Effect of Nitrogen, Phosphorus, and Potassium Fertilizer on Fruit Yield and Composition of Tomato Leaves

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Two experiments were conducted to determine the optimum rates of application of nitrogen, phosphorus, and potassium fertilizer for tomatoes on Rockdale soil. Although both the growing season and soil are different from other tomato-producing areas in the country, the yields, fertilizer requirements, and composition of the tomato leaves were not greatly different.

THE ONLY PREVIOUS work on tomato fertilizers on the soil was by Fifield and Wolfe (*5*), whose study was limited to newly scarified soils. They concluded that phosphorus deficiency more than any other factor limited tomato production and recommended 280 to 300 pounds of phosphorus pentoxide, 80 pounds of nitrogen, and 120 pounds of potassium oxide per acre. On the sandy soils of South Florida 120 to 180 pounds of nitrogen, 150 to 240 pounds of phosphorus pentoxide, and 150 to 240 pounds of potassium oxide per acre are currently recommended (*12*).

No agreement was found in the literature on the most suitable portion of the plant to sample. To show the relationship between mineral uptake and the age of the plant, Hester (*8*) analyzed the whole plant and fruit. Such a procedure is not suitable on a field plot where yields are to be taken. Leaf samples are taken in many different ways. With young plants Carolus (*1*) made a composite sample of all leaves. Phillips (*11*), selected "middle" leaves for his analyses. Thomas and his associates (*13*, *14*) recommended the oldest leaf on the plant because old leaves showed the greatest differences in composition, but at times they were forced to use younger tissue because of the effects of disease (*14*, *15*).

Emmert (*3*, *4*) recommended sampling leaf petioles and quick testing. He believed the time required for total analysis could not be justified by the information obtained. More rapid methods of analysis have removed this objection.

In an exhaustive review of tissue analysis Goodall and Gregory (*7*) concluded that each type of sample has its advantage, and while old tissue might be useful in diagnosing some deficiency, tissue at an active growth stage would probably be much better for assessing the current nutrient supply. Although they did not question quick tests, they concluded that total analysis had fewer errors. They also recognized the importance of the number of plots sampled, available help, design of the experiment, and the control possible in conducting it.

An increase in the content of an element with no corresponding increase in yield is considered luxury consumption. Where the growth is determinate, this is an accurate concept, but where the growth is indeterminate and there is no toxicity, a higher than normal concentration of an element represents an active reserve. Carolus (*2*) reported luxury consumption of both nitrogen and potassium by tomatoes.

Inverse relationships frequently have been reported among several elements in tomatoes. In solution culture Phillips (*11*) found that withholding potassium increased the phosphorus, calcium, and magnesium content of the leaves. The difference in the concentrations of each of these elements suggests that the apparent increases in concentration resulted from reduced growth. Jannsen, Bartholomew, and Watts (*9*) found that a reduction of nitrogen, potassium, or phosphorus resulted in an increase in the others.

In the field an inverse relationship was noted (*10*) between the nitrogen applied and the phosphorus and calcium content of tomato leaves, but magnesium bore an inverse relationship to leaf nitrogen rather than to applied nitrogen.

Ion competition is frequently cited as a major cause for these inverse relationships. Van Itallie (*16*) postulated that the sum of the cations, when expressed on the basis of chemical equivalents, remains constant. However, Carolus (*2*) found that high concentrations of calcium in solution were necessary before increasing the potassium in solution reduced the calcium uptake. Where the calcium and potassium content of the nutrient solution was varied from 9 to 1 to 6 to 4 holding the total cation content constant, the potassium in the tomato leaves increased almost 100% but the calcium remained constant. Even with high calcium, where the calcium–potassium ratio varied from 71 to 1 to 13 to 1, the reduction in calcium uptake was not proportional to the increase in potassium.

Applications of nitrogen, phosphorus, or potassium do not always reduce other elements in the leaf. Carolus (*1*) reported that nitrogen from a mixed source, and phosphorus increased the magnesium content of field grown tomato plants.

Experimental Procedure

A Rockdale fine sand–limestone complex (*6*) was chosen for the experiments. It is without counterpart in the United States. Solution holes in the oölitic limestone, which forms the coastal ridge along extreme South Florida, are filled with sand and vary in depth from 2 to 24 inches. Between the holes the limestone appears above the surface. This land is prepared for planting by clearing and scarifying to crush the limestone and to prepare a loose layer 4 to 6 inches deep which can be cultivated.

Almost identical experiments were run in two successive years. Although the location was shifted, the land was under the same management and had essentially the same history. Each field had had two previous crops of tomatoes, but no other cultivated crop. After clearing, the fields were scarified and treated with a broadcast application of superphosphate. In each of the two previous seasons both fields had received in excess of 4000 pounds of 4–8–10 fertilizer. This history distinguished these soils from those investigated by Fifield and Wolfe (*5*). Each summer the fields had a cover crop of velvet beans.

Soil samples, taken before the test from the sites of the experiments, ranged in pH 7.4 to 7.8. The free calcium carbonate content of the soil averaged 15%. This would have been much higher if particles larger than 10 mesh had not been removed by screening. According to the soil test using acid ammonium acetate, the levels of phosphorus, potassium, and magnesium were all high—62 to 160 pounds of phosphorus pentoxide, 150 to 350 pounds of potassium oxide, and 570 to 870 pounds of magnesium oxide per acre. By these tests there were no differences between the two locations. The nitrate nitrogen ranged from 18 to 24 pounds per acre in samples taken the first year and from 32 to 64 pounds the second. The extractable calcium was in excess of 25,000 pounds per acre at both locations.

The Homestead tomato variety was chosen, because it has resistance to fusarium wilt and can be planted on the same land in successive years. Plants were started in a bed and transplanted to the field. Both crops were grown during the winter. The first was planted September 24, 1954, with the final picking on January 18, 1955. The second was planted on November 4, 1955, and picked for the last time on March 25, 1956. Each crop was picked five times.

Nitrogen, phosphorus, and potassium were each applied at three levels, 0, 150, and 300 pounds of nitogen, phosphorus pentoxide, and potassium oxide per acre. These three levels were combined in a complete factorial design to give a total of 27 treatments. Each treatment was replicated three times in randomized blocks.

Sulfate of ammonia was the source of nitrogen and triple superphosphate the phosphorus source. At planting of the first crop potassium sulfate was used, but after that the chloride was used exclusively.

Fertilizer was applied three times. One sixth was applied in bands at planting time. At the first side dressing about 4 weeks after transplanting, one third of the total was applied and about 3 weeks later when the first hand was setting, the remaining half was applied. Forty pounds of borax per acre was applied the first year and 50 the second. Manganese was supplied by maneb fungicide, applied approximately 25 times each season.

Cultivation and irrigation were done according to the farm practice of the cooperator. In both seasons a top-dressing of 40 pounds of urea per acre was applied by airplane after each of the first four pickings. In the second year a severe frost, 10 days before the first picking, killed back the vines about a foot. It was followed by a long period of good weather and the plants made very rapid recovery.

Leaf samples were taken once the first year and twice the second. To assure a sample of healthy leaves the third or fourth leaf from the top of the plant was taken. By sampling new growth, excessive spray residue and other contamination of the sample were avoided. Leaf samples were taken after the first picking the first year to avoid spread of tobacco mosaic virus prior to harvest. The second year the first leaf samples were taken 10 days after the final side dressing, when the second hand of fruit was setting. To prevent transmitting virus, the knives and samplers' hands were dipped in 70% alcohol after each leaf was cut. The second sample was delayed until after the second picking, while the plants recovered from the frost. Two leaves were taken from each of the 20 plants on each plot for each sample.

Table I. Tomato Yields and Treatments in an N × P × K Factorial Fertilizer Experiment on Rockdale Fine Sand

Treatment No.	Treatment, Lb./Acre			Yields, Lb./0.01-Acre Plot			
				1954–1955		1955–1956	
	N	P_2O_5	K_2O	Marketable	Total	Marketable	Total
1	0	0	0	154.4	214.6	159.6	214.1
2	0	150	0	157.4	232.2	148.4	189.7
3	0	300	0	163.0	217.4	130.8	175.1
4	0	0	150	201.4	266.1	211.3	255.9
5	0	150	150	210.4	270.1	209.8	258.6
6	0	300	150	232.7	291.2	196.5	242.6
7	0	0	300	189.4	245.5	196.8	247.9
8	0	150	300	226.3	285.1	260.1	318.1
9	0	300	300	235.8	292.2	267.5	328.0
10	150	0	0	233.5	303.3	203.2	280.9
11	150	150	0	203.4	252.8	203.9	260.4
12	150	300	0	240.2	307.3	212.6	275.6
13	150	0	150	280.1	325.2	282.7	341.5
14	150	150	150	320.8	373.7	337.7	391.0
15	150	300	150	342.8	407.6	315.1	368.1
16	150	0	300	294.6	340.8	251.6	299.3
17	150	150	300	316.7	360.6	286.0	341.1
18	150	300	300	332.4	384.4	343.8	400.6
19	300	0	0	199.9	254.6	164.5	206.7
20	300	150	0	170.8	214.1	173.8	220.0
21	300	300	0	160.2	211.8	157.7	202.5
22	300	0	150	269.4	302.8	237.6	288.4
23	300	150	150	276.5	330.7	247.5	297.0
24	300	300	150	247.8	280.9	268.9	325.0
25	300	0	300	278.1	316.5	238.1	284.5
26	300	150	300	295.5	334.2	236.2	281.1
27	300	300	300	247.8	284.4	267.6	315.7

The leaf samples were dried in a forced draft oven at 85° C. and ground to pass a 0.5-mm. sieve in a Wiley mill. Nitrogen was determined by the Kjeldahl method. Phosphorus was determined colorimetrically by the phosphomolybdate method. Potassium, calcium, and magnesium were determined using a Beckman DU spectrophotometer with a flame photometer attachment. All analyses were in duplicate.

The results were examined by an analysis of variance. Where the sums of squares were sufficiently large, separate degrees of freedom were calculated for fit to a straight line and curvature.

Results and Discussion

Yields. Tomato yields were increased both years by nitrogen, phosphorus, and potassium. Table I shows the fertilizer applied and the marketable and total fruit yield for both years. In both years there was a very highly significant yield increase from the application of nitrogen and potassium. One hundred and fifty pounds of nitrogen gave a larger increase in yield over the check than 300 pounds of nitrogen. The curvature was very highly significant. The yields at three potassium levels also showed very high significant curvature, but no yield depression.

A highly significant nitrogen-phosphorus interaction in the first experiment obscured the increase from phosphorus. However, with 0 and 150 pounds of nitrogen, phosphorus gave a highly significant linear increase in yield. In the second year there was no significant interaction between nitrogen and phosphorus. The application of phosphorus gave a significant linear increase in total yield and a highly significant linear increase in yield of marketable fruit. The phosphorus response was overshadowed somewhat by a very highly significant linear interaction of phosphorus and potassium. At zero potassium the addition of phosphorus made little difference in the yields, but with 300 pounds of potassium oxide per acre, 150 pounds of phosphorus pentoxide per acre increased the yield of marketable fruit to 32 pounds per plot, and 300 pounds of phosphorus pentoxide increased the yield to 64 pounds per plot. Thus, although the interaction was different in each year, the yield increase resulting from adding phosphorus to the fertilizer was highly satisfactory, wherever near optimum amounts of nitrogen and potassium were supplied. Further, there is every indication in the data that even larger increases in yield could have been obtained with more phosphorus.

The depressing effect of the high nitrogen on yield was not the result of excessive vegetative vigor, because the vines were not as thrifty with 300 pounds of nitrogen per acre as with 150 pounds. Although the late top-dress-

Table II. Composition of Tomato Leaves from an N × P × K Factorial Fertilizer Experiment on Rockdale Fine Sand

Treatment No.	Composition, Dry Weight, %														
	1954–1955					1955–1956									
						First Sampling					Second Sampling				
	N	P	K	Ca	Mg	N	P	K	Ca	Mg	N	P	K	Ca	Mg
1	3.23	0.23	2.0	3.4	0.45	4.09	0.30	2.8	3.8	0.45	4.33	0.38	3.2	3.0	0.49
2	3.27	0.23	1.9	3.5	0.43	4.20	0.38	2.6	4.1	0.43	4.43	0.46	3.0	3.5	0.52
3	3.48	0.24	1.8	3.7	0.50	4.30	0.40	2.4	3.6	0.42	4.59	0.45	3.3	3.5	0.55
4	3.47	0.22	2.3	3.5	0.50	4.06	0.27	3.4	3.4	0.43	4.62	0.39	4.1	3.0	0.51
5	3.71	0.24	2.5	3.1	0.48	4.10	0.33	3.3	3.5	0.42	4.46	0.41	4.1	3.6	0.51
6	3.66	0.25	2.4	2.9	0.46	4.14	0.39	3.2	3.4	0.43	4.46	0.44	4.3	3.2	0.50
7	3.37	0.22	2.6	2.8	0.41	4.02	0.26	3.2	3.4	0.44	4.26	0.37	4.1	3.4	0.48
8	3.29	0.23	2.3	3.0	0.43	4.06	0.28	3.4	3.1	0.40	4.58	0.41	4.4	3.4	0.52
9	3.56	0.25	2.5	2.8	0.42	3.85	0.30	3.7	3.2	0.43	4.31	0.39	4.0	3.2	0.51
10	4.41	0.23	1.2	3.4	0.59	4.29	0.23	2.2	3.9	0.52	4.83	0.35	2.7	2.6	0.47
11	4.50	0.24	1.3	3.5	0.48	4.49	0.26	2.1	3.7	0.51	4.71	0.37	2.6	3.0	0.50
12	4.20	0.22	1.2	3.3	0.46	4.51	0.29	2.3	3.7	0.53	4.95	0.39	2.9	2.6	0.48
13	4.18	0.21	2.4	3.0	0.48	4.50	0.25	2.4	3.3	0.46	4.75	0.35	3.9	2.6	0.48
14	4.26	0.24	2.2	3.1	0.55	4.50	0.28	2.9	3.4	0.48	4.64	0.36	3.9	3.2	0.48
15	4.21	0.24	1.9	3.0	0.49	4.46	0.29	3.0	3.5	0.47	4.65	0.40	3.7	3.0	0.47
16	4.28	0.22	2.4	3.1	0.51	4.46	0.24	3.2	3.3	0.51	4.89	0.37	4.2	2.5	0.47
17	4.43	0.25	2.6	2.8	0.56	4.43	0.25	3.1	3.4	0.49	4.87	0.38	3.7	2.8	0.46
18	4.44	0.26	2.6	2.9	0.56	4.35	0.27	3.1	3.7	0.48	4.71	0.42	4.2	2.8	0.47
19	4.04	0.21	1.4	3.5	0.51	4.46	0.25	2.0	3.9	0.54	4.74	0.34	2.5	3.2	0.55
20	4.28	0.23	1.2	3.5	0.53	4.62	0.27	2.0	3.5	0.53	4.95	0.35	2.8	2.5	0.47
21	4.30	0.22	1.2	3.7	0.58	4.34	0.25	2.3	3.7	0.55	4.86	0.37	2.5	3.2	0.54
22	4.46	0.23	2.3	2.8	0.53	4.34	0.24	2.6	3.4	0.50	4.68	0.33	3.7	2.8	0.49
23	4.43	0.25	1.8	2.8	0.51	4.51	0.26	3.0	3.5	0.53	4.65	0.35	3.8	3.0	0.49
24	4.52	0.25	2.0	3.4	0.66	4.56	0.27	2.6	3.4	0.50	4.69	0.36	3.6	3.5	0.53
25	4.31	0.22	2.6	2.7	0.61	4.39	0.24	2.9	3.3	0.49	4.79	0.35	4.2	2.8	0.48
26	4.47	0.24	2.5	2.8	0.65	4.41	0.24	3.0	3.2	0.50	4.75	0.37	4.2	3.0	0.50
27	4.48	0.26	2.4	2.9	0.68	4.53	0.26	3.1	3.2	0.52	4.81	0.37	4.0	3.0	0.50

ing of urea may have had some influence on the total yield, there is no indication that the yield of fruit from the no nitrogen plots was relatively better toward the end of the season when compared to the plots receiving 150 pounds of nitrogen per acre.

Composition. The composition of the samples taken in the 1954–55 and 1955–56 seasons is shown in Table II, calculated on the basis of the oven-dry weight. Each value is the average of duplicate analyses from each of the three replicates.

In both years there was a relatively large increase in nitrogen in the tomato leaves where 150 pounds of nitrogen per acre was applied, compared to the no nitrogen treatments, but only a slight increase where the applied nitrogen was raised from 150 to 300 pounds. This reduction in uptake was statistically significant. Only in the first sampling the second year did another element significantly affect the leaf nitrogen. Then the application of potassium reduced the nitrogen. Because potassium increased yields to such a significant degree, this was probably a dilution of the nitrogen rather than exclusion of it. The second sampling in the second year was considerably higher in nitrogen than that from the first sampling, the combined effect of the urea top-dressing and the new growth which came out after the tops had been frozen. The difference in leaf nitrogen between the no nitrogen and the 150-pound treatment was the same at both samplings.

Where phosphorus was applied, there was a small, but very highly significant increase in the phosphorus content of leaves in all three samplings—linear in each instance. This indicates that the phosphorus supply was near the critical lower limit for tomato production and is consistent with the yield results. The phosphorus content of leaf samples taken after the freeze the second year was considerably higher than in the first sampling, as in other experiments in South Florida. After plants have developed a good root system and the amount of top is sharply reduced, the root is able to supply sufficient phosphorus to the smaller top.

In 1954–55 the phosphorus content of the tomato leaves was not significantly affected by the addition of nitrogen, but in the 1955–56 experiment, particularly with 300 pounds of phosphorus pentoxide per acre, addition of nitrogen sharply reduced the phosphorus content. There was a statistically significant, but very small, increase in the phosphorus content as a result of adding potassium to the fertilizer the first year The second year addition of potassium to the fertilizer significantly reduced the phosphorus content of the tomato leaves, but all this change took place at zero nitrogen, which severely limited the yields. The leaves showed a build-up of phosphorus only on treatments which produced unsatisfactory growth. In spite of the history of heavy use of phosphorus and high "available" soil phosphorus, more phosphorus could have been used profitably.

As with the other two major elements, the potassium applied increased the amount of potassium in the leaves. The first 150 pounds of potassium oxide per acre gave a significantly greater increase in leaf potassium than the second 150 pounds. At the lower levels of potassium, nitrogen significantly reduced the leaf potassium, but where 300 pounds of potassium oxide were applied the potassium content of the tomato leaves was not affected. The nitrogen-potassium interaction was significant in both years.

The yield response to potassium followed a curve similar to that of the potassium content of the leaves. There was no luxury consumption of potassium except where nitrogen was deficient. The phosphorus-potassium interaction in the yield data had no counterpart in the potassium contents of the tomato leaves.

In the 1954–55 samples and in the first samples taken in 1955–56 the calcium content of the tomato leaves was decreased as the rate of application of potassium increased. The reduction in the calcium content was not chemically equivalent to the increase in the potassium or even proportional to it. Although the increase in the potassium content of the leaves showed significant curvature in all cases, the decline in the calcium content was almost wholly linear. In the final samples there was no observable relationship between the rate of application of potassium to the soil and the calcium of the plant, although the potassium content of the plant increased.

Potassium did not decrease the magnesium content of the leaves. In the first season's samples and in the first samples taken, the second year, the magnesium content increased significantly as the rate of application of nitrogen increased. In the second samples, the second year, the magnesium followed a pattern which was the inverse of the yield pattern, probably determined by the amount removed by the fruit, which in turn was related to the nitrogen application. It seems probable that the increase in the magnesium from the first to the second sampling from the low nitrogen plots resulted from the top-dressing of urea. There was no evidence of a constant cation sum.

Both too little and too much nitrogen obscured the value of phosphorus and potassium. The yield reduction resulting from the application of 300 pounds of nitrogen per acre can be only the result of direct injury. The plants were not extremely vegetative, there was no large accumulation of nitrogen in the leaves, and there was no significant interference with the absorption of any element determined.

The results in the last sampling show that the pattern of the yields was set during the early part of the growth of the plants, whereas the composition of the leaves reflected the available nutrient supply at the time of sampling. The reaction of the fertilizer salts with the soil played a very significant role, particularly in the case of magnesium. Soil tests using acid ammonium acetate as extractant did not provide a useful index of the available nutrients in this Rockdale soil.

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Literature Cited

(1) Carolus, R. L., *Proc. Am. Soc. Hort. Sci.* **54**, 281–5 (1949).
(2) Carolus, R. L., Virginia Truck Expt. Sta., Norfolk, Bull. **81** (1933).
(3) Emmert, E. M., Kentucky Agr. Expt. Sta., Univ. Kentucky Bull. **430** (1942).
(4) Emmert, E. M., *Proc. Am. Soc. Hort. Sci.* **54**, 291–8 (1948).
(5) Fifield, W. M., Wolfe, H. S., Florida, Univ., Agr. Expt. Sta., Sub-Trop. Expt. Sta. Rept. 3 (1937).
(6) Gallatin, M. H., *et al.*, Soil Survey of Dade County, Fla., Ser. **1947**, No. 4, 1958.
(7) Goodall, D. W., Gregory, F. G., Imp. Bur. Hort. Plantation Crops, East Malling, Kent, Tech. Commun. **17** (1947).
(8) Hester, J. B., *Proc. Am. Soc. Hort. Sci.* **36,** 720–2 (1939).
(9) Jannsen, G., Bartholomew, R. P., Watts, V. M., Arkansas Univ., Agr. Expt. Sta. Bull. **310** (1934).
(10) Malcolm, J. L., *Soil Sci. Soc. Florida Proc.* **15,** 91–100 (1955).
(11) Phillips, J. G., *et al.*, New Hampshire, Univ., Durham, Agr. Expt. Sta., Tech. Bull. **59** (1934).
(12) Spencer, E. L., *et al.*, Florida, Univ., Agr. Expt. Sta., Gainesville, Bull. **563** (1955).
(13) Thomas, W., *Soil. Sci.* **59,** 353–74 (1945).
(14) Thomas, W., Mack, W. B., *Proc. Am. Soc. Hort. Sci.* **39,** 319–28 (1941).
(15) Thomas, W., Mack, W. B., Cotton, R. H., *Ibid.*, **42,** 525–44 (1943).
(16) Van Itallie, T. B., *Soil Sci.* **46,** 175–86 (1938).

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PLANT ANALYSES

Rapid and Accurate Automatic Titration Method for Determination of Calcium and Magnesium in Plant Material with EDTA Titrant

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A method is described for the determination of calcium and magnesium in plant materials which combines rapid precipitation and extraction techniques to eliminate interferences and automatic titrations to eliminate subjective evaluation of the end points. The method is more rapid than conventional methods and as accurate. After wet digestion of the plant samples, the total time for determination of both calcium and magnesium is about 15 minutes, including separations and titrations.

A NUMBER OF METHODS have been proposed for the determination of calcium and magnesium in plant material using disodium (ethylenedinitrilo)-tetraacetate (EDTA) as titrant and metal indicators for visual end point detection. One difficulty encountered in these methods is the presence of iron, manganese, aluminum, copper, and phosphate in amounts sufficient to interfere with both titrations. Many procedures for the elimination of these interfering ions have been presented (*1, 2, 4, 7, 8, 10, 11*). Investigation showed that the removal of phosphate by precipitation with zirconium nitrate (*8*) and extraction of the heavy metals with carbon tetrachloride as the diethyldithiocarbamate complexes (*4*), provided a simple, rapid, and good separation of interferences.

Another difficulty often encountered with EDTA titrations is the visual end point detection. This problem can be eliminated for calcium and magnesium titrations by using the Sargent Spectro-Electro titrator, whereby titrations can be automatically and precisely terminated at their end points (*6*). Calcium and magnesium in plant material are determined by automatic titrations after the interferences are removed by the relatively rapid precipitation and extraction techniques. An integrated, rapid, accurate, and easy to follow procedure incorporating the separation and automatic titration techniques is described.

The automatic titrations are both completed in a few minutes, and the results check closely with the values assigned to four standard fruit tree leaf samples: apple, cherry, citrus, and peach (*5*).

Apparatus and Reagents

Connect the Sargent-Malmstadt Spectro-Electro automatic titrator (E. H. Sargent Co., Chicago, Ill.) (*9*) and Teflon stopcock burets as previously reported (*6*).

STANDARD CALCIUM SOLUTION. Dry primary standard calcium carbonate (Mallinckrodt Chemical Co.) in an oven