

Effects of Iron Salts and Other Looseners on Fruit Abscission of Various Citrus Cultivars¹

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Abstract. A foliar spray of various iron salts induced preferential abscission of citrus fruits over that of leaves. This effect was related to Fe cations rather than to anions, and was enhanced at lower pH. The Fe salts can cause marked phytotoxicity, but fruit loosening was achieved at concentrations that caused little to no defoliation, some rind damage, and light to severe injury to young growth and fruit buds. Effective concentration varied for different species. 'Valencia' was more resistant to loosening than the other citrus cultivars tested.

Concentrations of Fe cation in the iron formulations were more than 100-fold that required by cycloheximide, 2 to 10-fold that required by ethephon and about one tenth-fold that required by ascorbic acid to loosen fruit. However, Fe formulations have the following advantages: a) low cost, b) provide essential nutrients to the tree, and c) cause very few problems in registration with the Food and Drug Administration.

The fruit removal force and the sensitivity of 'Calamondin' fruit to various looseners during its ontogenical development varied in the order, starting with the most sensitive phase, flowers, fruits just set, mature fruit, immature fruit, and green full-sized fruit.

Various chemicals offer promise as loosening agents. They induce the formation of the abscission layer and reduce fruit removal force without causing appreciable defoliation (6). Cycloheximide (1, 7), at present the leading loosener, is being tested under the trade name of Acti-Aid on a commercial scale in Florida. A temporary permit for experimental purposes has been granted by the U. S. Department of Agriculture (1). In conjunction with this permit, the Food and Drug Administration has established a temporary tolerance for residues of 3-[2-(3,5-dimethyl-2-oxocyclohexyl)-2-hydroxyethyl] glutarimide (the active ingredient in Acti-Aid) in or on oranges at 0.05 ppm (1). Nearly 1000 acres of citrus were sprayed, some of them over 3 years in succession.

Ascorbic acid and ethephon have been tried experimentally in California and Florida (3, 5, 9, 10). Various Fe compounds have also been suggested as potential citrus looseners (2).

We compared the effects of Fe compounds with other looseners on citrus cultivars.

Materials and Methods

Loosening studies were carried out with orange (*Citrus sinensis* Osbeck) cvs. Washington Navel, Hamlin, Parson Brown, Pineapple, Shamouti, and Valencia; 'Marsh' grapefruit (*Citrus paradisi* Macf.; and 'Calamondin' (*Citrus reticulata* var. *austera*? x *Fortunella* sp.). The orange and grapefruit trees were grown either in various parts of the coastal region of Israel or in the experimental orchard of the University of Florida at Gainesville and in the Clemens and Williams orchard at Citra. One-year old potted 'Calamondin' plants were kept in the greenhouse. This dwarf citrus cultivar, which bears fruit in its first year of growth, enables it to be used to run large-scale abscission studies under controlled greenhouse conditions.

The various loosener solutions were applied by spraying separate branches or whole trees until run-off. Each 20-year-old

tree was sprayed with 40 liters of solution. The fruit was clipped from the tree with a 5 cm long stem just before measurements were made. The following chemicals were tried: 1) cycloheximide, 2) ascorbic acid, 3) ethephon, and 4) various Fe and Cu salts.

Fruit removal force (FRF) was measured by the following instruments: hand-operated Chatillion tensiometer; Instron model TM stress and strain analyzer; and a home-made instrument measuring force by a straight pull at a constant rate of movement, as Instron does. The indicator for the FRF was a Chatillion tensiometer of 25 kg. Comparative studies with these instruments showed that the straight pull at a constant rate of movement is essential for consistent and accurate reading of the fruit-removal force.

Defoliation was measured by weighing and counting the leaves that were shed. Extent of defoliation was expressed as the percentage of the total foliage that abscised. Repeated checks were made of the investigators' estimates by directly counting the entire foliage of a treated branch.

The change in FRF during fruit development and the response of fruit of various ages to different looseners was investigated in 'Calamondin' plants since these trees simultaneously bear flowers and fruits of all ages. Thus, measurements at any one time of fruits in various stages of development could be made.

Results

Effects of Fe and Cu salts and other looseners. A foliar spray of various Fe salts induced in different citrus cultivars abscission of fruit preferentially over that of leaves (Tables 1, 2, 3 and 4). Induction of the abscission process was demonstrated both by the reduction of the FRF required to separate fruit from its stem and by the lower percentage of fruit that was plugged or stems broken when the FRF was determined.

Various Fe salts gave different and sometimes erratic results. For example, FeCl₃ usually gave adequate loosening with little defoliation, but at times, particularly in trees low in vigor, an injurious amount of defoliation accompanied loosening. On the other hand, Ben-Yehoshua et al. (4) reported that several other Fe formulations consistently induced loosening with little defoliation (0 to 10%) and at a lower dosage than FeCl₃. Iron salts also caused rind damage such as black pits in oranges and 'Calamondin' and red pits in grapefruit.

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Table 1. Effect of various materials on the attachment force of 'Shamouti' oranges 1 week after spraying^Z.

Treatment	Concn (ppm)	Fruit removal force lb.	Injury to fruit	Defoliation percentage
Untreated		13.9	None	0 - 2
Ascorbic acid	35.000	8.6	Some	0 - 5
Ethephon	1.000 to 2.000	5.9	None	5 - 50
Cycloheximide	20	5.9	Some	0 - 5
Copper chloride	1.700	5.5	Severe	5 - 20
Various Fe formulations	3.000 to 20.000	6.4	Some	0 - 5

^ZEach loosener solution also contained 0.02% surfactant.

Copper salts were both more phytotoxic and better defoliant than Fe salts in all cultivars (Tables 1, 2, and 4). Plants treated with 0.1 M CuCl₂ showed first wilting of their leaves and abscission of most leaves and fruit, followed by death of the treated branch or plant 10 to 20 days after treatment.

Table 2. Effect of sprays of various concn of CuCl₂, FeCl₂, FeCl₃, and ascorbic acid on abscission of fruit and leaves of intact 'Calamondin' plants 8 days after treatment.^Z

Treatment	Molar concn	Number of leaves abscising ^X	Percent mature fruit abscising		Fruit removal force (lb.) for fruit of color ^Y		
					yellow-orange	green-yellow	green
			Yellow	Green			
Control ^W	—	0	0	0	3.6 (100)	4.1 (100)	5.4 (100)
Ascorbic acid	0.284	21	59	37	1.0 (0)	2.4 (20)	4.4 (100)
	0.100	9	25	15	2.5 (50)	4.3 (20)	5.9 (80)
	0.010	10	0	2	3.5 (100)	5.1 (100)	5.1 (100)
	0.001	5	0	4	2.5 (70)	3.1 (90)	5.9 (100)
FeCl ₂	0.100	95	100	100	All abscised	2.9 (0)	5.1 (100)
	0.010	13	100	85	abscised	2.9 (0)	5.1 (100)
	0.001	5	0	4	2.5 (70)	3.1 (90)	5.9 (100)
	0.100	5	80	69	2.1 (10)	3.4 (100)	6.9 (100)
FeCl ₃	0.010	2	0	9	2.3 (50)	4.5 (100)	6.3 (100)
	0.001	0	0	6	2.6 (60)	4.7 (100)	5.0 (100)
	0.100	221	Dead	abscised	3.4 (50)	6.9 (100)	6.9 (100)
	0.010	133	100	79	2.2 (75)	5.5 (100)	5.5 (100)
CuCl ₂	0.001	11	75	6	2.1 (30)	3.6 (50)	4.4 (100)

^ZEach treatment was applied to 4 plants; the mean standard error of treatments was 0.56.

^YThe number in parentheses denotes the percentage of fruit plugged.

^XEach plant had about 200 - 250 leaves; abscission was measured 6 weeks after treatment.

^WControl plant sprayed with glass-distilled water.

The fruit had green pits over its rind. However, CuCl₂ at concn of 0.01 M and 0.001 M reduced the FRF with less defoliation (0 to 10%). Phytotoxicity at these concn was limited to slight defoliation, rind injury, and damage to young growth.

Cycloheximide, the most effective loosener, induced abscission at the lowest concn of all the chemicals tested (Table 1). A spray of 20 ppm cycloheximide adequately loosened various citrus cultivars with little or no defoliation. Ethephon was second in effectiveness. A spray of 1000 to 2000 ppm ethephon loosened the fruit, but at this dosage it also defoliated 5 to 50% of the tree depending on the season, weather, and other factors.

Ascorbic acid was less effective than the metal salts. Usually 3- to 10-fold higher concn were required to induce comparable loosening of fruit than that induced by various iron salts.

Cation versus anion effect. The induction of abscission was related mainly to the cation rather than the anion (Table 3). The chloride anion at similar pH did not cause any abscission at 0.01 M HCl. At 0.1 M, as KCl, it caused slight and erratic reduction in the FRF without any effect on the percentage of plugging. Iron salts were effective in loosening fruit to a similar extent when given as chloride, or as the complex form, ferric ammonium citrate.

Effect of pH of the loosener solution. The pH of the loosener solution affected both phytotoxicity and loosening

effectiveness. Lowering the pH increased the effectiveness of both FeCl₃ and CuCl₂ in loosening 'Pineapple' orange fruit (Fig. 1). At pH 1.0, HCl induced some loosening and reduced the FRF from 21.7 to 16.1 lb. However, adding 1 x 10⁻⁴ M CuCl₂

Table 3. Effect of spray of various cations and anions, at pH 2.0, upon abscission of orange fruit.^Z

Date of spray (1969)	Cultivar	Treatment	Molar concn	Fruits removal force lb.	Plugging %	Area injured %
Jan. 28	Pineapple ^Y	Untreated		21.1 a	100	0
		HCl	0.01	21.2 a	80	3
		H ₃ PO ₄	"	21.5 a	60	0
		FeCl ₃	"	11.6 b	0	7
		FeCl ₂	"	13.3b	0	7
		Untreated		20.3 a	100	0
April 4	Valencia ^X	KCl	0.1	15.0 ab	100	11
		Tris	"	17.8 a	80	12
		FeCl ₃	"	8.9 b	33	20
		Ferric ammonium citrate	"	10.4 b	25	22

^ZTreatments were applied to separate branches by spraying to run-off. Each value is an average of 10 fruits. Mean separation within dates by Duncan's multiple range test at 5%.

^YFruit removal force was measured 7 days after spray application.

^XFruit removal force was measured 19 days after spray application.

Table 4. Effects of FeCl₃ in loosening fruits of citrus cultivars.

Date	Type of treatment	Cultivar	Effective concn (M)	Fruit removal force %	Reduction in plugging ^y	Defoliation %
Oct. 3, 1968	Overall tree Spray	Calamondin	0.01	58	90	2
Nov. 26, 1968	Branch test	Hamlin orange	0.1	58	60	0-4
		Navel orange	0.1	63	40	0
		Marsh grapefruit	0.1	44	82	0-2
Jan. 28, 1969	Branch test	Pineapple orange	0.01	43	90	0
Feb. 17, 1969	Overall tree spray	Pineapple orange	0.01	47	20	—
		Parson Brown orange	0.1	50	60	—
		Marsh grapefruit	0.01	55	20	—
Feb. 26, 1969 ^x	Overall tree spray	Pineapple orange	0.03	43	44	0-5
		Valencia orange	0.03	0	0	0
		Grapefruit	0.03	61	0	0
	Branch test	Pineapple orange	0.05	53	90	—
April 2, 1969	Branch test	Valencia orange	0.1	50	64	—
Feb. 2, 1970	Overall tree spray	Shamouti	0.05	49	0	2-5

^zFruit removal force of untreated fruit is 100%.

^yPlugging of untreated fruit minus plugging of treated fruit.

^xYoung growth had light to severe injury.

to a solution of HCl at pH 1.0 reduced the FRF to 53%. As the pH rose, higher concn of the loosener were required to induce loosening (Fig. 1). Similar results were obtained with 'Marsh' grapefruit. At pH 1.0, 1×10^{-4} M CuCl₂ reduced the FRF of the grapefruit from 12.9 to 1.2 lb. Unfortunately, the phytotoxicity was also much enhanced by the lower pH. At pH 1.0, defoliation, up to prohibitive levels, was caused by those looseners that did not affect leaves at pH 2.0. Rind damage was also much more severe at pH 1.0 than at 2.0.

Other acids induced more phytotoxicity than HCl. Phosphoric and citric acids at pH 1.0 caused defoliation of more than half the foliage, injured the fruit rind markedly and reduced the FRF by approximately one half.

At pH 2.0 citric, HCl, and phosphoric acids rarely caused defoliation; however, sulfuric acid, even at pH 2.0, caused defoliation of more than 10% of leaves and rind damage as well as loosening.

Response of different cultivars. Tables 2 and 4 show that 0.01 M of iron salts is the optimal concn to induce loosening of 'Calamondin' fruit. A lower concn did not have any effect and a higher concn caused a higher percentage of defoliation, about 5 to 10%, and marked injury to the fruit. The 'Calamondin' was very sensitive to this treatment because of its thin rind (1 - 2 mm). At 0.1 M FeCl₃, the injury penetrated through the rind to the pulp whereas in other cultivars the injury did not penetrate the rind. However, concn which effectively loosened oranges or grapefruits (Table 4) also caused enough rind damage and deterioration in fruit appearance to render the fruit unsuitable for the fresh market. The extent of damage varied, but usually up to 20% of the area of the rind was covered with spots and pits. The effective dosage varied depending on season, weather, method of spray, and other factors (4).

The 'Valencia' fruit, known to resist various loosening treatments (6), did not respond to the Fe salts as favorably as other citrus cultivars (Table 4). The fruit was adequately

loosened only by about 2- to 4-fold higher concn than that dosage which was effective in other citrus cultivars. Furthermore, as the 'Valencia' was harvested just when the tree blossoms or sets fruit, the loosener caused the abscission of many flowers and very young fruit.

Natural and induced rate of loosening. Figure 2 shows the rate of loosening of fruit of the 'Pineapple' orange both as developed naturally during the season and also as induced by a spray of FeCl₃. Fruit of this cultivar shed naturally at the end of the season, unlike 'Valencia' which does not abscise even after two years, and indeed the FRF of 'Pineapple' declined gradually from 21.1 lb. on Feb. 4th to about 7 lb. at the end of March. The iron spray induced a rapid decline in the FRF. This decline continued for 12 days in the second loosening trial.

The fruit did not shed during the course of this experiment however, in other experiments, a strong wind caused shedding of treated fruit.

Loosening and abscission behavior during fruit development. The effects of different looseners upon abscission and loosening of fruit of 'Calamondin' plants varied during its ontogenical development. Tables 2 and 5 show that the iron and copper salts and ascorbic acid affected the mature orange-yellow fruit more than the yellow fruit, which in turn was affected more than the green fruit. Most flowers and fruit that just set were shed by a spray of the following: 0.28 M ascorbic acid, 0.01 M FeCl₂, 0.1 M FeCl₃, or by 0.001 M CuCl₂. The changes in the FRF of 'Calamondin' fruit during its development appear to relate directly to the sensitivity of the fruit to the various looseners (Table 5). The least sensitive and the strongest in FRF was the large green fruit. The mature orange-yellow fruit and the immature fruit were more sensitive to looseners and less strongly attached to the plant. Flowers and fruit that had just set were most sensitive to looseners. Leaf primordia and the young leaves were also much more sensitive to damage by the looseners.

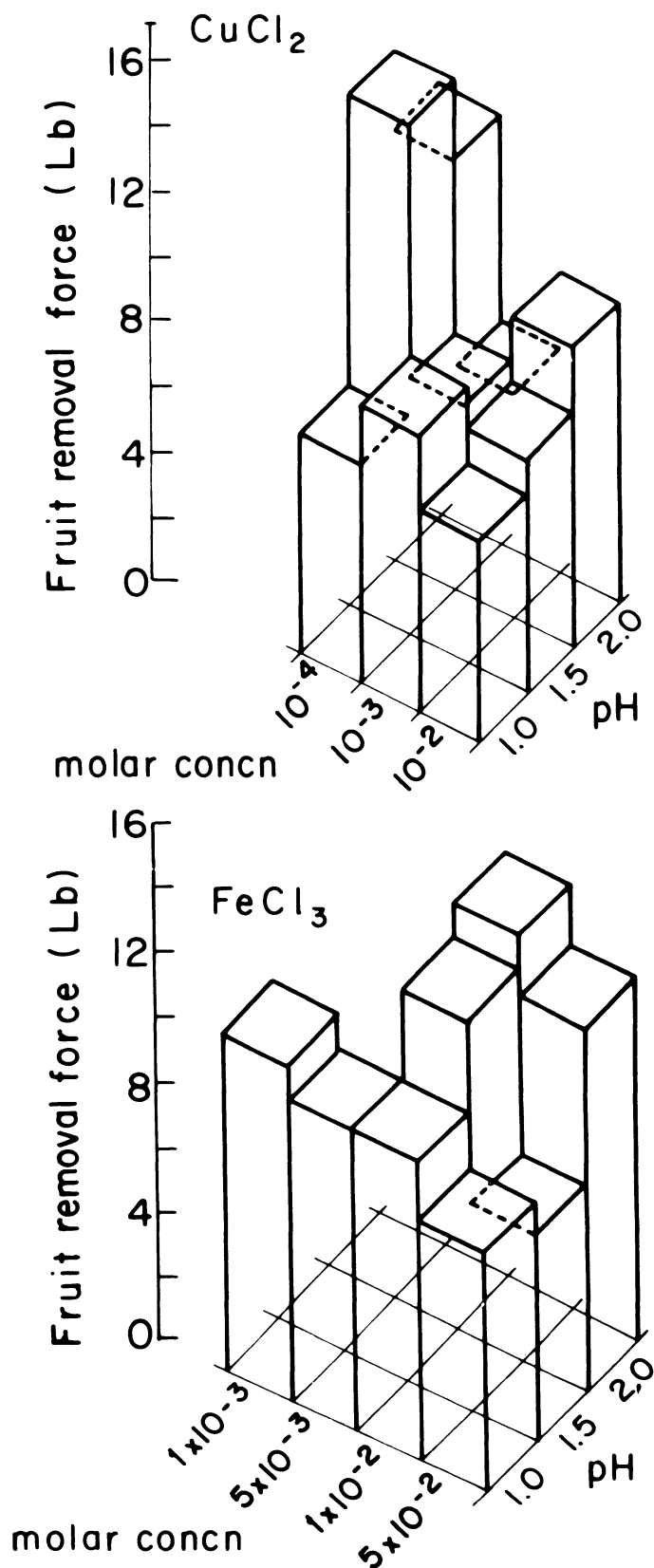


Fig. 1. Effect of low pH and different FeCl_3 and CuCl_2 concn on fruit removal force of 'Pineapple' orange 1 week after spraying. The pH was adjusted by adding 1N HCl to the solution.

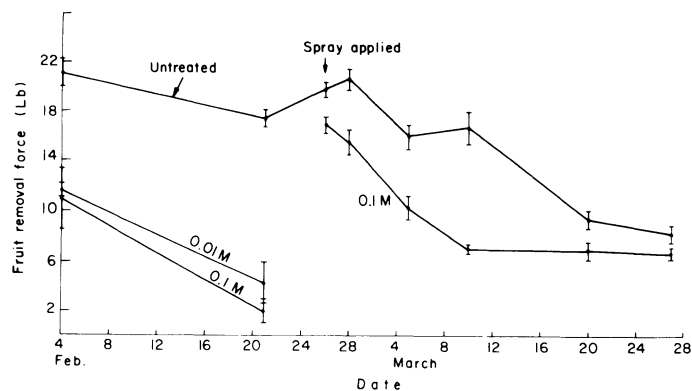


Fig. 2. Changes in the bonding force of fruit of 'Pineapple' orange along its natural reduction in fruit removal force (FRF) and as induced by a spray of 0.01 and 0.1 M FeCl_3 . The first spray was applied 1 week before the first measurement of FRF. The arrow indicates the time of the second spraying. The vertical lines represent 2 standard errors.

Discussion

Commercially, mechanical harvests of citrus fruit have consistently shown the need for a pretreatment to reduce the FRF (4, 6, 8, 10). Such a pretreatment has to fulfill the following requirements: effective and consistent loosening, lack of toxicity to humans, lack of phytotoxicity, and low cost.

Unfortunately, such a pretreatment is not yet available. A whole group of materials (such as cycloheximide, ascorbic acid, iron salts) can be sprayed before harvest and loosen the fruit, but a high degree of rind damage accompanies the loosening. Thus, their use is prohibited for fruit that is designated for the fresh market. So far, only ethephon induces loosening without causing any rind damage (6, 9). Still, this material cannot be used because it induces prohibitive amounts of defoliation. The observation that ethephon can loosen fruit without injuring the rind is most encouraging because it demonstrates that adequate loosening is not necessarily related to rind damage. Consequently, the search should be continued for looseners that do not cause defoliation, or for a combined treatment such as, adding a growth regulator to ethephon, that would prevent the defoliation induced by ethephon without affecting the induced loosening.

Cycloheximide appears, so far, to be the most promising material for loosening fruit of various citrus cultivars destined for processing. However, as cycloheximide is an effective inhibitor of protein synthesis and an antibiotic (7), the question of long range toxicity to the trees and the lack of final clearance by the Food and Drug Administration delays its commercial utilization.

The Fe formulations tried appear also to be effective looseners of processed fruit in Israel. Although rind damage

Table 5. Changes in fruit removal force of 'Calamondin' and its sensitivity to FeCl_3 spray during its development.

Fruit age	Diameter mm	Fruit removal force lb.	Rating of sensitivity to loosener
Flowers	—	—	Most sensitive
Fruit that just set	—	—	Very sensitive
Very small fruit	0.5	2.3 ± 0.2	Sensitive
Small fruit	0.5 - 1.0	4.6 ± 0.3	Less sensitive
Mature green fruit	Max. size	5.4 ± 0.2	Least sensitive
Yellow-green fruit	Max. size	4.1 ± 0.2	Less sensitive
Orange-yellow	Max. size	3.6 ± 0.4	Sensitive

caused by the iron spray might raise the incidence of decay, Ben-Yehoshua et al. (4) have recently reported that introducing the fungicide benomyl to the Fe formulation reduced decay of mechanically harvested grapefruit to a level below that of hand-picked fruit which had not received a benomyl spray.

The dosage required by the iron formulation is more than 100-fold higher than that of cycloheximide. However, the loosening action involves also a foliar spray of necessary nutrients for citrus trees and the iron formulation is lower in cost than cycloheximide.

The reports of Wilson (10) and of Hield et al. (9) that FeCl₃ causes much defoliation could be explained by the variability of the phytotoxic behavior of FeCl₃. However, other Fe formulations are less phytotoxic and loosen fruit at lower concn (4 and unpublished data).

The costs of ascorbic acid appears to restrict its feasibility as a commercial loosener, particularly since a high dosage is required to loosen fruit.

The loosening effects of acids and the action of low pH in enhancing the effects of looseners confirm previous studies (6, 7). However, the action of Fe salts cannot be ascribed altogether to low pH of their aqueous solutions, since loosening was achieved by Fe salts even at pH 5.0.

The loosening of citrus fruit has so far been found by all the investigators to involve a risk of phytotoxicity. Accordingly, more trials on a larger scale and of longer duration are necessary before final evaluation of a loosener can be made.

The changes in FRF of 'Calamondin' fruit during its development parallel the changes in sensitivity of this fruit to the various looseners. 'Valencia' fruit also responded similarly to looseners; that is, many flowers and small fruit abscised, but mature and immature fruit did not. Thus, loosening sprays

applied during the period of blossoming and fruit set involve great losses of these organs. The lower sensitivity of the green immature fruit compared with the mature fruit permits greater selectivity in mechanical harvesting of the mature fruit without affecting green fruit. Our work confirmed a previous report by Cooper et al. (5) that the green immature fruit of 'Valencia' is least sensitive to cycloheximide spray compared with both small 0.3 to 0.5 gm fruitlets or mature fruit.

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Effect of 2,3,5-Triiodobenzoic Acid on Bitter Pit and Calcium Accumulation in 'Northern Spy' Apples¹

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Abstract. Spraying 'Northern Spy' apple trees with 50 ppm TIBA 2 weeks after petal fall reduced the accumulation of Ca in the fruit and increased the amount of bitter pit. The greatest incidence of bitter pit was associated with the Ca content of the fruit during the middle of the growing season. Calcium accumulated rapidly during the period of cell division and seed development, and again 2 to 3 weeks before harvest. The late influx of Ca may explain the development of less bitter pit in storage in late harvested apples than in apples harvested immature.

The variability in the percentage of apples showing bitter pit, in the cv. Northern Spy, is generally as great between replications as it is between various spray treatments with Ca salts. The percentage of fruit developing bitter pit varies between trees and also between limbs on the same tree. The lack of uniformity in the amount of bitter pit makes it difficult to reproduce consistent results. However, if the amount of bitter pit can be increased, the effects of differences in mineral-element content may be more accurately measured.

The percent bitter pit has consistently been associated with Ca content of apple fruits (1, 5, 6, 9, 10, 12). Stahly and co-workers (9, 10) reported 2,3,5-triiodobenzoic acid (TIBA) reduced the Ca content of fruit and increased the amount of bitter pit in 'Golden Delicious' apples. The effect of TIBA on Ca

movement and accumulation in other plant tissues has also been reported (3, 4).

Data were collected for 2 years at Ithaca, New York on the effect of TIBA on Ca accumulation in apple fruits throughout the growing season and the relation of total fruit Ca to the percentage of bitter pit.

Materials and Methods

Eight, 40-year-old, 'Northern Spy' apple trees, in the Cornell Orchard, were selected for uniformity of crop load. Full bloom dates for the 'Northern Spy' trees were May 18, 1970, and May 22, 1971. Two weeks following petal fall, an aqueous solution of 50 ppm TIBA was applied to alternate trees with a hydraulic sprayer at 500 psi. The trees were wet to runoff.

1970. Fruit samples were collected at weekly intervals, starting about one week past petal fall, May 28, and continuing

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