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TECHNICAL REPORT
BWL 16

DEFOLIATION AND DESICCATION

William H. Preston
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July 1959

U.S. ARMY CHEMICAL CORPS
BIOLOGICAL WARFARE LABORATORIES
FORT DETRICK

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U. S. ARMY CHEMICAL CORPS RESEARCH AND DEVELOPMENT COMMAND
U. S. ARMY BIOLOGICAL WARFARE LABORATORIES
Fort Detrick, Frederick, Maryland

BWL Technical Report 16

DEFOLIATION AND DESICCATION

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Crops Division
DIRECTOR OF BIOLOGICAL RESEARCH

Project 4-11-01-004 July 1959

FOREWORD

Defoliation and desiccation experiments were conducted during 1956 and 1957 under Project Number 4-11-01-004.

Estimates of area coverage and of casualty effects in this report do not represent the official Chemical Corps position at this time. Rather, they will serve as the basis for subsequent approved estimates prepared and issued by the Office of the Chief Chemical Officer.

ABSTRACT

In 1956 and 1957, 577 chemicals were screened for the best available defoliants, desiccants, and vegetation control agents. Environmental conditions, spray techniques, and formulations which increase the effectiveness of defoliants and desiccants are discussed. 2-Butyne-1, 4-diol and tributyl phosphate were the most effective defoliant and desiccant of those tested.

DIGEST

Five hundred and seventy-seven chemicals selected from data of previous tri~~als were sprayed on greenhouse test~~ plants during a screening program for defoliants, desiccants, and vegetation controls. Eighty-nine caused 20 ~~per cent~~ or more defoliation or injury on at least one plant species.

Tests of spray coverage, droplet size, and droplet placement revealed that (a) droplets that cover an entire leaf will defoliate that leaf, but good coverage on the proximal half is often sufficient; (b) defoliants sprayed on the undersurfaces of leaves cause more defoliation than when sprayed on the upper surfaces; (c) a spray of small droplets is more effective than one of large droplets; (d) drops placed on the midpoint of bean leaf petioles defoliated only the leaf blade attached to the treated petiole; and (e) droplets placed on stems of woody plants defoliated all leaves terminal to the point of application.

In several field tests, butynediol, Endothal, Folex, and P, P-dibutyl-N, N-diisopropyl phosphinic amide (a) defoliated branches of 31 different woody species in 6, 7.3, 8.1, and 8.8 days, respectively, and (b) defoliated entire trees. Each time butynediol caused the most rapid defoliation; Endothal was the next most effective.

The influence of temperature and humidity upon the effectiveness of butynediol on woody plants was studied under controlled environmental conditions. In general, the combination of a continuous moderate temperature and high humidity induced the fastest rate of defoliation and a continuous low temperature and high humidity caused the least defoliation.

Forty-four organic phosphates, phosphinic amides, oil soluble surfactants, and solvent oils were tested during a secondary screening program, and the chemicals with a butyl-phosphorus linkage were the most effective desiccants. Tributyl phosphate, butyl dibutyl phosphonic acid, dibutyl butyl phosphinic acid, and P, P-dibutyl-N, N-diisopropyl phosphinic amide caused severe desiccation in the shortest time. Desiccant activity was high in other materials, but it was slower in action.

Studies of vegetation control were carried out previously with formulations similar to VKL. Complete kills or nearly complete control of growth for at least two to three months were achieved in both greenhouse and field tests with several compounds.

Compounds showing the highest conifer defoliation and desiccation activity were pentachlorophenol, tributyl phosphate, 2,3,5,6-tetrachlorobenzoic acid, 2,3,6-trichlorobenzoic acid, and commercial formulations containing these compounds. The greatest amount of injury from all treatments occurred during the warmer months.

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I. INTRODUCTION

In 1956 and 1957 defoliation and desiccation experiments were carried out at Fort Detrick and Camp Ritchie, Maryland to (a) select and develop chemicals capable of defoliating or desiccating woody plants within the conditions and time requirements established by the Continental Army Command and (b) facilitate future research on defoliation by providing data on experimental techniques.

In the experiments, plants and environmental conditions representing various parts of the world were utilized to provide a better understanding of plant response to defoliants and desiccants under a wide variety of conditions.

II. STUDIES WITH HERBACEOUS PLANTS AND DECIDUOUS WOODY PLANTS

A. SCREENING OF CHEMICALS

1. Primary Screening

a. Compounds

Five hundred and seventy-seven compounds used in primary screening for defoliants were selected from a group which (a) exhibited certain attributes in the primary anticrop screening tests, and (b) were structurally similar to known defoliants and desiccants, or were considered potentially active compounds.

b. Techniques

Seventy-seven and one-half milligrams of each compound were weighed on a Roller-Smith balance and dissolved or dispersed in five milliliters of a suitable solvent containing 0.5 per cent Tween 20, a surfactant. In order of preference, the following solvents were used: distilled water, ethyl alcohol, acetone, ethyl cellosolve, and mixtures of these solvents. Relatively insoluble compounds were sprayed as suspensions. Using a glass atomizer or a Devilbiss spray nozzle No. 275, the compounds were sprayed at five pounds per acre on four test plants which were selected from the greenhouse and arranged within an area of one-sixth square yard. Control plants were included with each group of test plants, and were sprayed with distilled water containing 0.5 per cent Tween 20; then all plants were returned to the greenhouse.

c. Plant Materials

Two Black Valentine bean plants, one *Ligustrum amurense* (Amur River south privet), one *Ulmus chinensis* (Chinese elm), and one *Pereskia aculeata* (Pereskia included in later tests only) were treated. The bean plants, grown in one-quart plastic containers, were 10 to 12 days old and had one fully expanded trifoliate leaf when they were treated. The woody species, planted in quarter-gallon glazed crocks, varied in age, but they were chosen for uniform size and for mature and immature leaves.

d. Method of Recording Data

Data on plant response were recorded on cards. Observations were made 24, 48, and 72 hours, one, two, and three weeks after treatment. Data recorded at the above intervals were grouped into three classes: (a) per cent injury (including contact injury, necrosis, and desiccation); (b) per cent abscission; and (c) other effects. The latter class included chlorosis (Ch), curvature (E), leaf curvature (Le), formative effects (F), killing (K), stunting (S), terminal bud killing (TK), wilting (W), and galling (G). Numerical ratings of one to four were assigned to each of the above lettered responses to denote the degree of severity: (1) very slight, (2) slight, (3) moderate, and (4) severe. A rating of S-3 indicates moderate stunting. A second rating was used to denote defoliation as follows:

0. No defoliation
1. 1 to 19 per cent
2. 20 to 49 per cent
3. 50 to 74 per cent
4. 75 to 100 per cent

e. Results of Tests

Eighty-nine of the 577 compounds screened caused 20 per cent or more injury and/or defoliation on at least one of the three woody species used as test plants. The 89 compounds are listed in Tables I and II. Several of these active compounds were tested in secondary screening trials.

2. Micro-Screening

When insufficient chemical was available for spraying, it was mixed with lanolin and four per cent Tween 20 and applied as a smear to stems or leaves of Black Valentine beans, privet, and elm. Eighty-two chemicals were tested in this manner.

Endothal and butynediol used as standards caused defoliation, but 72 compounds tested on bean and 10 tested on elm and privet caused no defoliation.

TABLE I. COMPOUNDS ACTIVE IN PRIMARY SCREENING TESTS CAUSING 20 PER CENT OR MORE DEFOLIATION

NUMBER	NAME
380	acetamide, 2,4,5-trichlorophenoxy-
4474	acetic acid, 2,4-dichloro-5-fluorophenoxy-n-butyl ester
4501	acetamide, 2-fluoro-4,6-dichlorophenoxy-
4505	acetamide, 2,4-dinitro-6-fluorophenoxy-
4513	acetic acid, 2,4-dichloro-5-nitrophenoxy-
4587	acetic acid, 2-chloro-4-fluorophenoxy-amyl ester
4588	acetic acid, 2-chloro-4-fluorophenoxy-2-chloroethyl ester
4589	acetic acid, 2-chloro-4-fluorophenoxy-2-(2-hydroxyethoxy)-, ethyl ester
4590	acetic acid, 2-chloro-4-fluorophenoxy-tetra hydrofurfuryl ester
4599	acetic acid, 2,4-dichloro-5-fluorophenoxy-n-propyl ester
4604	acetic acid, 2,3,6-trichlorophenyl
10542	thiocyanic acid, 4-hydroxy-3-tolyl ester
10544	thiocyanic acid, 3-ethyl-4-hydroxyphenyl ester
12542	phenol, 2-fluoro-4,6-dinitro-
12640	phenol, 2,6-dinitro-4-fluoro-
12642	phenol, 2,4-dinitro-5-fluoro-
12645	xanthate, sodium ethyl-
12897	acetic acid, 2,4-dichlorophenoxy-,2,4-dichlorophenoxy ethyl ester
12898	acetic acid, 2-methyl-r-chlorophenoxy-,2,4-dichlorophenoxy ethyl ester
12901	α-propionic acid, 2,4-dichlorophenoxy-,2,4-dichlorophenoxy ethyl ester
12902	α-propionic acid, 2,4,5-trichlorophenoxy-, 2,4-dichloro-phenoxy ethyl ester
12906	L-asparagine, N-α-(2,4,5-trichlorophenoxy-α-propionyl)-
12961	s-triazine, 2-chloro-4,6-bis(diethylamine)-
12965	propionic acid, 2,2,3-trichloro-, sodium salt
12976	benzoic acid, 2,3,5,6-tetrachloro-
13033	propionic acid, 3-bromo-2-bromoethyl ester
13146	benzene, 1,2,3,5-tetramethoxy-
13865	1-naphthol, 2,4-dinitro-
14141	quinolinium iodide, 1-methyl-
14154	pseudourea, 2-decyl-2-thio-, hydriodide
14160	pseudourea, 1,3-diethyl-2-tetradecyl-2-thio-, hydriodide
14434	phenol, 2-cyclohexyl-4,6-dinitro-
14808	1,2,4-triazole, 3-acetalamino-
14814	pentachlorophenol, 1,2,4-aminotriazole salt
50076	perdikeflin
50348	pyrocatechol, 4 (?) -chloro-

TABLE II. COMPOUNDS ACTIVE IN PRIMARY SCREENING TESTS CAUSING 20 PER CENT OR MORE DESICCATION BUT LESS THAN 20 PER CENT DEFOLIATION

NUMBER	NAME	NUMBER	NAME
516C	arsonic acid, p-hydroxyphenyl-	12978	acetic acid, 2,4,5-trichlorophenoxy-, 2-hydroxy-3-
1624	benzoic acid, 2-hydroxy-5-amino-	13030	hexanoic acid, 2-chloroethyl ester
4392	s-2,3'-dibenzofurna-3(2H), 2'(3'H)dione, 5,5',7,7-tetrachloro-	13115	phthalic anhydride, 3,6-endoxohexahydro-
4496	acetamide, 2,4-dichloro-5-fluorophenoxy-	13721	pentachlorophenol, sodium salt, monohydrate
4497	acetic acid, 2,4-dichloro-5-fluorophenoxy-, beta-chloroethyl ester	13851	chalcone, α-bromo
4555	anthranilic acid, 4-iodo-	13899	ammonium iodide, (6-hydroxythymyl)-trimethyl-
4573	benzoic acid, 3-bromo-2,6-dichloro-	13925	pyrimidine, 1-butyl-2-hendecyl-1,4,5,6-tetrahydro-
4578	benzoic acid, 3,6-dibromo-2-chloro-	13950	pseudourea, 2-decyl-1,3-diethyl-2-thio, hydrobromide
4584	benzoic acid, 2,3,4,6-tetrachloro-	13952	psuedourea, 2-dodecyl-1,3-diethyl-2-thio, hydrobromide
4585	acetic acid, 2-chloro-4-fluorophenoxy-, isopropyl ester	13953	pseudourea, 2-dodecyl-1,3-diethyl-2-thio-, hydriodide
12647	phenol, 2-tert-butyl-4,6-dinitro-	14039	coumarilamide, N,N-diethyl-
12740	phenol, 2-fluoro-4-nitro-	14088	quinoline, 8-nitro-
12783	arsine, tri-p-tolyl-	14170	phenol, 2-chloro-4,6-dinitro-
12867	benzene, 1,2,4-trichloro-3,5-dinitro-	14175	aniline, 2-chloro-4-nitro-
12870	carbanilic acid, m-chloro-, 2-propynyl ester	14494	febrifugine, dihydrochloride-
12903	L-asparagine, N-α-(2,4-dichlorophenoxy-α-	14553	phenol, 2-amyl-4 (?)-chloro-
12905	D-asparagine, N-α-(2,4,5-trichlorophenoxy-α-	14584	benzene, 1-fluoro-3-chloro-4,6-dinitro-
12913	DL-leucine, N-(2,4-dichlorophenoxy-α-propionyl)-	14585	benzene, 1,3-difluoro-4,6-dinitro-
12917	L-alanine, N-(2,4-dichlorophenoxy-α-propionyl)-	14587	benzene, 1-fluoro-3-bromo-4,6-dinitro-
12920	L-methionine N-(2,4-dichlorophenoxy-α-propionyl)-	14803	1,2,4-trizaole, 3-salicylideneamino-
12922	L-threonine, N-(2,4-dichlorophenoxy-α-propionyl)-	14850	phosphinic acid, bis (2-ethylhexyl)-, ethyl ester
12936	benzoic acid N-(2,4-dichlorophenoxy-α-propionyl)-	14900	phosphinic acid, 2,5-dibromophenyl-, diethyl ester
12943	salicylic acid, N-(2,4,5-trichlorophenoxyacetyl)-p-amino	50093	1-dodecanol, 2-diethylamino-1-phenylhydrochloride
12945	α-propionic acid, 2,4-dichlorophenoxy-, hydronopyl ester	50180	6,9-tetradecanediamine, N'N'N'N', 2,2,4,11,13-13-decamethyl-
12947	acetic acid, 4-chlorophenoxy-, hydronopyl ester	50181	s-triazine, 1,3,5-tridodecylhexahydro-
12948	acetic acid, 2,4,5-trichlorophenoxy-, 2-hydroxy-3-	50352	o-cresol, α-dimethylamino-4-(1,1,3,3-tetraethyl-butyl)-

3. Secondary Screening

a. Miscellaneous Compounds

Thirty-two compounds were selected from the 89 showing activity in primary screening tests. Four hundred and sixty-four milligrams of each chemical were dissolved in ten milliliters of a suitable solvent. Sprays were applied at ten pounds per acre over test plants arranged in an area of one-half square yard. Seven woody species were treated: Ulmus chinensis (Chinese elm), Ligustrum amurense (Amur River south privet), Robinia Pseudo-Acacia (black locust), Prunus Lauroceraceous (laurel-cherry), Pinus sylvestris (Scotch pine), Tsuga canadensis (Canadian hemlock), and Picea abies (Norway spruce). Nine of the 32 compounds tested, (Table III) caused more than 50 per cent defoliation and/or injury on at least three species.

TABLE III. COMPOUNDS IN SECONDARY SCREENING TESTS ACTIVE AS DEFOLIANTS AND DESICCANTS

NUMBER	NAME
380	acetamide, 2,4,5-trichlorophenoxy-
10542	thiocyanic acid, 4-hydroxy-3-tolyl ester
10544	thiocyanic acid, 3-ethyl-r-hydroxyphenyl ester
12542	phenol, 2-fluoro-4,6-dinitro-
12640	phenol, 2,6-dinitro-4-fluoro-
12642	phenol, 2,4-dinitro-5-fluoro-
12645	xanthate, sodium ethyl-
12965	propionic acid, 2,2,3-trichloro-, sodium salt
50076	perdikeflin

b. Phosphinic Amides

Nine phosphinic amides received from the Shell Development Company were dissolved in Deobase oil or ethyl cellosolve and were sprayed on three plant species. Desiccation results given in Table IV were compared with results caused by P, P-dibutyl-N, N-diisopropyl-phosphinic amide. Practically no defoliation occurred on plants in any of the treatments.

TABLE IV. DESICCANT ACTIVITY OF PHOSPHINIC AMIDES ON HERBACEOUS AND WOODY PLANTS FIVE DAYS AFTER TREATMENT

COMPOUND		PER CENT INJURY								
Number	Name	Bean			Privet			Elm		
		lb/acre			lb/acre			lb/acre		
		20	2	0.2	20	2	0.2	20	2	0.2
14924	N-methyl-N-phenyl-P,P-dibutyl-	100	100	10	100	15	5	100	70	0
14925	N,N-dimethyl-P,P-dibutyl-	100	100	5	75	15	0	95	60	0
14926	N,N-diethyl-P,P-dibutyl-	100	100	85	100	15	2	100	90	30
14927	N,N-diisopropyl-P,P-dioctyl-	100	100	70	20	50	0	20	5	10
14928[a]	N,N-diethyl-P,P-dihexyl-	100	100	100	100	30	10	100	95	5
14929	N,N-diisopropyl-P,P-diamyl-	100	100	65	100	0	0	100	25	0
14930[a]	N,N-dimethyl-P,P-dihexyl-	100	100	100	100	70	0	100	95	0
14931	N,N-diethyl-P,P-diamyl-	100	100	50	100	20	5	100	60	5
14932	N,N-diisopropyl-P,P-diisobutyl-	-	50	0	-	0	0	-	15	0
13539	N,N-diisopropyl-P,P-dibutyl-	-	100	100	-	40	25	-	98	50

a. Essentially equal to 13539 in desiccant activity.

c. Organic Phosphates

Eight organic phosphates related to tributyl phosphate (12063) were dissolved in Deobase oil, water, or ethyl cellosolve and sprayed on beans, elm, and privet at 1.2, 2.5, 5.0, and 10.0 pounds per acre. The compounds used were: triethyl phosphate, triphenyl phosphate, tricresyl phosphate, tri-m-tolyl phosphate, tri-o-tolyl phosphate, phenyl disodium phosphate, di decyl phosphate, and didodecyl phosphate.

None of the compounds approached the desiccation activity of tributyl phosphate.

d. Oil-Soluble Surfactants

Eleven oil-soluble surfactants were sprayed at concentrations of 1.5 and 8 per cent on beans and privet. The chemicals used were: Ethomeen 5/20, Ethomeen T/15, Blendene, Armeen 2C, Mulson 3CW, Duponal, Alkaterge A, Alkaterge C, Alkaterge E, Triton GR7, and Triton X-45.

Surfactants Armeen 2C, Ethomeen T/15, Triton GR7, and Triton X-45 severely injured bean plants but only Ethomeen T/15 caused significant damage to privet.

e. Solvent Oils

Sixteen solvent oils were sprayed on plants to test for desiccant activity. Results of this test are given in Table V.

f. Butynediol-Type Compounds

Compounds closely related to 2-butyne-1,3-diol were sprayed on Pereskia, locust, privet, philedendron, and elm, (woody species in the greenhouse) to determine defoliant activity. Five milliliters of 2.5, 10, and 40 per cent aqueous solutions of each of the following chemicals were sprayed on groups of these species placed in an area of one-third square yard:

2,4-Hexadiyne-1,6-diol
3-Hexyne-2,5-diol,2,5-dimethyl-
3-Hexyne-2,5-diol,2,5-diphenyl-
4-Octyne-3,6-diol,3,6-diisopropyl-2,7-dimethyl-
8-Hexadecyne-7,10-diol,7,10-dimethyl-
9-Octadecyne-8,11-diol,8,11-dimethyl-
4-Octyne-3,6-diol,3,6-dimethyl
2-Butyne-1,4-diol

Defoliation in this experiment was comparatively low, even for butynediol. Only one chemical, 2,4-hexadiyne-1,6-diol, showed activity which approached that of butynediol. This experiment should be repeated for more conclusive results.

TABLE V. DESICCANT ACTIVITY OF SOLVENT OILS AFTER SPRAYING ON HERBACEOUS AND WOODY SPECIES

SOLVENT OIL 100 lb/acre	PER CENT INJURY TWO DAYS AFTER TREATING		
	Bean	Privet	Elm
Amsco Solvent HCC	95	70	70
Amsco Solvent A80	50	5	5
Amsco Ink Oil 10-550	0	0	0
Amsco Solvent RT	95	5	75
Skellysolve	0	0	0
Richsol	60	2	2
Velsicol-AR60	100	50	5
Socony-Vacuum Sovacide 544B	98	50	90
Socony-Vacuum Agronyl B	30	5	0
W9308 Shell Aromatic Solvent 54	100	15	90
W8310 Shell Solvent TS-28	65	0	0
Esso Heavy Aromatic Naphtha Formula 132	98	3	2
Varsol	65	0	0
Sohio Insecticide Base Oil 30	0	0	0
Sohio Insecticide Base Oil 130	15	0	0
Shell Dispersol	0	0	0

Two of the solvent oils caused severe injury (Amsco Solvent HCC and Socony Vacuum Sovacide 544B); four caused moderate injury; six caused slight injury; and four were inactive (Amsco Ink Oil 10-550, Skellysolve, Sohio Insecticide Base Oil 30, and Shell Dispersol).

B. CONCENTRATION-RANGE TESTS

1. Desiccants

Butyl dibutyl phosphinate (BDP), dibutyl butyl phosphinate (DBP), and ammonium sulfamate were tested at eleven concentrations ranging from 0.04 to 80 pounds per acre on seven woody plants. Both phosphorus materials induced rapid and similar desiccation at five to eighty pounds per acre; responses were similar to those induced by sprays with tributyl phosphate. The least responsive plant to treatments with phosphorus compounds was philodendron (representing some tropical vegetation), and the most responsive were elm and locust. Ammonium sulfamate caused severe desiccation at the highest rate and then only on five of the seven woody plants.

2. Defoliants

Five plant species were sprayed with butyl phosphorotrithioite (14796), formulated diphenyl chlorothiophosphate (B-1613), and ammonium thiocyanate at 0.15 to 40 pounds per acre. The above chemicals, listed in order of decreasing ability to induce defoliation, were moderately to highly active on at least one species at forty pounds per acre. However, the most active chemical, magnesium chlorate, sprayed at 0.15 to 20 pounds per acre defoliated 50 to 95 per cent of all species at 10 and 20 pounds per acre.

Ammonium thiocyanate caused moderate to severe desiccation on all plants within one day. However, this action was relatively slow in comparison to that of tributyl phosphate, BDP, DBP, and pentachlorophenol.

C. DROPLET AND COVERAGE STUDIES WITH DEFOLIANTS

1. Translocation from Droplet Applications

A test was devised to determine if defoliants were translocated within plants by applying 0.005-milliliter droplets from a calibrated volumetric micro-pipette (ACUP) to leaves, petioles, and stems of plants.

In the first series of tests 3,6-endoxohexahydro-phathalic acid (Endothal) was applied to Black Valentine bean plants at 34 different combinations of application points involving petioles, pulvini, primary leaves, and trifoliate leaves. Concentrations used per application point were 0.3 milligram of formulated Endothal and five micrograms of pure Endothal dissolved in an aqueous solution containing 0.05 per cent Tween 20. When droplets were applied (a) to the mid-points of petioles, the leaf blades defoliated; (b) to stems, the stems were injured and some of the leaf blades defoliated above the point of application; (c) to leaflets or primary leaves, only those leaflets or primary leaves defoliated. The defoliation activity of Endothal appeared to be exerted terminal to the point of application; in no instances were there indications of downward movement of the compound through stems or out of leaves or petioles into stems.

In a second series of treatments, 0.005-milliliter droplets of commercial formulations of Endothal, butynediol (NP1098), and sodium chlorate (Shed-a-leaf) were applied to leaf axils (involving petiole base, axillary bud, and adjacent stem) of elm, privet, and locust trees. When leaf axils on lower portions of branches were treated, adjacent leaf blades and all leaves terminal to the point of application were injured by all three chemicals, but Endothal and butynediol subsequently defoliated the terminal leaves. When leaf axils on the terminal portions of branches were treated, the lower, untreated parts were not affected. When droplets were applied to leaf blades, just the leaves that were treated defoliated regardless of the position on the branch.

Results of this series of treatments confirmed the previous results. Defoliants are translocated only upward in stems and move from leaves into the stem and upper foliage.

2. Location and Number of Droplets on Leaves

Droplets of three chemicals were placed on primary leaves of bean plants to determine the number and location of droplets required to induce defoliation (Table VI).

TABLE VI. DEFOLIATION OF PRIMARY LEAVES OF BLACK VALENTINE BEANS BY CHEMICALS

TREATMENT		PER CENT DEFOLIATION		
0.002 ml/droplet		Days After Treatment		
		10	12	14
Drops	Location on Leaf	Endothal	Butynediol	Tributyl Phosphate
3	Random	0	0	0
3	Near Base[a]	0	0	0
5	Random	0	5	40
5	Near Base	0	5	40
10	Random	25	30	100
10	Near Base	100	30	-
20	Random	100	70	100
30	Random	100	100	-

a. Droplets placed on leaf blade in an arc one-half inch from base.

Diameters of Endothal droplets were measured on leaves and 30 droplets covered approximately 1.3 per cent of the area of an average mature primary leaf.

Complete defoliation occurred only in treatments where ten or more droplets were used; translocation of three to five droplets, even when placed proximally on the leaf, was not sufficient to induce defoliation. Rapid, severe injury occurred on all leaves treated with tributyl phosphate.

3. Coverage Required Per Leaf

Butynediol (full strength formulated NP 1098) was applied to various sectors of leaves on young elm trees to determine the type and coverage required to defoliate the leaf. Droplets of 0.005 milliliter were applied to leaves and smeared over the selected areas with a glass rod. Ten leaves on each of ten plants were treated similarly. Defoliation responses recorded 10 days after treatment are given in Table VII.

TABLE VII. EFFECT OF AREA COVERAGE UPON DEFOLIATION OF ELM LEAVES TREATED WITH DROPLETS OF BUTYNEDIOL

AREA TREATED	PER CENT DEFOLIATION
Proximal Half	100
Distal Half	30
Proximal Quarter	60
Distal Quarter	0
Lateral Quarter	50
Along Midvein	30
Distal Lateral Quarter	10
Proximal Lateral Quarter	60
Center (Droplet)	40
Entire Leaf	100

Subsequent tests were conducted on Black Valentine bean leaves by applying several rates of Endothal and butynediol to the same areas as those listed in Table VII. The results were essentially the same as those obtained in the first experiment.

4. Application to Upper Versus Lower Leaf Surface

Sprays of butynediol on either the upper or lower leaf surfaces of six species were compared for efficiency in defoliating these species. Six-milliliters of 35 per cent formulated butynediol at full-, half-, or quarter-strength were sprayed on plants placed in an area of two-thirds square yard. Results given in Table VIII indicate that, in general, butynediol is distinctly more effective when applied to the lower leaf surfaces of the species treated.

TABLE VIII. PLANTS DEFOLIATED BY SPRAYING LEAVES WITH BUTYNEDIOL

LEAF SURFACE	BUTYNE-DIOL, Per cent	PER CENT DEFOLIATION					
		Pereskia	Pomegranate	Elm	Locust	Oak	Privet
Upper	100	36	33	45	95	50	15
	50	40	5	15	90	0	20
	25	23	15	3	90	0	10
Lower	100	52	95	70	90	13	90
	50	40	80	99	85	40	95
	25	3	90	70	90	18	25

The conclusions of the above experiment have been substantiated by experiments reported by W. A. Brun and H. J. Cruzado from the Federal Experiment Station in Puerto Rico. The two chemicals, butynediol and Shed-a-leaf (Formulation of 18.2 per cent sodium chlorate and 10.0 per cent sodium pentaborate) were distinctly more effective when applied to the lower leaf surfaces versus applications to the upper leaf surfaces. Stomates of the species treated were located on lower surfaces of leaves only and were open at the time of treatment. These results indicated that the defoliants entered the leaves most effectively through stomatal openings. Therefore, the effectiveness of a defoliant may decrease if applied under dry conditions or at times when the stomates are closed.

5. Effect of Droplet Size

Droplets of butynediol (35 per cent commercial formulation) were sprayed on individual Pereskia plants (greenhouse-grown) at 0.5 milliliter per one-third square yard with a droplet sizer apparatus developed at Fort Detrick.

Eight to twenty-four hours after treatment, droplets measuring 100 and 500 microns in diameter caused 95 and 50 per cent defoliation, respectively; droplets measuring 1,000 microns in diameter induced minimal defoliation.

D. FIELD STUDIES WITH DEFOLIANTS

1. Stage-of-Development Test

Four woody species, Quercus castanea (chestnut oak), Hamamelis virginiana (witch-hazel), Acer rubrum (red maple), and Betula lenta (sweet birch) were sprayed with full-, one-fourth-, and one-eighth-strength formulations of Endothal, butynediol, and sodium chlorate. One to three milliliters of solution were sprayed over branch areas of approximately one square yard when leaves of the plants were approximately half expanded and at two succeeding weekly intervals.

At the earliest stage, Endothal caused practically no defoliation, but butynediol and sodium chlorate defoliated chestnut oak and sweet birch trees, respectively. Sprays, especially butynediol, applied during the following two weeks resulted in good to excellent defoliation of all species; however, the rate of defoliation was slow. The trees did not defoliate until 14 days after they were sprayed.

2. Species Test

The following defoliants were sprayed on 31 different species of woody plants at Fort Ritchie and Fort Detrick, Maryland:

Number	Name
1626	3,6-endoxchexahydrophthalic acid (Endothal)
-	Sodium chlorate (Shed-a-leaf)
13539	P,P-dibutyl-N,N-diisopropyl-phosphinic amide
14580	bis (ethylxanthogen) trisulfide
-	Tributyl phosphorotrithioite (Folex)
12959	2-butyne-1,4-diol
-	Magnesium chlorate

Chemicals were applied at full-, half-, and quarter-strength with the exception of magnesium chlorate which was made up as a fully saturated solution in water (56.5 grams per 100 milliliters), and then diluted half- and quarter-strength. Bis (ethylxanthogen) trisulfide was temporarily emulsified in water only with the addition of 0.5 milliliter Tween 20, and liquid soap, respectively, per 150 milliliters of solution. A battery-powered sprayer equipped with a Devilbiss No. 275 nozzle disseminated chemicals at one milliliter per square foot over branches selected within an area of one to three feet. The areas were sprayed from 25 July through 15 August 1957. Progress of defoliation was observed 4, 5, 6, 7, and 15 days after treatment. Results are given in Table IX.

TABLE IX. DEFOLIATION OF WOODY SPECIES AFTER SPRAYING WITH CHEMICALS

SPECIES	NUMBER OF DAYS FOR 75 PER CENT DEFOLIATION						
	CD 1626	Folex	CD 13539	Butyne-diol	$NaClO_3$	$MgClO_3$	CD 14580
Acer rubrum (Red Maple)	6	6	9-15	6	-[a]	-	-
Acer saccharinum (Silver Maple)	4	4	5	-	5	7	8
Alnus sp. (American Alder)[b]	-	9-15	-	4	-	-	-
Amorpha fruticosa (False Indigo)[c]	4	5	5	6	7	6	5
Benzoin aestivale (Spicebush)	7	7	6	-	-	9-15	7
Betula lenta (Sweet Birch)[c]	4	5	6	5	7	9-15	6
Carya sp. (Hickory)[b]	9-15	9-15	9-15	-	-	9-15	-
Castanea dentata (American Chestnut)	7	-	9-15	7	7	-	-
Celtis occidentalis (Hackberry)	6	7	7	5	7	7	-
Cornus florida (White Flowering Dogwood)[b]	-	7	9-15	9-15	9-15	-	-
Corylus americana (Filbert or Hazelnut)	9-15	5	7	7	5	5	9-15
Fraxinus sp. (Ash)	9-15	9-15	-	8	9-15	9-15	-
Hamamelis virginiana (Witch-hazel)[b]	-	-	9-15	5	-	-	-
Liriodendron tuliperifera (Tulip Tree)	6	6	8	-	6	-	9-15
Malus (Apple Seedling)	6	6	-	7	-	-	-
Morus alba (White mulberry)	4	4	7	5	7	-	8
Nyssa sylvatica (Black Gum)	6	7	6	5	-	-	-
Prunus serotina (Black Cherry)[c]	4	4	4	4	4	5	4
Pyrus (Pear-Seedling)[b]	4	-	-	-	-	-	-
Quercus alba (White Oak)[b]	-	9-15	-	-	9-15	8	-
Quercus muhlenbergii (Chestnut Oak)[b]	9-15	9-15	-	7	9-15	-	-
Quercus velutina (Black Oak)[b]	9-15	9-15	-	-	9-15	9-15	-
Rhus sp. (Sumach)	9-15	-	6	7	9-15	7	-
Robinia Pseudo-Acacia (Black Locust)[c]	4	4	4	4	4	4	5
Rosa sp. (Rose)	4	4	8	4	-	9-15	4
Salix sp. (Willow)	4	5	-	4	-	-	-
Sambucus canadensis (American Elder)	5	8	9-15	-	6	6	-
Sassafras albidum (Sassafras)[b]	8	9-15	9-15	8	-	9-15	-
Tilia americana (American Linden)[c]	4	7	6	4	7	6	5
Ulmus fulva (Slippery Elm)	4	4	-	4	4	4	4
Viburnum dentatum (Arrow-wood Viburnum)	5	-	4	-	4	4	-
Average Days to Defoliate	7.3	8.1	8.8	6.0	8.5	9.2	7.2
Total Species Defoliated	27	26	22	22	20	19	12

a. More than 15 days.
b. Species most difficult to defoliate.
c. Species most easily defoliated.

Weather conditions for one month previous to the test and during the testing period were unusually dry. No rain fell during the experiment and many plants were in a semi-wilted condition when they were sprayed.

All compounds, with the exception of bis (ethylxanthogen) trisulfide, defoliated more than half the species tested. Endothal sprays defoliated the greatest number, but butynediol defoliated species more rapidly; an average of six days was recorded. Butynediol, Shed-a-leaf, and Folex formulations sprayed at 50 and 100 per cent concentrations defoliated 50 per cent more species than when sprayed at the 25 per cent concentration. The three rates of Endothal, magnesium chlorate, and bis (ethylxanthogen) trisulfide sprayed on species were not significantly different in general effectiveness.

Species most difficult to defoliate and most easily defoliated are given in Table IX. However, at least one chemical caused defoliation in less than 15 days of all of the above species with the exception of black oak and hickory. On species with compound leaves, for example, false indigo, black locust, and rose, Endothal generally defoliated the entire leaf, whereas butynediol defoliated leaflets first, followed by rachises and petioles sometime later.

3. Sprays on Entire Trees

During 26 to 30 August 1957, at both Fort Detrick and Fort Richie, trees and shrubs which were well developed and isolated somewhat from the surrounding vegetation were sprayed with chemicals. Eight species each were sprayed with butynediol and Endothal, seven with Folex, and one with magnesium chlorate. Chemicals and concentrations were selected on the basis of the results obtained from the field species tests, Section II, D, 2.

From the ground or a six-foot stepladder (if trees were over 12 feet high) very small droplets of agent were sprayed at 100 milliliters per minute from a five-gallon Hudson "Xpert" sprayer equipped with a six-foot spray rod extension and a Tee-jet nozzle automatically regulated at 50 pounds per square inch (psi). Good coverage was obtained without run-off except for seven plants that were again treated 8 to 13 days later. Estimates of per cent defoliation were made daily for the first 13 days after treatment, then later during a period between 18 and 23 days after treatment. Results are given in Table X and are illustrated in Figures 1 through 7.

As in the field species test, butynediol defoliated the most rapidly, followed by Endothal and Folex. All plants defoliated 75 per cent or more within three weeks except wild black cherry, white mulberry, hickory and white oak. The latter, sprayed with magnesium chlorate, defoliated only five per cent in three weeks.

TABLE X. DEFOLIATION OF TREES AND SHRUBS AFTER SPRAYING WITH THREE CHEMICALS

PLANT			DEFOLIANT				PER CENT DEFOLIATION								
							Days After Treatment								
Species	Height ft	Width ft	Compound	conc	Vol. ml	lb/acre[a]	3	5	7	9	11	13	16	19	22
Alnus sp. (Alder)	13	12	Butyne-diol	100%	400	50.5	0	20	80	80	85	85	85		90
Betula lenta (Sweet Birch)	15	9			317	48.3	0	0	5	95	98	98	98		98
Fraxinus sp. (Ash)	11.5	10			253	45.1	0	5	50	60	65	65		98	
Hamamelis virginiana (Witch-Hazel)	6.5	5			100	62.4	0	0	75	85	85	85	95		98
Celtis occidentalis (Hackberry)	9	9		50%	200	23.6	0	70	80	95	95	95	96		98
Salix sp. (Willow)	12.5	5			283	50.5	10	50				95			
Ailanthus altissima (Tree of Heaven)	14.5	9.5		25%	600	22.6	0	20	90	90	90	90	90		90
Ulmus fulva (Slippery Elm)	9	7.5			316	21.5	0	60	75	75	75	95	96		98
Liriodendron tulipifera (Tulip Tree)	13.5	9.5	Endothal	100%	605	17.2	0	30	65	80	90	90		90	
Nyssa sylvatica (Black Gum)	10	8.5			125	4.3	5	25	70	75	[illegible]	80		80	
Robinia Pseudo-Acacia (B. Locust)	19.5	11.5			852	13.8	30	85	90	95	95	[illegible]		97	
Acer saccharinum (Silver Maple)	14	8		50%	662	10.7	0	20	[illegible]	50	60	65		85	
Tilia americana (Am. Linden) or Basswood	14	11.5			337	3.8	5	40	90	90	90	90		95	
Benzoin aestivale (Spice Bush)	9.5	8		25%	267	3.1	0	5	10	15	55	55		75	
Prunus serotina (Wild Black Cherry)	12.5	10			667	4.8	15	55	65	65	65	65		65	
Sassafras albidum (Sassafras)	10	7			173	2.5	0	0	5	5	30	75		80	
Morus alba (White Mulberry)	15	19.5	Folex	100%	628	83.8	0	20	20	30	35	35			45
Prunus persica (Peach)	14	9			417	146.2	60	80	95	95	95	95	98		
Carya sp. (Hickory)	14.5	6		50%	108	27.9	0	0	1	5	5	10	15		25
Cornus florida (White Fl Dogwood)	7	5			59	36.5	0	0	5	15	40	75	75	97	
Juglans nigra (Black Walnut)	14	6			158	43.0	0	0	70	75	85	85	85		90
Acer rubrum (Red Maple)	15	6		25%	390	49.5	0	10	50	50	65	65		90	

a. Pure agent
Butynediol - 30%
Endothal - 6.3%
Folex - 7[illegible]%

FIGURE 1. ALDER BEFORE AND AFTER SPRAYING WITH BUTYNEDIOL. (FD NEGS C-31[illegible] AND C-31[illegible])

FIGURE 2. ASH BEFORE AND AFTER SPRAYING WITH BUTYNEDIOL. (FD NEGS. C-31-3 AND 31-4)

FIGURE 3. SWEET BIRCH BEFORE AND AFTER SPRAYING WITH BUTYNEDIOL. (FD NEGS C-3210 AND C-3211)

FIGURE 4. TULIP TREE BEFORE AND AFTER SPRAYING WITH ENDOTHAL. (FD NEGS C-31[illegible] AND C-31[illegible])

FIGURE 5. RED MAPLE BEFORE AND AFTER SPRAYING WITH FOLEX. (FD NEGS C-31[illegible] AND C-31[illegible])

FIGURE [illegible]. SASSAFRAS BEFORE AND AFTER SPRAYING WITH ENDOTHAL. (FD NEGS C-31-3 AND C-31-)

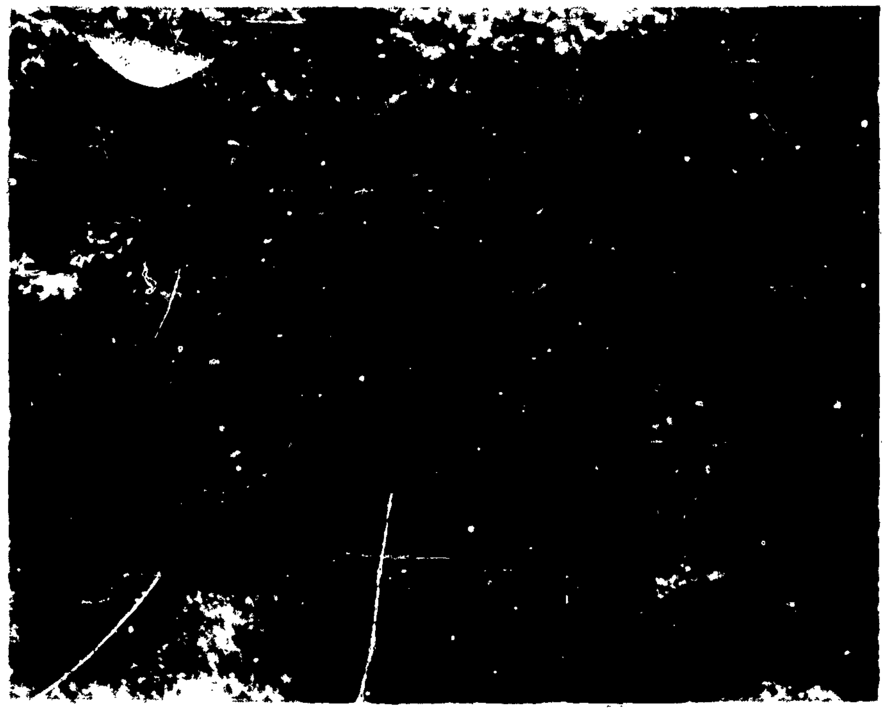

FIGURE . WITCH-HAZEL BEFORE AND AFTER SPRAYING WITH BOTYNEDICL. (FU NEGS C-31 - AN [illegible]

E. CONTROLLED ENVIRONMENTAL STUDY WITH BUTYNEDIOL

A controlled environmental study was initiated in October 1957 to determine the influence of temperature and humidity upon the effectiveness of butynediol. These tests were conducted because results of similar or identical field tests varied at different times of the year or under different meteorological conditions.

The following species were used in the experiment:

Species	Height, feet	Number Per Treatment
Acacia	1.0	5
Diospyros (Persimmon)	0.75	3
Hibiscus	2.0	6
Ligustrum (Privet)	0.75	6
Morus (Mulberry)	1.5	6
Pomegranate	1.5	12
Prunus laurocerasus (Cherry-Laurel)	1.0	6
Quercus (Oak)	0.75	6
Robinia (Locust)	1.0	6
Ulmus (Elm)	1.0	6

Prior to the experiment the plants were grown at sufficiently low temperatures so that active growth was retarded and all leaves were mature. The plants were placed in the environmental chambers seven days before treatment with butynediol to acclimate them.

All treated plants and the controls were exposed to a 16-hour day by artificially illuminating a chamber with a bank of 12 six-foot fluorescent tubes (100-watt daylight) placed 2.5 feet above the plants.

The treated plants were sprayed for four hours with 46.1 milliliters formulated butynediol per square yard. The high rate was necessary to obtain complete and uniform coverage of all leaves. Untreated plants were left in the environmental chambers to determine the effect of the environment *per se*.

Estimates of defoliation were recorded 24, 35, 48, 59, 72, 83, 96, 107, 144, 179, and 227 hours after spraying. The plants were rated in descending order of reaction. Results of the temperature-humidity combinations are given in Table XI. It was impossible to analyze conclusively all the effects of temperature, humidity, and their interactions; however, in general, the combination of a continuous moderate temperature and high humidity induced the fastest rate of defoliation and a continuous low temperature and high humidity caused the least amount of defoliation. Before a valid interpretation of effects of temperature, relative humidity, and their interactions is possible, the species in this experiment should be grouped according to similar defoliation responses (Table XII).

TABLE XI. RELATIVE EFFECT OF TEMPERATURE AND HUMIDITY BASED ON SPEED OF DEFOLIATION
50 Per Cent Defoliation Used as Criterion

Temperature		Humidity		SPECIES SPRAYED WITH 46.1 MILLILITERS BUTYNEDIOL PER SQUARE YARD										Rating Totals
Day	Night	Day	Night	Hibiscus	Acacia	Cherry Laurel	Elm	Locust	Mulberry	Oak	Persimmon	Pomegranate	Privet	
M	M	H	H	4	4	1	1	2	4	1	5	5	2	29
H	M	L	H	5	5	6	2	4	6	10	1	1	1	41
H	H	L	L	2	1	7	3	5	5	4	10	4	6	47
M	M	L	H	10	8	2	6	7	2	2	2	7	3	49
M	H	H	H	7	3	8	7	8	3	6	7	2	5	56
H	M	L	L	3	2	5	4	10	9	7	9	3	4	56
M	M	L	L	8	5	3	10	1	1	8	8	9	11	65
M	L	H	H	6	10	9	5	3	8	5	6	6	8	66
M	L	L	H	11	9	4	8	6	7	3	4	8	7	67
H	L	L	H	9	7	10	9	9	11	11	2	10	9	87
L	L	H	H	1	11	11	11	11	10	9	11	11	10	96

Temperature H = High (90-96°F) M = Moderate (77-83°F) L = Low (51-60°F); Humidity H = High (70-86%) L = Low (47-53%)

TABLE XII. GEOMETRIC MEAN TIME (HOURS) TO OBTAIN 50 PER CENT DEFOLIATION OF PLANTS SPRAYED WITH 2-BUTYNE-1,4-DIOL

Less Resistant	Hours to 50 Per Cent Defoliation
Privet	29.2
Acacia	34.4
Locust	37.5
Persimmon	40.7
Pomegranate	61.7
More Resistant	
Hibiscus	93.1
Mulberry	105.4
Elm	109.7
Cherry Laurel	145.3
Oak	173.6

F. FORMULATION STUDIES

1. Desiccant Combinations

The following desiccants, surfactants, and a solvent oil combined with Deobase and tributyl phosphate were tested on bean and privet plants: Shell Formulation 1369, and formulated dinitro-o-sec-butylphenol (desiccants); Triton GR-7 and Triton X-45 (surfactants); and dibutoxy tetraglycol (solvent oil). Solutions containing 20, 40, 100, and 200 milligrams of chemical were made with four milliliters of a 2.5 per cent solution of tributyl phosphate in Deobase. The solution of Shell Formulation 1369 and tributyl phosphate was most effective in causing desiccation. Mixtures containing dinitro-o-sec-butylphenol caused severe injury but subsequent drying was very slow.

2. 2-Butyne-1, 4-diol Formulations

Five to 20 per cent dextrose or 0.02 to 20 per cent ammonium sulfamate, ammonium nitrate, urea and 0.02 per cent ammonium phosphate added to butynediol did not improve its effectiveness in defoliating woody species.

Cellosolve used as a solvent for 20 per cent butynediol or five per cent Endothal was superior to a water formulation when sprayed on Pereskia. However, when a surfactant, Tween 20, was added to each of the formulations, greater defoliation resulted and the difference in the effectiveness of the formulations was no longer evident.

3. Formulations of Aminotriazole and 2,4-D Amide with Defoliants

Combinations of 3-amino-1,2,4-triazole and 2,4-dichlorophenoxyacetamide with butynediol, 3,6-endoxohexahydrophthalic acid (Endothal), and sodium chlorate were tested on beans and four woody plants at three application rates. The results indicated that (a) the additional aminotriazole and 2,4-D acetamide somewhat reduced the defoliant properties of butynediol and sodium chlorate; (b) 2,4-D amide combined with Endothal induced much greater defoliation of privet than Endothal alone, (99 per cent vs 10 per cent); (c) combinations of aminotriazole and 2,4-D amide with the defoliants appeared to prevent most of the regrowth which ordinarily follows rapid defoliation.

4. Effect of Tween 20 in Combination with Defoliants

Endothal solutions with and without Tween 20 were sprayed on bean plants to determine the effect of the addition of the surfactant. Endothal dissolved in water, ethyl alcohol, and ethyl cellosolve and sufficient

Tween 20 to make a 0.5 per cent Tween concentration caused severe injury and marked defoliation of Black Valentine bean plants (75 to 100 per cent). Without the surfactant mild to moderate injury occurred (0 to 50 per cent defoliation).

In another experiment with Endothal dissolved in water, the effect of the concentration of the surfactant was investigated. Insignificant differences between defoliation results were noted for surfactant concentrations ranging from 0.005 to 0.5 per cent.

G. VEGETATION CONTROL STUDIES

1. Secondary Screening

Dormant hibiscus, privet, elm, locust, and oak trees were brought into a greenhouse and with pereskia and bean plants were sprayed with full-, half-, and quarter-strength concentrations of the following materials:

benzoic acid formulation 103A
benzoic acid formulation 177
Dow pentachlorophenol formulation M562 JT895-3
Formulation of 2,4-D in tributyl phosphate (VKL)

Two milliliters of 1250, 2500, and 5000 parts per million of 2,4-D acetamide and 2,4,5-T acetamide were sprayed on test plants placed in an area of one-third square yard.

The benzoic acid treatments caused severe malformation of subsequent shoot growth, but did not substantially suppress bud sprouting. 2,4-D amide and 2,4,5-T amide slowed bud sprouting and shoot growth, and caused some malformation of leaves. Pentachlorophenol induced bud sprouting and apparently normal shoot growth at a much faster rate than in the untreated plants. Five weeks after treatment, all woody plants with the exception of elm that were sprayed with full-strength VKL were still apparently dormant; most plants were still alive and some were developing proliferated stem tissue. Elm sprouts in this treatment were fewer and developed more slowly than the controls.

2. VKL-Type Formulations

A second vegetation control test was carried out with 12 to 25 per cent rates of aminotriazole (ATA), 2,4-D, 2,4,5-T and 2,4-dichloro-5-fluoro-phenoxyacetic acid, each combined with full-strength tributyl phosphate (TBP), dibutyl butyl phosphate (DBP), and butyl dibutyl phosphate

(BDP). Dormant and sprouted trees and shrubs were sprayed with six milliliters of solution per two-thirds square yard. Twelve to eighteen milliliters of spray solution were used when compounds had to be applied separately because of solubility factors. Results of this test are given in Table XIII.

Treatments containing TBP, DBP, or BDP caused rapid killing of leaves and shoots on the sprouted plants. The combination of 2,4-dichloro-5-fluorophenoxyacetic acid with TBP, DBP, or BDP appears to be most effective in killing vegetation. The phenoxy compounds alone caused slow death of leaves and shoots of sprouted plants and were not more effective than the desiccants TBP, DBP, and BDP except that in most instances phenoxy compounds prevented new growth and the desiccants did not. Aminotriazole caused severe chlorosis and a temporary cessation of growth, but most plants resumed growth prior to the nine-week observation time. Elm and privet were the most easily killed with these compounds and hibiscus was the most resistant.

3. Field Test with VKL-Type Compounds

Three and fifteen-hundredths pounds of chemicals per gallon of tributyl phosphate were sprayed on dormant vegetation during the first part of April 1957. One to three milliliters of the compounds were sprayed on areas of branches one to three square feet. Results are tabulated below:

Tributyl phosphate	Suppressed bud growth on 7 of the 9 species treated.
Tributyl phosphate + 4 - fluorophenoxyacetic acid (1661) or - methoxy-4-fluorophenyl acetic acid (4547)	Increased effectiveness to 8 out of 9 species.
Tributyl phosphate + 2,4-dichloro-5-fluorophenoxyacetic acid (10778) or 3,5-dichloro-2-pyridoxyacetic acid (14213) or 2,4-dichlorophenoxyacetic acid	Prevented bud development of all species tested.

TABLE XIII. EXTENT OF VEGETATION CONTROL OBTAINED WITH MIXTURES OF CONTACT AND SYSTEMIC HERBICIDES SPRAYED ON WOODY PLANTS

Treatment	Condition of Plants[a]									
			Sprouted					Dormant		
	Oak	Hibiscus	Privet	Elm	Locust	Mesquite	Oak	Privet	Elm	Locust
25% 2,4-D	D	A	D	D	AG	A	A	D	D	D
20% 2,4,5-T	A	A	D	D	A	D	AG	D	D	AG
25% 10778	D	A	D	D	D	D	A	D	D	A
12% ATA	AG	AG	AG	AG	A	A	D	A	AG	AG
100% TBP	AG	A	D	D	A	AG	D	D	D	AG
+ 25% 2,4-D	D	A	D	AG	D	D	A	D	D	D
+ 20% 2,4,5-T	D	AG	D	D	D	D	D	D	D	A
+ 25% 10778	D	D	D	D	D	D	D	D	D	D
+ 12% ATA + 25% 10778	D	A	D	D	D	D	D	D	D	D
100% BDP	D	AG	D	AG	AG	A	D	AG	AG	AG
+ 20% 2,4,5-T	D	AG	D	D	D	D	A	D	D	D
+ 25% 10778	D	A	D	D	D	D	D	D	D	D
+ 12% ATA + 25% 10778	D	A	D	D	D	D	A	D	D	D
100% BDP + 12% ATA + 25% 10778	D	AG	D	D	D	D	D	D	D	D
Untreated Controls	AG	AG	AG	AG	A	AG	AG	AG	AG	AG

a. A = Alive G = Growing D = Dead

4. Field Tests with Benzoic Acids

Dormant branches of six species were sprayed with benzoic acid formulations 177 and 103A either during late fall 1956 or early spring 1957. Marked suppression of growth of the treated branches resulted. Later, small dormant trees of black cherry and tulip were thoroughly sprayed with the benzoic acid formulations. At first, bud development and growth were greatly suppressed, but following the development of several severely malformed leaves, new growth appeared normal. The plants responded to both benzoic acid formulations essentially in the same manner.

5. Field Test of Phenoxy Compounds

Trees sprayed during the summer of 1956 with solutions of 5, 60, 500, and 1000 parts per million of 2,4-D, 2,4-D amide, 2,4,5-T, 2,4,5-T amide, and 2,4 dichloro-5-fluorophenoxyacetic acid were examined in the spring of 1957 for residual effects. One- to three milliliter amounts of the chemicals had been sprayed on branch areas of one to three square feet. In all cases where the branches were not killed, no unusual or malformed growth developed. Sprays with the butyl esters of 2,4-D and 2,4,5-T killed most of the treated branches.

III. DEFOLIATION AND DESICCATION STUDIES WITH CONIFERS

A. GREENHOUSE AND CONTROL ROOM TESTS

Greenhouse and control-room tests on the defoliation and desiccation of conifers were initiated in 1957 on Tsuga canadensis (Canadian hemlock), Picea abies (Norway spruce), and Pinus sylvestris (Scotch pine). These plants were grown individually in quarter-gallon glazed pots in the greenhouse.

1. Primary Screening

Primary screening of compounds was conducted initially on spruce and hemlock and later on pine. Compounds were selected on the basis of activity determined by tests on broad-leaf species, those suggested by literature reviews, and those thought to be potentially active because of their structural formulae.

Compounds used in the greenhouse test and their defoliation and desiccation activity are shown in Table XIV.

2. Control-Room Tests

An experiment was conducted to study the effects of variations in light intensity, temperature, and relative humidity upon conifer defoliation. Tsuga canadensis (Canadian hemlock), Pinus sylvestris (Scotch pine) and Picea abies (Norway spruce) were sprayed with sodium 2,3,6-trichlorobenzoic acid and tributyl phosphate at 30 pounds per acre. Injury and defoliation were much greater under conditions of high temperature, 90°F, than at medium or low temperatures, 72° and 65°F, respectively. Slight to no effect resulted from high or low light intensity or humidity. The majority of plants so treated and exposed to high temperature died within two to three weeks; plants treated with tributylphosphate survived. Injury caused by 2,3,6-trichlorobenzoic acid developed more slowly than that caused by tributyl phosphate, but the injury was more severe.

B. FIELD TESTS

In 1957 field tests on conifers were conducted to determine (a) the relative effectiveness of several chemicals, (b) the effect of seasonal changes upon plant response, and (c) the response of different conifer species to the same or similar chemicals.

In April and May single branches of Canadian hemlock were thoroughly sprayed (without run-off); in August and October small trees were sprayed at 10 milliliters per one-half square yard. A battery-operated sprayer equipped with a Devilbiss No. 275 nozzle was used during the experiments. Compounds and formulations tested and the results from this experiment are given in Table XV.

TABLE XIV. DEFOLIATION AND DESICCATION OF CONIFERS BY SPRAYING WITH COMPOUNDS

COMPOUNDS
2,4-Dichlorophenoxyacetic acid
2,4,5-Triclorophenoxyacetic acid
2,3,5,6-Tetrachlorobenzoic acid[a]
2,3,6-Trichlorobenzoic acid plus isomers[a]
≠ 18 Other halogenated benzoic acids[b]
Sodium salt of 2,3,6-trichlorobenzoic acid plus isomers
Active polychlorobenzoic acids, 2 lb/gallon (ACP 103 formulation)[a]
Active polychlorobenzoic acids, 2 lb/gallon (ACP 177 formulation)[a]
3,6-Endoxohexahydrophthalic acid, 6.3% formulation (Endothal)
2-Butyne-1,6-diol (35% formulation, aqueous)
P, P-dibutyl-N,N-diisopropylphosphinic amide (2 lb/gallon Shell formulation 1369)
Sodium chlorate and sodium pentaborate (18.2 and 10.0% respectively) (Shed-a-leaf formulation)
Bis(ethylxanthogen) trisulfide (25%, Phillips 66 cotton defoliant 713D)
Pentachlorophenol (4 lb/gallon formulation-Golden Harvest M562 Jt 895-3)
2,4-D in tributyl phosphate formulation (VKL)
Butyldibutyl phosphonate
Tributyl phosphate[a]
Tributyl phosphorotrithioite
Ammonium phosphate
Ammonium sulfate
Ammonium thiocyanate
Ammonium sulfamate
Potassium nitrate
3-amino-1,2,4-triazole
Esters of 2,3,5-tribromobenzoic acid
Esters of 2,3,5-triiodobenzoic acid
Isopropyl ester of 4-fluorobenzoic acid
Sodium salt of 4-fluorobenzoic acid

a. Compounds showing the highest conifer defoliation and desiccation
b. Less active compounds than 2,3,5,6-TBA and 2,3,6-TBA

TABLE XV. DEFOLIATION OF CANADIAN HEMLOCK SEVEN DAYS AFTER SPRAYING WITH CHEMICALS
Figures Represent Per Cent Defoliation

COMPOUNDS AND FORMULATIONS	DATES TREATED			
	11 Apr	24 May	2 Aug	15 Oct
2,3,6-trichlorobenzoic acid[a]	22	50	70	0
Tributyl phosphate	22	100[b]	95	100[b]
Diesel Oil	0	0	0	0
2-butyne-1,4-diol formulation (35 per cent active)	0	0	5	0
3,6-endoxohexahydrophthalic acid formulation (Endothal)[c]	0	0	5	0
Polychlorobenzoic acid formulation (ACP-M-103-A)[c]	0	40	60	0
Polychlorobenzoic acid formulation (ACP-M-177)	0	40	40	0

a. 20 per cent in ethyl alcohol and 0.5 per cent Tween 20.
b. Mature needles only.
c. Sprayed as a 50 per cent solution in ethyl alcohol.

Fifty-four days after the treatment of 24 May, all compounds except Endothal and diesel oil had caused 90 to 100 per cent defoliation. Hemlock completely defoliated more readily after spring growth had matured. The increase in activity of most chemicals in the 2 August treatment is attributed to the higher ambient temperature. This suggestion is supported by the results of the control-room experiments.

In a second test, single branches of red pine were treated on 2 May, 10 June, 10 July, 30 August, and 22 October with the same group of chemicals used on hemlock in the first test. Tributyl phosphate and the benzoic acid materials caused greater injury than butynediol, Endothal, or diesel oil. The greatest amount of injury from all treatments occurred during the warmer months. In comparison with hemlock, red pine was much more resistant to chemically induced defoliation. Two weeks after spraying, only a few per cent of the needles had abscissed.

In a third test, scrub pine trees three to four feet high were sprayed with sodium 2,3,6-trichlorobenzoic acid, Phillips 713D, ACP-M-103-A, and formulated pentachlorophenol at 10 and 30 pounds per acre. The chemicals were dissolved in 10 milliliters of ethyl alcohol with sufficient Tween 20 added to make a 0.5 per cent Tween concentration. Pentachlorophenol, sprayed at 30 pounds per acre, was the only chemical in this experiment that caused significant defoliation; both young and mature needles defoliated. Sodium 2,3,6-trichlorobenzoic acid and ACP-M-103-A (benzoic acid materials) caused the greatest amount of injury, but they did not induce defoliation.

IV. DISCUSSION AND CONCLUSIONS

A. DEFOLIANTS OF WOODY SPECIES

1. Deciduous Plants

Data collected from visual observations indicate that the six best defoliants for deciduous plants in order of preference are: 2-butyne-1, 4-diol (butynediol); tributyl phosphorotrithioite (Folex); 3,6-endoxohexahydrophthalic acid (Endothal); sodium chlorate (Shed-a-leaf); P, P-dibutyl-N, N-diisopropylphosphinic amide, and magnesium chlorate.

On the basis of two years' work, butynediol defoliated the greatest number of species in the shortest time. It caused reasonably good defoliation on all plants treated at almost any time trees were in leaf. However, it is apparently less effective during dry conditions. Whether this decrease in effectiveness is due to low humidity, a decrease in entry efficiency because of closed stomates, or a combination of these factors has not been determined.

Tributyl phosphorotrithioite (Folex) caused defoliation of approximately the same number of species as butynediol but the response was slower. The branches of some species were killed when sprayed with Folex.

The results obtained in 1957 with Endothal were superior to those obtained in 1956. The increased activity is attributed to the higher temperatures during the 1957 test.

Sodium chlorate performed equally well in 1956 and 1957 with little change in the amount of defoliation or the number of species defoliated. Treatments with sodium chlorate are usually associated with considerable injury to leaf tissue. In some instances the excessive injury appeared to reduce abscission. The effects of magnesium chlorate were essentially the same as those induced by sodium chlorate. However, magnesium chlorate defoliated fewer species.

Many plant species were defoliated by treatments with P, P-dibutyl-N, N-diisopropylphosphinic amide, but since the rate of defoliation was so slow, this compound was rated next to last. There is a possibility that the leaves abscissed more as a result of the physical injury caused by the compound than as a direct result of the chemical treatment.

Species were defoliated more rapidly and in greater numbers by applying defoliants (a) in combination with surfactants, and (b) in small droplets 100 microns in diameter to under surfaces of leaves.

Considerable effort was made to determine the effects of environment upon chemically induced defoliation. In general, under controlled day length the rate and amount of defoliation of treated plants (a) increased under conditions of continuous moderate temperature and a high continuous humidity, and (b) decreased under conditions of continuous low temperature and continuous high humidity.

Defoliants are translocated only upward in stems and move from leaves into the stems and upper foliage.

2. Conifers

Halogenated benzoic acids, pentachlorophenol, and tributyl phosphate were the most effective defoliants on the conifer species tested. Canadian hemlock is an excellent test plant for defoliation experiments because of its comparatively rapid, sensitive response to defoliant chemicals. However, results obtained from hemlock screening tests are only preliminary and do not necessarily apply to conifers in general.

B. DESICCANTS

The following compounds rate highest in desiccant activity: tributyl phosphate, butyl dibutyl phosphonic acid, dibutyl butyl phosphinic acid, and P, P-dibutyl butyl-N, N-diisopropyl phosphinic amide.

All of the compounds have a common property, a butyl-phosphorus linkage. Results of experiments with organic phosphates indicate that the butyl-phosphorus linkage may be the only combination that causes severe, rapid desiccant activity. However, an experiment with phosphinic amides similar to P, P-dibutyl-N,N-diisopropyl phosphinic amide indicated that the butyl group may not necessarily be the only alkyl group to impart high activity to this series of compounds. Unfortunately, all compounds in the test have a high level of activity and it was not possible to obtain significant differences between compounds. Phosphinic amides were not re-evaluated on a wide number of species for desiccant activity.

C. VEGETATION CONTROL

Mixtures of phenoxy-type compounds with desiccants such as tributyl phosphate show definite promise in attaining rapid, reasonably permanent vegetation control. A combination of 2,4-dichloro-5-fluorophenoxyacetic acid and tributyl phosphate is particularly effective.

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REPLY TO
ATTENTION OF

RDCB-DPC-RS

03 MAR 2011

3 MAR 11

MEMORANDUM THRU Edgewood Chemical Biological Center, Technical Director, (RDCB-D/Mr. Wienand), 5183 Blackhawk Road, Aberdeen Proving Ground, MD 21010-5424

FOR RDECOM Office of Chief Counsel (AMSRD-CC/Mr. Brian May), 5183 Blackhawk Road, APG, MD 21010-5424

SUBJECT: RDECOM Freedom of Information (FOIA) Request

1. References:

a. Army Regulation 380-86, Classification of Former Chemical Warfare and Biological Defense, and Nuclear, Biological, and Chemical Contamination Survivability Information, dated 22 Jun 05.

b. Army Regulation 25-55, The Department of the Army Freedom of Information Act Program, dated 1 Nov 97.

2. The request from RDECOM asks for release of the following three documents pertaining to agent orange. ECBC subject matter experts have recommended allowing public release for these documents.

a. Technical Memorandum 46, Field Screening of Desiccants and Defoliants, Kenneth D. Demaree, April 1964.

b. Proceedings of the First Defoliation Conference, 29-30 July 1963, published January 1964.

c. Technical Report BWL 16, Defoliation and Desiccation, Preston, W.H., Downing C.R., and Hess, C.E., July 1959.

3. The ECBC point of contact is the undersigned at 410-436-7232 or june.sellers@us.army.mil.

JUNE K. SELLERS
ECBC Security Manager

Concur with ECBC's recommendation.
BRIAN A. MAY
FOIA Officer, HQ RDECOM

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