

## EFFECT OF ETHYLENE DEGREENING ON DECAY OF FLORIDA CITRUS FRUIT

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### ABSTRACT

Decay of 'Hamlin' and 'Valencia' oranges and 'Temples' was increased significantly by degreening with ethylene. Degreening conditions used were those recommended by the Agricultural Research and Education Center at Lake Alfred except for the higher ethylene concentrations tested. Comparisons were made between nondegreened fruit and fruit degreened with ethylene at concentrations of approximately 5 ppm, 50 ppm, and 120 ppm. There was a direct relationship between ethylene concentration and decay. Degreening increased stem-end rot but not green mold. A Unico Model 400<sup>1</sup> Precision Gas Detector was used to measure ethylene concentrations.

### INTRODUCTION AND LITERATURE REVIEW

Early varieties of Florida citrus fruit are usually mature and palatable before the green peel color is replaced by the characteristic color of these cultivars. For citrus fruit to be acceptable on the retail market, the green color must be removed. Degreening with ethylene, sometimes referred to as "coloring," destroys the chlorophyll in the peel with little effect on the edible portion of the fruit.

Before green-colored citrus fruit can be harvested for sale as fresh fruit, the internal quality must pass minimum standards for the variety (7). Citrus fruit are degreened in rooms where ethylene concentration, air movement, humidity, and temperature can be controlled (3, 6).

This study was made to determine the effect of degreening citrus fruit with known concentrations of ethylene and to compare the amount and types of decay that developed at the different ethylene concentrations used.

Degreening of citrus fruit, prior to 1960, has been reviewed by Grierson and Newhall (3). Since that time, a more accurate method of measuring the concentration of ethylene in degreening rooms

has been developed. Until recently, the ethylene concentration was estimated by the volume of this gas metered into the degreening room. The actual concentration of ethylene in a degreening room depends on several factors, particularly the tightness of the room and how much fresh air is introduced during degreening.

Jahn et al. (5) found 5 to 10 ppm of ethylene to be adequate to degreen Florida citrus fruit. Hall et al. (4) reported that in Australia ethylene degreened citrus fruit without affecting internal maturity. Burg and Burg (1, 2) have reported on the action of ethylene in ripening of different fruits.

### EXPERIMENTAL METHODS

Citrus fruit were degreened in two identical cabinets (50 cu. ft. capacity) where humidity, temperature, ethylene concentration, and the amount of fresh air entering the cabinets could be controlled. Degreening conditions used were those recommended by the Agricultural Research and Education Center at Lake Alfred (6) except for the higher ethylene concentrations. Ethylene from a commercial cylinder was piped into the cabinets through "trickle units" (3, 6) to control the amount of ethylene entering each cabinet. Rates of ethylene flow were adjusted with micro metering valves to maintain about 5 and 50 ppm, respectively. Degreening time was 48 hours. The concentration of ethylene was checked daily using a precision Gas Detector, Unico Model Number 400 with disposable ethylene detector tubes.

Fruit for each experiment were distributed at random into lots of 80 to 100 fruit before degreening. The number of fruit per lot was determined by fruit size in most experiments. Varieties included in these tests were 'Hamlin,' 'Pineapple,' and 'Valencia' oranges; 'Temples,' and 'Dancy' tangerines. After degreening, each lot was washed

Table 1. Effect of two concentrations of ethylene during degreening on decay of 'Hamlin' oranges.

Treatment	Avg. % total decay*			
	Weeks after picking			
	1	2	3	4**
Not degreened	1.7a	8.1a	17.0a	20.1a
Degreened, 4 ppm ethylene	2.0a	15.1a	23.6ab	28.6b
Degreened, 50 ppm ethylene	5.8b	22.9b	32.5b	36.3c

\*Average of three experiments.

\*\*Data followed by the same letter in each column do not differ at a probability level of 5%.

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<sup>1</sup>Trade names are used for purpose of identification and are not an endorsement of the product.

Table 2. Comparison of the amount and types of decay resulting from ethylene degreening of 'Hamlin' oranges.

Treatment	Avg. % decay*			
	4 weeks after picking			
	SER†	M††	O†††	Total**
Not degreened	17.0a	3.1a	0a	20.1a
Degreened, 4 ppm ethylene	23.4b	4.7a	0.5a	28.6b
Degreened, 50 ppm ethylene	31.0c	4.8a	0.5a	36.3c

†Stem-end rot, ††green mold, †††other decay.

\*Average of three experiments.

\*\*Data followed by the same letter in each column do not differ at a probability level of 5%.

separately, waxed with nonfungicidal Flavorseal, and packed in 4/5 bushel, ventilated, telescope-type fiberboard cartons. Storage temperature was 70°F. A nondegreened check lot, handled in the same way as the degreened fruit, was included in each experiment for comparison. Culls and off-grade fruit were removed before packing. Examinations for decay were made 1, 2, 3, and 4 weeks after the picking date, except for 'Temples' which were discarded at the end of the third week of storage. All fruit were examined for evidence of peel injury. Visual color comparisons were made to determine if differences resulted from the ethylene rates.

## RESULTS AND DISCUSSION

'Hamlin' oranges.—Decay of 'Hamlin' oranges degreened with 50 ppm ethylene was increased significantly for the four-weeks holding period (Table 1). One week after picking, the increase in decay was sufficient to have caused rejection of the fruit at the market. Increases in decay due to degreening with 4 ppm ethylene were not statistically significant until the fourth week after picking. All of the increase in decay was due to stem-end rot which is usually caused by *Diplodia natalensis* P. Evans in degreened fruit (Table 2). The increase in green mold, *Penicillium digitatum* Sacc., was not significant.

'Valencia' oranges.—Total decay was lower in 'Valencia' oranges than in 'Hamlin's' but the decay

Table 3. Effect of two concentrations of ethylene during degreening on decay of 'Valencia' oranges.

Treatment	Avg. % total decay*			
	Weeks after picking			
	1	2	3	4**
Not degreened	1.0a	4.2a	6.5a	8.2a
Degreened, 5 ppm ethylene	0.9a	5.2a	11.1a	18.4b
Degreened, 50 ppm ethylene	1.8a	10.9b	21.1b	26.8c

\*Average of four experiments.

\*\*Data followed by the same letter in each column do not differ at a probability level of 5%.

Table 4. Comparison of the amount and types of decay resulting from ethylene degreening of 'Valencia' oranges.

Treatment	Avg. % decay*			
	4 weeks after picking			
	SER†	M††	O†††	Total**
Not degreened	0.4a	5.9a	1.9a	8.2a
Degreened, 5 ppm ethylene	12.6b	3.2a	2.6a	18.4b
Degreened, 50 ppm ethylene	22.5c	2.0a	2.3a	26.8c

†Stem-end rot, ††green mold, †††other decay.

\*Average of four experiments.

\*\*Data followed by the same letter in each column do not differ at a probability level of 5%.

trend was the same. Decay 2, 3, and 4 weeks after picking was increased significantly by degreening with 50 ppm ethylene. Decay 4 weeks after picking was increased significantly by degreening with 5 ppm ethylene (Table 3). All of the increase in decay was due to stem-end rot (Table 4). In three experiments, 'Valencias' were degreened with approximately 120 ppm ethylene, a concentration not unknown in commercial degreening. This ethylene concentration doubled decay at 3 and 4 weeks after picking compared to fruit degreened at 50 ppm.

'Temples'.—The 'Temples' used in the four experiments summarized in Table 5 were injured by freezing weather in January 1971. Degreening significantly increased stem-end rot. Green mold decay was suppressed by degreening with 4 ppm ethylene but not with 50 ppm (Table 5).

Decay of 'Pineapple' oranges and 'Dancy' tangerines was also increased by degreening. Most of the increased decay in citrus fruit degreened with ethylene was caused by stem-end rot. More of this type of decay occurred when fruit were degreened at ethylene concentrations higher than that recommended for commercial use. Green mold decay was not increased by degreening room conditions. No color differences were noted visually in comparable fruit due to degreening with different ethylene concentrations. No peel injury developed that could be attributed to degreening with ethylene.

Table 5. Comparison of the amount and types of decay resulting from degreening of freeze-injured 'Temples.'

Treatment	Avg. % decay*			
	3 weeks after picking			
	SER†	M††	O†††	Total**
Not degreened	7.4a	49.8a	2.7a	59.9a
Degreened, 4 ppm ethylene	18.8b	35.5b	6.2a	60.5a
Degreened, 50 ppm ethylene	23.2b	51.3a	6.8a	81.3b

†Stem-end rot, ††green mold, †††other decay.

\*Average of four experiments.

\*\*Data followed by the same letter in each column do not differ at a probability level of 5%.

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## A COLORIMETRIC METHOD FOR THE DETERMINATION OF RESIDUES OF THIABENDAZOLE IN CITRUS FRUITS

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### ABSTRACT

The fungicide, thiabendazole (TBZ), has been found to be very effective in reducing the decay of citrus fruits. An analytical method for residues of this material was required for evaluation of the various methods of application. A colorimetric method for TBZ in animal feeds was adapted for use with citrus fruits. Stripping of residues from whole fruits with chloroform gave good recoveries of TBZ. Cleanup methods were found to be very important to insure maximum color development. When TBZ was applied by recommended methods, the residues were usually of a magnitude sufficient to meet the minimum 0.1 ppm required by Florida regulations and yet safely below the federal legal tolerance of 2 ppm. TBZ residues were found to decrease slowly during storage.

### INTRODUCTION

The chemical compound, 2-(4-thiazolyl) benzimidazole (thiabendazole or TBZ) has been found to be very effective as a postharvest fungicide for citrus fruits in Florida (4, 5). It has been cleared for use on citrus by the Food and Drug Administration with a tolerance of 2 ppm (1). In the study of methods of application of TBZ to citrus fruit, it was necessary to set up an analytical

method for the determination of residues. Two methods were found in the literature. One, designed for residues in fruits, was based on spectrophotofluorimetry (3) and the other, for determining TBZ in animal feeds, was a colorimetric method (6, 7). Modified extraction and cleanup procedures from the first method were combined with the colorimetric procedure of the second to provide a somewhat simpler means of determining residues of TBZ in citrus fruits.

### EXPERIMENTAL METHODS

Three steps were involved: (1) extraction of TBZ from the fruit, (2) cleanup of the extract to remove interfering materials, (3) development of a color to measure the amount of fungicide present. Two methods of extraction and four cleanup procedures were compared.

#### *Extraction Methods*

1. *Solvent extraction of comminuted fruit.*—Cut fruit was ground in a Waring Blendor with an equal weight of buffer solution (3.3% sodium acetate and 20% sodium chloride). Twenty grams of slurry were shaken in a centrifuge tube with 10 ml buffer and 10 ml ethyl acetate. One, 2 or 4 tubes of a sample were used for each analysis, depending on the expected TBZ content to come within the range of the colorimetric method. Tubes were centrifuged for 10 minutes and the ethyl acetate layer drawn off. Two more extractions were made and the combined extracts evaporated to a volume of 20 ml on a hot plate without active boiling.

2. *Solvent stripping of whole fruit.*—One grapefruit, 2 oranges or 3 tangerines were weighed and placed in a 1 gallon square, wide mouth jar secured in the carrier of a mechanical shaker. After addition of 200 ml of chloroform, a screw cap was placed on the jar and it was shaken for