Proc. Fla. State Hort. Soc. 90:272-273. 1977.

RESIDUES OF NAPHTHALENEACETIC ACID (NAA) IN 'BEARSS' LEMONS AND THEIR PROCESSED PRODUCTS FOLLOWING SPRAYING OF TREE TRUNKS[1]

H. Anson Moye and A. E. Willson[2]
Pesticide Research Laboratory,
Food Science and Human Nutrition Department,
University of Florida, Gainesville, Florida 32611

Additional index words. sprouting, high performance liquid chromatography.

Abstract. **Naphthaleneacetic acid (NAA) as the ethyl ester, has been found to be effective as a spray to lemon tree trunks after pruning. Fruit harvested 5 and 7 months after the initiation of a six month, monthly spraying program, at the 1% and 2% levels showed negligible residues of NAA (less than 0.004 ppm). Of processed samples analyzed, peel oil (from nine month fruit) contained residues of 4.0 ppm (2% spray), whereas others showed much less or negligible residues.**

The effectiveness of spraying solutions of naphthaleneacetic acid (NAA) on the pruned trunks of lemon trees to prevent sprouting, and thereby an overabundance of smaller mature fruit, has been demonstrated (2, 4). Lundberg and Smith utilized a 1% and 2% asphalt based solution of NAA-ethyl ester (Amchem 82-A112) to control such sprouting (2).

This report presents the residue data from such an experiment and includes determinations on the processed fractions obtainable after fresh fruit has been subjected to a pilot plant feed mill at the Lake Alfred Research and Education Center, Lake Alfred, Florida.

Materials and Methods

A 'Bearss' lemon grove at Indiantown, Florida in healthy horticultural condition was used for this study. The 20 ft spacing of the trees permitted a completely randomized spraying with 35 control trees (no spray), 38 trees receiving a 1% spray and 41 trees receiving a 2% spray. All trees were pruned from ground level to 3 to 4 feet on the scaffold branches (February 15, 1973) and then treated with the appropriate spray. Spraying was accomplished on a monthly schedule, on the 15th of each month, from February through July inclusively. On July 13th, 17 field boxes representative of each treatment were randomly harvested from all trees. Two cases were held at −8°C for residue analyses on the fresh fruit; 15 field boxes were transported to the A.R.E.C. Lake Alfred for processing through the pilot plant feed mill (Group I). On September 14th a similar harvest was made and likewise handled (Group II).

Residues of naphthaleneacetic acid were examined on unwashed peel, unwashed pulp, (fresh fruit sent directly to Gainesville); washed peel, washed pulp, chopped peel, dried peel, peel oil, press liquor, fruit juice, emulsion water, peel frit, finisher pulp, pre-water rinse, after water rinse, dried pulp and molasses.

Since the naphthaleneacetic acid was in the form of an ethyl ester, and information regarding its hydrolysis in and on plant constituents is lacking, an analytical procedure was devised which firstly hydrolyzes all of the ester to free NAA, except for molasses which required that each sample be split into 2 parts and 1 part extracted for the ester and the other extracted for the acid, the sum of which provides total NAA.

Coggins *et. al.* (1) have shown that free NAA fluoresces intensely and used this as a basis for residue analysis. By coupling reversed phase liquid chromatography to the fluorometric measurement we were able to simplify the clean-up considerably and lower the sensitivity of the method approximately ten-fold (3). Samples were analyzed as duplicates with 2 chromatographic injections made for each sample.

Results and Discussion

Table 1 illustrates the recoveries which can be realized for most of those fractions for which residues were found. Free acid in molasses gave the lowest recoveries obtained, however, other fractions were much higher.

Table 1. Recoveries (%) of NAA from lemon fractions.[z,y]

Sample	ppm added					
	0.0	0.008	0.01	0.05	0.1	0.15
Washed peel	0	—	82	69	79	74
Washed pulp	0	—	73	61	78	67
Peel oil	0	—	—	60	68	61
Press liquor	0	—	75	67	89	74
Molasses (ethyle ester)	0	81	100	87	97	—
Molasses (free acid)	0	0	0	75	53	55

[z]Average of three separate determinations.
[y]Recoveries were not attempted for those indicated by a dash.

Table 2 summarizes the results of residues found in the Group I samples, harvested immediately after the six month spraying program. As can be seen, residues were found only in the dried peel and the peel oil, with dried peel only slightly higher than the 0.004 ppm limit of detection of the method. Slightly higher residues were found in the peel oil, but still at an insignificant level.

Table 2. Residues (ppm) NAA in Bearss lemons, Group I.[z,y,x]

Sample	1% spray	2% spray
Dried peel	0.005	0.006
Peel oil	0.019	0.011

[z]No residues were found at the limit of detection (0.004 ppm) in any other fraction.
[y]Average of two separate field replications.
[x]Harvested July 13, 1973.

Table 3 summarizes the results found in the Group II samples. Residues were found in all processed samples except the pre-water rinse and after water rinse. No residues were found in the fresh fruit. Highest residues were in the peel oil, an average of 0.28 ppm for the 1% spray and an average of 3.0 ppm for the 2% spray. Since the procedure for the molasses was able to distinguish free acid from ethyl ester it is interesting to note that only 10% of total residue appeared in the ester form, indicating that hydrolysis by various modes is operative.

[1]Florida Agricultural Experiment Station Journal Series No. 853.
[2]Deceased; Food Division, Coca Cola Company, Inc., P. O. Box 3189, Orlando, Florida 32802.

Table 3. Residues (ppm) NAA in Bearss lemons, Group II.[x,y,z]

Sample	1% spray	2% spray
Chopped peel	0.012	0.12
Dried peel	0.010	0.07
Peel oil	0.28	3.0
Press liquor	0.008	0.027
Fruit juice	<0.004	0.005
Emulsion water	0.009	0.047
Peel frit	0.028	0.29
Finisher pulp	<0.004	0.009
Molasses (ester)	0.005	0.013
Molasses (free acid)	0.028	0.158

[x]No residues were found at the limit of detection (0.004 ppm) in any other fraction.
[y]Average of two separate field replications.
[z]Harvested September 14, 1973.

Literature Cited

1. Coggins, C. W., V. A. Jolliffe, W. W. Shindy and J. C. F. Knapp. 1972. Naphthaleneacetic acid disappearance and residue studies in citrus. *Journal of Agricultural and Food Chemistry*. 20:76-79.
2. Lundberg, E. C. and T. S. Smith. 1974. A possible sprout inhibitor for Florida citrus. *Proc. Fla. State Hort. Soc.* 87:20-23.
3. Moye, H. A. and T. A. Wheaton. Residues of naphthaleneacetic acid in citrus. in preparation for *Journal of Agricultural and Food Chemistry*.
4. Phillips, R. L. and D. P. H. Tucker. 1974. Chemical inhibition of sprouting of pruned lemon trees. *Hortscience*. 9:199-200.

Proc. Fla. State Hort. Soc. 90:273-275. 1977.

RESPONSE OF PINEAPPLE (ANANAS COMOSUS MERR.) GROWING IN SAVANNA SOIL OF BRASILIA TO LEVELS OF N, P AND K

E. A. VILELA-MORALES[1] AND R. A. COSTA
Fundação Zoobotânica do D.F., Brazil

O. ANDERSEN
Universidade Federal de Vicosa, Brazil

Additional index words. Cerrado and fertilization.

Abstract. **'Pernambuco' pineapple was grown under 3 levels of NPK on a tropical savanna soil of Brazil. The fertilizers used were ammonium sulphate, superphosphate regular, and potassium sulphate with the following levels of g/plant, N: 3.0, 6.0, and 9.0; P_2O5: 0.9, 1.8, and 2.7; and K_2O: 5.0, 10.0, and 15.0.**

Observations were made on weight and number of fruits, slips and suckers produced, plant height, and production of precocious fruits called "Maritacas". In general, fruit weight and number, plant height, and number of slips and suckers produced were increased with increased levels of N and K, but there was no effect of the 3 levels of P. Increased soil levels of N, P, and K produced correspondingly increased leaf levels. Production of "Maritacas" was not affected by NPK levels.

Research in Brazil recommends the pineapple as a crop for cultivation on the Brazilian Savanna, called "Cerrado" (14). This crop has an adequate foliar structure to tolerate dry seasons (11), does not demand high soil levels of P (8), and the soil pH for best fruit quality is around 4.5-6.0 (11, 13). Thus the characteristics of this crop and region are well matched (14).

It has been suggested that N is the main nutrient that controls the rate of growth (5, 6, 7, 8), thereby improving the yield and the fruit quality, particularly if the plant has an adequate level by bloom time (12). Responses to P are not reported to be significant (8), but this element is needed most during the flowering stage (13). K has been suggested as the second most important nutrient (6, 7, 8), and reportedly needs to be available immediately after planting, and during fruit ripening (13).

Materials and Methods

This work was established on a dark-red latosol of the savanna region of Brasilia. The soil analysis was N: 0.13%; P: 2ppm; K: 52ppm; Al+++: 0.3 eq. mg/100cc soil; and pH: 5.24. The experimental design was a fractorial 3^3 with 3 replications in a randomized block design. All the treatments were established on the double-row planting system, with a density of 35,000 plants/ha (1). There were 40 plants per plot, from which 16 plants were measured. The cultivar was 'Pernambuco'. The slips selected were 35-45 cm long (2).

The nutrient levels were, N: 3.0, 6.0, and 9.0; P_2O5: 0.9, 1.8, and 2.7; and K_2O: 5.0, 10.0, and 15.0 g/plant. As sources of N, P, and K, ammonium sulphate, superphosphate regular, and potassium sulphate were used (12, 13). All the P was applied at planting time, but N and K were applied 3 times: 2, 9, and 12 months after planting.

Before being set the planting material was "cured", in order to avoid the disease caused by *Fusarium moniliforme* Sheld. var. *subglutinans* Wr. & Rg. and treated with insecticides to control pests such as *Dysmicoccus brevipes* Geyer (3). Fruit weight and number, slips and suckers produced, plant height, and production of the undesirable precocious fruits called "Maritacas", were observed.

Results and Discussion

Higher levels of N, applied along with K or P, resulted in higher yields, as indicated by an increase in fruit number and weight as well as an increase in height of the plants and number of slips and suckers produced (Table 1). However, the foliar levels of this nutrient observable by harvest time showed that it was much depleted. Also, mean fruit weight was below the standard weight for the cultivar. The positive and highly significant linear response (Nl) to the N levels may lead one to conclude either that all N levels selected were below the optimum, that there was a deficient

[1]Present address: c/o Fruit Crops Dept., 1172 McCarty Hall, University of Florida, Gainesville, FL 32611.