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CHEMICAL CORPS RESEARCH & DEVELOPMENT COMMAND

BIOLOGICAL WARFARE LABORATORIES

INTERIM REPORT 140

DEFOLIATION INVESTIGATIONS DURING 1954 AND 1955 (U)

FORT DETRICK
MARYLAND

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INTERIM REPORT 140

DEFOLIATION INVESTIGATIONS DURING 1954 AND 1955 (U)

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Work completed October 1955

This is a report of record and does not necessarily reflect the doctrine of the Chemical Corps.

CHEMICAL CORPS RESEARCH AND DEVELOPMENT COMMAND
BIOLOGICAL WARFARE LABORATORIES
CROPS DIVISION
Fort Detrick, Frederick, Maryland

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ABSTRACT

In field experiments at Camp Detrick, Maryland, Avon Park AFB, Florida and Fort Ritchie, Maryland, disodium 3,6-endoxohexahydrophthalate and 2-butyne-1,4-diol were the most effective defoliants of woody vegetation. Pentachlorophenol, tributyl phosphate, di-(ethylxanthogen) trisulfide and 2-sec-butyl-4,6-dinitrophenol showed promise as desiccants of woody species. There was considerable variation in the response of deciduous species to defoliants, but results indicate that highly effective defoliants can be developed for use in tactical military situations.

SUMMARY

This report is a summary of work on the Camp Detrick defoliation program prior to October 1955. This long-term program was established to discover, evaluate, and develop to standardization the chemical compounds which may be used to cause leaves of natural woody vegetation to defoliate, dry up, or change color. Screening, field testing, species variation, and a field comparison of two promising compounds are described and discussed.

In preliminary greenhouse and field screening experiments at Fort Detrick, compounds found to be most effective as defoliants were disodium 3,6-endoxohexahydrophthalate and 2-butyne-1,4-diol. Compounds which induced rapid desiccation of foliage were tributyl phosphate, pentachlorophenol, and 2-sec-butyl-4,6-dinitrophenol.

These same compounds were, for the most part, demonstrated to be the best desiccants and defoliants on various woody species at Avon Park Air Force Base, Florida. Outstanding in these trials were pentachlorophenol, tributyl phosphate and di-(ethylxanthogen) trisulfide in diesel oil as desiccants, and 2-butyne-1,4-diol as an abscissant.

Application of a commercial formulation of disodium 3,6-endoxohexahydrophthalate to 29 deciduous species in Maryland demonstrated considerable species variability. Elm, basswood, maple, cherry, locust, and birch were readily defoliated. Somewhat more resistant were dogwood, willow, hickory, hophornbeam, ash, tuliptree and beech. Extremely resistant to abscission were all four species of oak, whose leaves characteristically became desiccated.

Endothal and 2-butyne-1,4-diol were about equally effective in inducing defoliation on a number of deciduous species in Maryland. 2-Butyne-1,4-diol was slightly more effective in causing defoliation of one species of oak.

I. INTRODUCTION

Chemical defoliation and desiccation of forest and jungle vegetation can be extremely beneficial in tactical military operations. It is envisaged that these chemical agents might be employed to reduce enemy concealment, to expose fortifications, to clear fields of fire, and to mark targets. Improvement of visibility may be accomplished by the use of defoliants, which cause plants to shed their leaves, and by the use of desiccants, which kill and shrivel the leaves. Both defoliants and desiccants may be used alone or they may be used prior to the use of fire.

Herbicidal chemicals causing contact injury (desiccation) and compounds stimulating abscission (defoliation) are used extensively in weed control and cotton defoliation. Some of these compounds, or others causing a similar response, may be effective as defoliants or desiccants of trees, shrubs, and vines.

This report summarizes greenhouse and field defoliation investigations conducted during 1954 and 1955, including selection of active compounds, field testing, variation in response among species, and comparison of the effectiveness of two of the most promising compounds.

II. SCREENING TRIALS AT CAMP DETRICK

A. GREENHOUSE SCREENING

With the inception of the present defoliation program in June 1954, a need was recognized for a screening test to evaluate various compounds. To evolve a screening method, 70 compounds dissolved in acetone with 1.0% Tween-20 were sprayed on Black Valentine beans (Phaseolus vulgaris) at rates of 2, 4, and 8 pounds per acre. Plants were treated 11 to 12 days after planting, when the first trifoliolate leaves had expanded to about one-third of mature size. Responses were noted 3 to 6 days after treatment and classified as follows: leaves partially necrotic, leaves fully necrotic but not abscised, and leaves detached.

On the basis of responses so obtained, the compounds were separated into three groups of activity: (a) 53 compounds, caused partial necrosis of all leaves or complete necrosis of some; (b) 11 produced complete necrosis of all leaves; and (c) 6 chemicals induced defoliation.

Following these preliminary tests with beans, it was decided that a more critical evaluation of compounds would be possible if additional species were included as test plants. Species used were Chinese elm (Ulmus pumilla), English ivy (Hedera helix), privet (Ligustrum sinense) and a small cactus (Pereskia aculeata). Elm and privet, being woody were used most extensively to simulate field species to be encountered.

In general, the compounds used in these tests were applied in water with 1% Tween-20, or in acetone with 1.0% Tween-20 or diesel oil. The plants were either sprayed to run-off with a known concentration, or else a given area was uniformly sprayed at a designated rate per acre. Following treatment, the degree of contact injury and defoliation was estimated visually over a period of 10 days to 2 weeks.

Between 13 December 1954 and 1 October 1955, 300 additional compounds were evaluated in the greenhouse by the test described above. Of these, 65 of the more promising compounds were tested on woody species in the field.

B. FIELD SCREENING

In field applications, plants were treated with a battery-operated portable sprayer (McLane et al 1954)[1/]*. This equipment permitted application of small volumes to small areas of foliage (approximately 2 square feet). Species included in field tests at Camp Detrick were white ash (Fraxinus americana), cherry (Prunus sp.), black locust (Robinia pseudo-acacia), elm (Ulmus americana), tuliptree (Liriodendron tulipifera), mulberry (Morus sp.), hickory (Carya sp.) and spicebush (Lindera benzoin).

C. COMPARISON OF FIELD AND GREENHOUSE SCREENING RESULTS

The results summarized in Table I include only a few compounds but typify responses commonly obtained. Compounds causing contact injury in the greenhouse generally performed similarly in the field. The same was not always true of abscission-inducing compounds, since variation in species behavior was marked. The complexity of the abscission process and the exacting requirements conditioning it undoubtedly contribute to this variation. Many compounds produced abscission and/or necrosis, depending upon the rate of application.

D. DISCUSSION AND CONCLUSIONS

The compounds found to be most efficacious in causing abscission were disodium 3,6-endoxohexahydrophthalate (endothal) and 2-butyne-1,4-diol (butynediol). Two or three days were required for noticeable defoliation with these chemicals with maximum response elicited by seven to ten days. Variation in species response was considerable.

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* See References

Most effective in causing desiccation injury were tributyl phosphate, pentachlorophenol, and 2-sec-butyl-4,6-dinitrophenol. Both phenols are formulated commercially. Necrosis of 75 to 100 percent was obtained within 24 hours on nearly all species with these chemicals.

Rates of application and volumes of solution required for effective defoliation or desiccation (necrosis) were not critically evaluated in these early tests.

III. FIELD TRIALS AT AVON PARK, FLORIDA

These trials were run to evaluate further under field conditions those chemicals which showed potentialities in the preliminary tests. This work was carried out at Avon Park Air Force Base, Florida, during March and April 1955. Emphasis was placed on species variation and also on the effects of varying rates, volumes, and solvents.

A. MATERIALS AND METHODS

Wild orange (*Citrus sp*), holly (*Ilex glabra*), wax myrtle (*Myrica cerifera*), oak (*Quercus virginiana* or *laurifolia*), and willow (*Salix nigra*) were treated in these trials.

The chemicals selected for testing were as follows:

1. Applied in oils: tributyl phosphate, pentachlorophenol, di-(ethyl-xanthogen) trisulfide, butyl 2,4-D and butyl 2,4,5-T mixtures, and butyl 2,4,5-T alone.

2. Applied in water: 2-butyne-1,4-diol, ammonium thiocyanate, 2-sec-butyl-4,6-dinitrophenol, disodium 3,6-endoxohexahydrophthalate, magnesium chlorate (hexahydrate), monosodium cyanamid, sodium chlorate - sodium pentaborate mixture, and 3-amino-1,2,4-triazole.

3. Applied in acetone: 2,4-dichlorophenoxyacetamide. With the exception of butyl 2,4,5-T, tributyl phosphate, ammonium thiocyanate and 2,4-dichlorophenoxyacetamide, the chemicals were commercial formulations. Consequently, the results obtained were due to the combined effect of the active ingredient, additives such as surfactants, and solvents.

The following oils were tested as solvents:

a. Diesel oil - distilled from petroleum after the kerosene fraction.

b. Deobase - deodorized derosene, non-phytotoxic.

c. Varsol - a petroleum solvent.

TABLE I. COMPARISON BETWEEN GREENHOUSE AND FIELD RESULTS *
(five days after treatment)

		GREENHOUSE			FIELD					
CD No.	lb/Acre	Bean	Elm	Privet	Ash	Cherry	Mulberry	Elm	Tulip	Spicebush
12965	2	0	0	0	C-2	0	0	0	C-1	0
	4	-	-	-	A-1	A-1	0	0	C-1	0
	8	-	-	-	C-2	A-1	C-1	0	A-2	C-1
12160	2	0	0	0	0	0	0	0	0	0
	4	A-1	0	0	0	0	0	0	0	Ch-1
	8	A-2	C-1	0	C-1	0	0	0	0	0
13104	2	0	0	0	C-1	C-1	0	0	0	0
	4	A-1	C-1	C-1	C-1	C-1	0	0	C-1	C-2
	8	A-2	C-1	C-1	C-3	A-2	C-1	C-1	C-2	C-2
13106	2	0	0	0	C-1	0	C-1	0	0	0
	4	C-1	C-1	A-3	C-2	A-2	C-1	0	C-1	Ch-1
	8	A-2	C-1	A-3	C-3	A-4	A-1	C-1	C-1	0
13109	2	0	0	0	0	0	0	0	0	0
	4	0	0	0	C-1	C-1	0	0	0	Ch-1
	8	A-3	0	0	C-1	0	0	0	0	0
13110	2	0	0	0	0	0	0	0	0	0
	4	C-1	0	0	C-1	C-1	0	0	C-1	Ch-1
	8	C-2	C-1	A-2	C-2	C-1	C-1	0	0	C-1
13112	2	0	A-1	A-1	C-2	A-1	C-1	A-1	C-1	C-1
	4	A-1	A-4	A-1	C-4	A-2	A-2	A-4	A-1	C-2
	8	A-3	A-2	A-3	C-3	A-1	A-4	A-2	A-3	C-2
13113	2	C-4	C-2	C-1	C-2	C-2	C-1	C-1	C-2	C-2
	4	-	-	-	C-4	A-2	C-4	A-1	C-4	C-4
	8	-	-	-	C-4	A-3	C-4	A-2	C-4	C-4
13115	2	A-4	A-1	A-1	A-2	C-4	A-3	A-4	A-1	A-3
	4	-	-	-	C-4	C-4	A-1	A-3	C-4	A-4
	8	-	-	-	C-4	C-4	A-1	A-4	C-4	A-2
13116	2	C-1	0	0	C-2	0	C-2	0	C-1	Ch-1
	4	C-4	A-1	0	C-3	A-3	A-2	A-4	A-3	Ch-1
	8	A-4	C-3	C-1	C-3	A-3	A-1	A-3	A-4	Ch-1

* A - Abscission (defoliation)

C - Contact Injury

Ch - Chlorosis

0 - No effect
1 - 0 to 25%
2 - 26 to 50%
3 - 51 to 75%
4 - 76 to 100%

TABLE I (continued)

CD No.	Chemical Name
12965	Propionic acid, 2,2,3-trichloro-, sodium salt
12160	4-Octyne-3,6-diol, 3,6-dimethyl-
13104	1,3-Diphenyl-1-butyne-3-ol
13106	2,5-Diphenyl-3-hexyne-2,5-diol
13109	Propargyl chloride
13110	2-Butene-1,4-diol
13112	2,4-Hexadiyne-1,6-diol
13113	o-Chlorophenol sulfonyl fluoride
13115	Endothal anhydride
13116	3,6-Endoxohexahydrophthalic acid, one-half tridecyl alcohol (primary branched chain by OXO process) ester: cupric salt

d. Richsol - a highly volatile dry-cleaning fluid.

e. Sevacide - a phytotoxic oil.

The chemicals were applied with a 250-ml hand sprayer operated by carbon dioxide and equipped with a flat-spray Tee jet nozzle, usually to 16 square feet of area. In the case of small shrubs such as holly and willow, several plants were included in each treatment. Applications to the larger plants included only a few branches. The volume of solution sprayed varied with the experiment and ranged from 20 to 100 ml per plot. One ml per 16 square feet was equivalent to 0.75 gallon per acre.

Treated plots were observed at frequent intervals over a period of several days. The degree of defoliation, desiccation, or chlorosis was rated as 1 (0-25%), 2 (26-50%), 3 (51-75%) or 4 (76-100%).

B. RESULTS AND DISCUSSION

The chemicals applied in oil generally produced severe contact injury and desiccation, but little defoliation. Those applied in aqueous solution caused more defoliation, chlorosis, and discoloration with little contact injury. Results obtained with chemicals causing contact injury in these trials were more closely related to the greenhouse screening results than were those obtained with defoliant compounds.

Using 40 percent of formulated material in diesel oil, pentachlorophenol and tributyl phosphate usually caused 75 to 100 percent contact injury on willow, orange, wax myrtle, holly and oak within a day following treatment (Table II). Di-(ethylxanthogen) trisulfide was less effective on these species. Ammonium thiocyanate produced red coloration of leaves of holly and wax myrtle in 24 hours, and 75 to 100 percent contact injury on all species within 6 days.

2-Butyne-1,4-diol caused 75 to 100 percent defoliation on all species except willow within six days (Table III). Defoliation was evident on orange, wax myrtle and holly in three days. Endothal was most effective on orange and holly.

Of the organic solvents tested, none appeared to be more efficacious than diesel oil when applied with tributyl phosphate, pentachlorophenol and to a lesser extent di-(ethylxanthogen) trisulfide on the species treated.

IV. SPECIES VARIATION TRIALS AT FORT RITCHIE, MARYLAND

In view of the variation in species response to many of the abscissants tested, the effectiveness of disodium 3,6-endoxohexahydrophthalate, (endothal) was compared on 29 species of trees and shrubs characteristic of the eastern deciduous forest of this country. This work was done

TABLE II. CONTACT INJURY CAUSED BY THREE CHEMICALS ON VARIOUS SPECIES ONE DAY FOLLOWING TREATMENT

Diesel Oil Was Used as a Solvent

Compound	Formulation Concentration	Percent of Formulation[a]	Citrus	Oak	Wax Myrtle	Holly	Willow
Tributyl phosphate	100	10	0[b]	2	2	2	2
		40	3	4	4	3	4
Pentachlorophenol	40	10	3	4	1	1	3
		40	3	4	4	2	4
Di-(ethylxanthogen) trisulfide	25	10	0	1	1	1	2
		40	0	1	2	1	3

a/ Percent of formulation mixed with diesel oil. Rate of 36 gal/acre.

b/ - 0 - No effect; 1 - 0 to 25%; 2 - 26 to 50%; 3 - 51 to 75%; 4 - 76 to 100%.

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TABLE III. DEFOLIATION AND CONTACT INJURY CAUSED BY FIVE CHEMICALS ON VARIOUS SPECIES SIX DAYS FOLLOWING TREATMENT.

Aqueous Solution.[a/]

Compound	Formulation Concentration	Citrus		Oak		Wax Myrtle		Holly		Willow	
		C[b/]	D	C	D	C	D	C	D	C	D
2-Butyne-1,4-diol	35	-	3[c/]	3	3	4	4	-	4	3	0
Endothal	6.3	-	4	4	0	4	0	-	4	3	1
Magnesium chlorate	58	1	1	2	0	4	3	4	2	4	0
Sodium chlorate - pentaborate	28	3	1	2	0	4	1	4	1	4	2
Ammonium thiocyanate	100	4	2	4	1	4	0	4	1	4	0

a/ Aqueous solution consisting of 40% of the formulation and 1% Tween-20. Rate of 54 gal/acre.

b/ C - Contact injury; D - Defoliation.

c/ 0 - No effect; 1 - 0 to 25%; 2 - 26 to 50%; 3 - 51 to 75%; 4 - 76 to 100%.

during August and September 1955 on the forested areas of Fort Ritchie, Maryland. Various concentrations and rates of endothal (Pennsalt formulation S4069 containing 6.3% active ingredient) were applied as a spray to selected branches of the various species on 9 and 11 August, using the battery-operated sprayer previously employed in the Camp Detrick field trials. Periodic observations were made until 22 September. The treatments are summarized in Table IV.

Differences between results of treatments were small and somewhat variable. In general, the higher rates of application were more effective. Species may be grouped according to their responses into three categories in descending order of defoliability (Table VI). Poplar, cherries (Figure 1), elm, basswood, maples, locust and birch were readily defoliated, and dogwood, hickory, willow, hop-hornbeam, ash, tuliptree and beech were fairly well defoliated. Oaks were extremely resistant to abscission, but they were readily desiccated (Figure 2). All four of the shrub species treated responded about equally, falling at the end of the Group II species. Only one species, willow, sprouted new leaves during the period of observation. Regarding the amount of time elapsing until the first response occurred, three days after treatment there was some abscission on 11 species, and 75 to 100 percent on five others. After eight days, 20 species were 75 to 100 percent defoliated.

V. COMPARISON OF TWO DEFOLIANTS AT FORT RITCHIE, MARYLAND

To compare the relative efficacy of two agents which previous trials showed to be most promising as potential defoliants, a test was conducted at Fort Ritchie, Maryland, during September and October 1955. Endothal (disodium 3,6-endoxohexahydrophthalate, Pennsalt formulation S4069 containing 6.3 percent active ingredient) and 2-butyne-1,4-diol (Pennsalt formulation NP1098 containing 35 percent active ingredient) were sprayed as in the previous test at Fort Ritchie on selected branches of a number of species on 28 September. Visual observations of the responses were continued until 14 October. Treatments are shown in Table V.

Differences between results of application of a given compound were small and variable, but, in general, the higher concentration was the most effective. Both compounds gave essentially the same results, allowing the species to be grouped again in approximate descending order of defoliability (Table VII).

The time interval between the treatment applied in the earlier experiment and the present application with endothal allows for a comparison of variation in species response with maturity of leaves. In the current test, endothal caused greater defoliation than previously in some species (sweet birch, American ash, silver maple, tuliptree and sassafras), less defoliation in several others (beech, dogwood and spice-

TABLE IV. SUMMARY OF TREATMENTS AT FORT RITCHIE, MARYLAND WITH ENDOTHAL

Percent of Formulation[a]	Pounds active ingredient per acre	Volume	
		gal/acre	ml/1.9 sq. ft.
10.0	0.5	9	1.5
20.0	1.0	9	1.5
40.0	2.0	9	1.5
5.0	0.5	18	3.0
10.0	1.0	18	3.0
20.0	2.0	18	3.0
2.5	0.5	36	6.0
5.0	1.0	36	6.0
10.0	2.0	36	6.0

a/ Aqueous solution with 0.5% Tween-20.

TABLE V. SUMMARY OF TREATMENTS WITH ENDOTHAL AND BUTYNEDIOL AT FORT RITCHIE, MARYLAND

Compound	Concentration, (%)[a]	Active ingredient/acre, lb	gal/acre	ml/1.9 sq. ft.
Endothal	5	0.5	18	3.0
Endothal	20	2.0	18	3.0
Butynediol	5	2.6	18	3.0
Butynediol	20	10.4	18	3.0

a/ Aqueous solution with 0.5% Tween-20

TABLE VI. SUMMARY OF THE NUMBER OF TREATMENTS (OF NINE) WITH ENDOTHAL PRODUCING THE RESPONSES INDICATED WITHIN 7 TO 9 DAYS AFTER TREATMENT ON EACH SPECIES.

The Species are Listed in Decreasing Order of Defoliability.

Species	Percent Defoliation				Percent Contact Injury			
	to 25	25-50	50-75	75-100	to 25	25-50	50-75	75-100
GROUP I (readily defoliated)								
Ulmus americana (elm)				9[a]				
Robina pseudo-acacia (locust)				9				
Tilia americana (basswood)			1	8				
Populus tremuloides (poplar)			1	8				
Acer rubrum (maple)				8	1			
Acer saccharinum (maple)			2	7				
Prunus serotina (cherry)			2	7				
Prunus susquehanae (cherry)			2	7				
Fraxinus pennsylvanica (ash)	1	1		7				
Betula lenta (birch)		1	2	6				
GROUP II (Fair defoliation)								
Cornus florida (dogwood)	2	3		4				
Carya ovalis (hickory)	3	1	2	3				
Salix nigra (willow)	2		3	2				
Ostrya virginiana (hop-hornbeam)	1	1	6	1				
Carya glabra (hickory)		5	1	3				
Fraxinus americana (ash)	1	2		3				
Liriodendron tulipifera (tuliptree)	4	1	3	1				
Fagus grandifolia (beech)	2	3	4					
Populus grandidentata (poplar)	2	4		2				
Rhus typhina (sumac)	2	4	3					
Sassafras albidum (sassafras)	5	3	1					
Lindera benzoin (spicebush)	2	2	1		4			
Hammamelis virginiana (witch-hazel)	2	1	1		1	2	2	
GROUP III (poor defoliation)								
Quercus velutina (black oak)	2			1	3	3		
Quercus rubra (red oak)						2	5	1
Quercus alba (white oak)						5	4	
Quercus prinus (chestnut oak)						1	2	6
Carya ovata (hickory)								9

a/ For brevity, the total number of treatments that produced the response are recorded. In general, the higher rates of application were more effective.

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Figure 1. Response of Poplar (Populus tremuloides) and Cherry (Prunus serotina to Endothal. The treatments were distal to the tags.

Figure 2. Response of Chestnut Oak (Quercus prinus) to Endothal. The treatments were distal to the tag.

TABLE VII. COMPARISON (TWO TREATMENTS) OF THE RELATIVE EFFICACY OF ENDOTHAL AND BUTYNEDIOL AS DEFOLIANTS EIGHT DAYS AFTER TREATMENT

The Species are Listed in Approximate Descending Order of Defoliability

Species	Percent Defoliation and Contact Injury[a]							
	Endothal				Butynediol			
	to 25	25-50	50-75	75-100	to 25	25-50	50-75	75-100
GROUP I (readily defoliated)								
Betula lenta (birch)				C[b]				C
Tilia americana (basswood)				C				C
Fraxinus americana (ash)				C			C	
Acer saccharinum (maple)				C		A		B
Fraxinus pennsylvanica (ash)			B	A				C
Liriodendron tulipifera (tuliptree)			B	A		C		
Sassafras albidum (sassafras)	A			B				C
GROUP II (fair defoliation)								
Ostrya virginiana (hop-hornbeam)	A			B	A	B		
Lindera benzoin (spicebush)	C				C			
Fagus grandifolia (beech)	C					A		B
Carya ovata (hickory)				C	B			A
Hammamelis virginiana (witch-hazel)	A, A				C			
GROUP III (poor defoliation)								
Cornus florida (dogwood)	A	B					A	B
Quercus rubra (red oak)	A	B			A	B		
Quercus prinus (chestnut oak)			C					C
Quercus alba (white oak)				C				C

a/ Underlined letters represent defoliation.
b/ Concentrations are designated as follows:
A - 5% solution
B - 20% solution
C - Both 5% and 20% solution

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bush), and about the same degree in still others (basswood, green ash, hop-hornbeam and witch-hazel). This compound gave the same degree of contact response in shagbark hickory and chestnut oak, more contact injury in white oak, and less in red oak.

Butynediol was as effective as endothal on most of the species treated, and considerably more effective on at least one species of oak. At 12 days following treatment, chestnut oak and white oak were denuded by both compounds, and red oak was defoliated 50 to 75 percent by butynediol at the high application rate.

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