter fertilizer mixtures had been used along with supplementary applications of nitrate of soda. The trees were vigorous and showed no signs of malnutrition. Detailed leaf analyses from these trees for this same period have been published (7, 9).

Table 1 shows the mean weights of the fruit and the amounts of the various elements in each fruit. The fruit of the last two collections were about commercial size 200 (2.75 to 3.00 inches in diameter). The fact that the crop was picked early in February indicates that the fruit was nearly mature at the last date of sampling. There were more potassium, nitrogen and calcium and less copper and

[1]/The authors wish to gratefully acknowledge the assistance of both A. W. Specht and G. Hrnciar in performing the laboratory work involved in this report.

Table 1. Mean weight and mineral content of Valencia orange samples taken at different stages of maturity.

Sampling date	Weight (grms.) Fresh	Weight (grms.) Dry	Milligrams per fruit N	P	K	Ca	Mg
6-2-48	46	9.5	102	12	115	72	17
7-16-48	82	13.9	143	16	159	92	24
9-2-48	136	20.9	192	24	240	88	30
10-21-48	172	25.4	193	27	260	89	35
12-8-48	201	28.3	226	28	295	83	35
1-24-49	191	30.9	269	29	342	87	36
L.S.D. @ 5%	4.1	0.7	23	2.0	28	11	1.1
L.S.D. @ 1%	5.6	0.9	33	2.7	38	15	1.5

Sampling date	Micrograms per fruit Cu	Zn	Mn	Fe	B	Al	Na
6-2-48	48	209	80	380	200	61	1002
7-16-48	77	181	103	550	283	61	1390
9-2-48	94	229	144	1290	409	96	1980
10-21-48	123	312	149	1850	478	161	2470
12-8-48	121	311	150	1500	515	212	3180
1-24-49	138	320	153	2165	551	360	3580
L.S.D. @ 5%	28	19	10	317	20	16	69
L.S.D. @ 1%	38	26	14	439	27	22	94

L.S.D. - the least difference between two means required for significance at the odds indicated.

manganese present than other elements determined.

The elements do not all enter the fruit continuously, although nitrogen, potassium, sodium, boron, aluminum, and possibly iron appeared to increase throughout the period of study. Nitrogen showed a non-accumulative period in September and October but accumulation in the fruit was resumed following the October fertilization. Calcium entered the fruit only during the first few months and was constant thereafter. Phosphorus, magnesium, copper, zinc, and manganese showed a continuous increase until October and little change thereafter.

Mineral composition of fruit in relation to fertilization. The total mineral elements comprise about 0.5 percent of the fresh weight of a citrus fruit. Table 2 gives the concentrations of the various elements determined in Valencia oranges sampled in March 1953. The trees had been differentially fertilized for six years as indicated. Large differences in the rates of nitrogen and potassium applied resulted in only small differences in the concentrations of the mineral elements in the fruit. A three-fold increase in the rate of applied nitrogen (ammonium nitrate) stimulated yield (4) but did not affect the amount of nitrogen in the fruit. It caused a slight decrease in phosphorus and potassium concentrations and a slight increase in calcium. An eleven-fold increase in applied potash resulted in about an 80-percent increase in the potassium concentration in the fruit, which is the largest effect found. Small reductions in the calcium and

Table 2. Mean fresh weight, percent dry matter and mineral composition of Valencia oranges grown with different levels of nitrogen and potassium.

Treatment	Fresh Wt. (grams)	Percent in dry whole fruit N	P	K	Ca	Mg
0.9# N per tree	142	0.72	0.121	1.08	0.172	0.096
1.8# N per tree	137	0.71	0.115	1.03	0.209	0.102
3.6# N per tree	136	0.73	0.106	0.99	0.215	0.104
0.3# K_2O per tree	117	0.72	0.115	0.70	0.232	0.096
1.8# K_2O per tree	140	0.71	0.112	1.13	0.193	0.103
3.6# K_2O per tree	158	0.73	0.114	1.28	0.170	0.100
L.S.D. @ 5%	4.7	N.S.	0.005	0.046	0.027	N.S.
L.S.D. @ 1%	6.3	--	0.007	0.063	0.036	--

Treatment	Percent D.M.	P.p.m. in dry whole fruit Cu	Zn	Mn	Fe	B
0.9# N per tree	17.6	4.2	13.5	7.2	29	14.7
1.8# N per tree	18.0	4.4	12.0	6.3	22	13.7
3.6# N per tree	18.2	4.1	15.2	8.0	30	15.8
0.3# K_2O per tree	19.6	4.5	13.0	7.0	28	15.8
1.8# K_2O per tree	17.4	3.6	14.1	6.4	23	14.7
3.6# K_2O per tree	16.8	4.6	13.6	7.8	30	13.7
L.S.D. @ 5%	0.6	N.S.	N.S.	N.S.	N.S.	1.17
L.S.D. @ 1%	0.8	--	--	--	--	1.58

boron concentrations accompanied the rather large increase in potassium.

Potassium increases the degree of hydration of the fruit (increases the water content slightly as shown in table 3). In terms of the dry weight, the high-potash fruit contains about 15 percent less dry matter than low-potash fruit (table 3). This is of about the same order of magnitude as the reduction in total soluble solids in the juice (4, 8) and may be the explanation for such a lowering. Potassium has similar effects on the vegetative parts of the tree (8). It is also known to affect the hydration of non-living colloidal systems in this same manner.

This effect of potassium on the water content of the fruit has considerable bearing on the amounts of the elements removed by cropping (table 3). Thus a ton of high-potash fruit removes about 15 percent less nitrogen than a ton of low-potash fruit even though both showed essentially the same percentage of nitrogen on a dry weight basis (table 2). The difference in the amount of potassium removed is only about 54 percent as compared with a difference of over 80 percent in the concentration values shown in table 2. Likewise, the differences in the amounts of calcium and boron removed in fruit from low- and high-potash trees are larger than would be calculated if the difference in dry-matter content were ignored (compare tables 2 and 3).

From consideration of the information just given, it is evident that the potassium content of the fruit is a rather weak criterion of the potassium requirements of the tree. The higher the rate of supply the greater the amount of potassium in the fruit. Fruit size, texture, and internal quality can all be correlated with leaf potassium (4, 8), which ap-

Table 3. Pounds of individual elements in 2,000 pounds of fresh fruit of Valencia and Pineapple oranges (on Rough lemon stock) with different fertilization and comparison with data of previous fruit analyses.

Treatment or Stock	N	P	K	Ca	Mg	Mn	Fe	Cu	Zn	B	% Water
						Valencia oranges					
0.9 lbs.N per tree	2.54	0.42	3.81	0.61	0.34	0.0026	0.010	0.0014	0.0048	0.0051	82.4
1.8 lbs.N per tree	2.55	0.41	3.95	0.74	0.36	0.0022	0.008	0.0014	0.0043	0.0049	82.0
3.6 lbs.N per tree	2.65	0.39	3.60	0.78	0.38	0.0029	0.011	0.0017	0.0055	0.0057	81.8
0.3 lbs.K_2O per tree	2.82	0.45	2.78	0.91	0.38	0.0027	0.011	0.0017	0.0051	0.0062	80.4
1.8 lbs.K_2O per tree	2.47	0.39	4.00	0.69	0.36	0.0022	0.008	0.0013	0.0049	0.0051	82.6
3.6 lbs.K_2O per tree	2.45	0.38	4.30	0.57	0.34	0.0026	0.010	0.0015	0.0046	0.0045	83.2
						Pineapple oranges					
0.0 lbs.P_2O_5 per tree	2.39	0.39	4.61	1.55	0.51	0.0028	0.011	0.0014	0.0031	0.0060	82.4
0.6 lbs.P_2O_5 per tree	2.36	0.38	4.67	1.53	0.49	0.0028	0.013	0.0015	0.0035	0.0056	82.6
1.8 lbs.P_2O_5 per tree	2.26	0.36	4.78	1.82	0.52	0.0026	0.010	0.0014	0.0037	0.0062	81.9
4.8 lbs.P_2O_5 per tree	2.46	0.39	5.04	1.81	0.48	0.0025	0.012	0.0014	0.0025	0.0065	82.0
						Marsh grapefruit 1/					
Rough lemon stock	2.12	0.27	4.04	0.82	0.22	0.0008	0.006	-	-	-	90.0
						Valencia oranges 2/					
Rough lemon stock	2.46	0.46	5.55	1.39	0.27	0.0009	0.004	0.0009	0.0027	-	83.9
Sour orange stock	3.14	0.50	5.09	1.40	0.24	0.0017	0.005	0.0014	0.0021	-	85.5
						Pineapple oranges 2/					
Rough lemon stock	3.04	0.53	5.62	1.20	0.33	0.0018	0.006	0.0009	0.0027	-	84.6
						World citrus 3/					
- - - - - - - - -	2.35	0.55	4.50	2.10	0.40	0.0015	0.0056	0.0008	0.0013	0.0052	-

1. Calculated from data by Fudge (3).
2. Calculated from unpublished data by Fudge (1940).
3. Calculated from averaged data by Chapman and Kelley (2); mostly oranges from 1892-1895.

pears to be a more reliable guide to fertilization since the leaf is more sensitive to potash changes and to possible disturbances in the pattern of other elements.

The data on phosphate fertilization in table 3 came from fruit form 10-year-old Pineapple orange trees which had been differentially fertilized with superphosphate since planting (5). Yield has not been affected so far. Only one sample of fruit per treatment was taken in this exploratory test. The slight differences found would not seem to indicate that analysis of all plots was justified at this time.

The differences in the mineral content between the Valencia and the Pineapple oranges are relatively small. The Valencias are considerably lower in calcium content, which is probably related to the difference in the soil properties of the light Lakeland fine sand in which they are growing and the Eustis fine sand in which the Pineapples are growing. Eustis soil has a higher clay content and greater exchange capacity which probably increase the efficiency with which base elements are used. This probably accounts for the higher potassium, calcium, and magnesium in the Pineapple oranges. The difference may, however, be varietal in nature.

Analyses for seven elements on whole grapefruit were made by Fudge (3) and his values for the Marsh variety are included in table 3 for comparison with present values along with some 1940 unpublished data for oranges by this same worker. Two samples of Valencias were averaged for each rootstock. The Pineapple orange values are from only one fruit sample. Also some averaged orange values from different parts of the world, published by Chapman and Kelley (2), are included in the table.

Considerable similarity may be seen in the data from all sources. Calcium, which occurs mainly in the peel, varies more than the other major elements. Present-day fruit would appear to contain somewhat more magnesium, manganese, and zinc than Fudge found 13 or 14 years ago, and possibly less potash, although the data are not adequate to make detailed comparisons.

Minerals in juice. The amounts of potassium and calcium were determined in the juice of fruit samples taken in March 1952 from the Valencia orange fertilizer plots. The calcium content of the juice was virtually constant regardless of the level of nitrogen or potassium. This is in agreement with the results of Fudge (3) in which the calcium content of the juice was unaltered following several years of heavy liming and the work of Roy (6) which showed that when potash was in short supply no other element took its place in the ash content of the juice. The juice simply showed a lowered ash percentage when potash was limited rather than being compensated for by extra calcium as in leaves. Roy found that potassium carbonate accounted for about 70 to 80 percent of the weight of the ash of Valencia orange juice.

Since potassium is the dominant mineral element in citrus juice, it was desired to see what effect the rates of nitrogen and potassium applied would have on it. Mean figures each from 12 replications are given in table 4. That is, individual determinations were made on random-sized fruit samples from the 108 field plots. Nitrogen rate had no appreciable effect on the amount of potassium in the juice. The rate of potash fertilization affected juice potash, but the effect is small in relation to the potash supplied (see table 2 for rates). The potassium grand means show that the difference between medium and high potassium is not significant. These results confirm those of Roy (6) that fruit composition is a rather insensitive indication of the fertilization requirements of a tree. Apparently the vegetative parts of the tree act as a mineral reservoir for the fruit to draw on and this tends to make the mineral composition of fruit rather uniform. In addition, the peel apparently acts as a buffer so that little variation occurs in the mineral composition of the juice. This is illustrated by the fact that whole fruit from high-potash trees shows about 80 percent more potassium than that from low-potash trees (table 2) while the difference in juice potassium is only about 30 percent.

Summary

The various mineral elements enter the maturing fruit at different rates. Calcium is taken up only in the first few months of fruit development. Potassium is the dominant element in the fruit and is taken in continuously as the fruit develops to maturity. This is true to a lesser extent for nitrogen, boron, sodium, aluminum, and possibly iron. Certain elements such as copper, zinc, manganese, phosphorus and magnesium appear to stop entering the fruit in the fall, several months before Valencia oranges reach maturity.

The mineral composition of fruit is rela-

Table 4. Effect of nitrogen and potassium fertilization on the potassium content (p.p.m.) of the juice of Valencia oranges.

Nitrogen rate	Potassium Rate: Low	Potassium Rate: Medium	Potassium Rate: High	N-Means
Low	1,200	1,485	1,550	1,412
Medium	1,165	1,500	1,450	1,372
High	1,160	1,400	1,600	1,387
K-means	1,175	1,462	1,533	

L.S.D. @ 5% - - 168 (between any two values)

N and K means L.S.D. @ 5% - - 97

tively unaffected by nitrogen or phosphorus fertilization although the potassium content of the fruit is increased by fertilization with that element. The potassium content of the peel is affected to a larger extent than that of the juice.

LITERATURE CITED

1. Bryan, O. C. The quality of citrus fruit as affected by cultural practice. Proc. Fla. State Hort. Soc. **53**: 98-100. 1940.

2. Chapman, H. D., and W. P. Kelley. The mineral nutrition of Citrus. Chap. VII. The Citrus Industry, Vol. 1, Univ. of Calif. Press. 1943.

3. Fudge, B. R. Relation of magnesium deficiency in grapefruit leaves to yield and chemical composition of fruit. Bull. 331. Fla. Agr. Exp. Sta. 1939.

4. Reuther, W., and P. F. Smith. Relation of fertilizer treatment to fruit quality of Valencia oranges. Proc. Fla. State Hort. Soc. **64**: 29-35. 1951.

5. Reuther, W., F. E. Gardner, P. F. Smith, and W. R. Roy. A progress report on phosphate fertilizer trials with oranges in Florida. Proc. Fla. State Hort. Soc. **61**: 43-60. 1948.

6. Roy, W. R. Effect of potassium deficiency and of potassium derived from different sources on the composition of the juice of Valencia oranges. Jour. Agr. Res. **70**: 143-169. 1945.

7. Smith, P. F., and W. Reuther. Seasonal changes in Valencia orange trees. I. Changes in leaf dry weight, ash, and macro-nutrient elements. Proc. Amer. Soc. Hort. Sci. **55**: 61-72. 1950.

8. Smith, P. F., W. Reuther, and G. K. Scudder, Jr. Effect of differential supplies of nitrogen, potassium and magnesium on growth and fruiting of young Valencia orange trees in sand culture. Proc. Amer. Soc. Hort. Sci. **61**: 38-48. 1953.

9. Smith, P. F., W. Reuther, and A. W. Specht. Seasonal changes in Valencia orange trees. II. Changes in micro-elements, sodium, and carbohydrates in leaves. Proc. Amer. Soc. Hort. Sci. **59**: 31-35. 1952.

10. Smith, P. F., W. Reuther, and A. W. Specht. Mineral composition of chlorotic orange leaves and some observations on the relation of sample preparation technique to the interpretation of results. Plant Physiol. **25**: 496-506. 1950.

MINERAL COMPOSITION OF VALENCIA ORANGE SEEDLINGS GROWN IN SOLUTION WITH VARYING AMOUNTS OF COPPER, ZINC, MANGANESE, AND IRON

Paul F. Smith and Alston W. Specht
U. S. Department of Agriculture
Orlando, Florida, and Beltsville, Maryland

Last year a report was made to this Society on various aspects of the toxic effect of heavy metals on citrus seedlings (3). The present paper gives some chemical analyses of the plants grown in the solutions described in table 1 of last year's report. Pictures of some of the plants involved and discussed here were published last year as figures 2 and 3 (3).

The plants were grown in nutrient solutions (pH near 4) by placing newly germinated seeds in corks in 1-quart fruit jars. Supplementary amounts of copper, zinc, and manganese were added to a complete nutrient solution. Chelated iron (K-Fe EDTA) was added to each of the treatments at a low rate (0.5 p.p.m. Fe) or a high rate (5 p.p.m. Fe). After 75 days in the greenhouse the plants were harvested, divided into tops and roots and analyzed for 12 elements. The tops were detergent-washed before drying, while the roots were only rinsed in distilled water. There were four replicates for each treatment, but these were combined in order to have sufficient dry matter to analyze. Even so, certain treatments were so toxic that the amount of growth made did not provide enough material for chemical analysis. This accounts for certain missing treatment numbers in the present tables as compared with those in table 1 of the previous report (3).

It is felt that the comparative values obtained will be helpful primarily in planning future studies of heavy-metal toxicities and in elucidating the behavior of the metals within the plant.

Results

The data in tables 1 and 2 are arranged in 3 groups on the basis of plant size. The mean dry weights indicate the degree of stunting. The plants which made normal growth tended to have lower nitrogen concentrations than those in the two stunted groups. The differences are not large and are possibly of no particular significance. Phosphorous concentrations tend to be similar regardless of treatment. The same is true of boron. Sodium and aluminum were not intentionally supplied, but apparently were present as contaminations in the salts used or came into solution from the glass culture jars. Neither shows any definite trend in relation to treatment.

The three main base elements, potassium, calcium, and magnesium, showed variations that are apparently related to the heavy metals. In the moderately stunted plants, potassium concentrations were unchanged but calcium and magnesium showed lowered concentrations in plant tops when compared with the