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ALVIN 1 YOUNG, Major, USAF Consultant, Environmental Sciences

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SECOND & THIRD

SEMI-ANNUAL REPORT

CONTRACT DAAA13-67-C-0113

PERIOD 1 NOV. 67 TO 30 OCT. 68

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Type of Report - Second Semiannual Report

Title: Development of arsenic based defoliants

Dates covered by report: 1 November, 67 to 30 April 68

Author: Charles A. Burleson

Contract Number: DAAA13-67-C-0113

Work Performed for: Fort Detrick; Frederick, Maryland

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Name and address of organization preparing report:

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#### ABSTRACI

Development of arsenic based defoliants - - 
Second semiannual progress report

#### <u>Phase I</u>

Thirty-eight (38) new arsenical compounds were tested in primary screening against six plant species. Commercial formulation of cacodylic acid and monosodium acid methanearsonate were also tested for comparison purposes.

Preparation of 100g or greater quantities of the more active compounds tested against gardenia and privet were prepared and shipped to Fort Detrick.

This completes Phase I of the contract.

#### Phase II

No work was done on Phase II due to the time of year. The portion of Phase II that is incomplete will be completed and reported in the third semiannual progress report.

Development of arsenic based defoliants - - Second semiannual progress report

The second semiannual progress report will cover that research completed during the period of 1 November 67 through 30 April 68.

#### Phase I

- a. Primary Screening: Thirty-eight (38) new arsenical compounds prepared and selected by the contractor from some fifty synthesized were subjected to a primary screening program in the greenhouse using two replicates and the following test plants
  - (1) 7 day old plants:
    - (a) Beans --- Black Valentine
    - (b) Soybean -- Chippewa
    - (c) Morningglory--Heavenly Blue
    - (d) Radish ---- Comet
    - (e) Oats ---- Lodi
    - (f) Rice ---- Belle Patna
  - (2) 14 day old plants: .
    - (a) Beans ---- Black Valentine

In some tests, commercial formulations of cacodylic acid and monosodium methanearsonate (Phytar 160 and Ansar 170, respectively) were included for comparison.

#### PROCEDURE

#### Growing of Test Plants

Seeds of the six test species used were planted in a soil medium in either six-inch earthern pots or 4 1/4  $\times$  4 1/8 inch plastic pots. The soil medium consisted of three parts local high organic clay loam soil, one part peat moss and one part sand. To assure adequate fertility, the equivalents of 1000 lb/A lime and 10 lb/A each of N,P205, and K20 were added to and thoroughly mixed into the soil medium prior to use.

The temperature was maintained at a constant 78-82°F in the growing greenhouse, but relative humidity varied from 30 to 55%, and variation in light quantity and intensity gave variable germination rates. Because of these differences in germination and seedling growth rates from one season of the year to another, seedings made for the 7-day treatments were made from 7 to 10 days prior to treatment. This allowed time for grasses to develop 3 to 4 true leaves. Likewise, the seedings made for the 14-day treatments were made from 14 to 18 days prior to treatment. This allowed time for at least two trifoliate leaves to fully develop on the Black Valentine Beans.

#### Chemical Sample Preparation

Preliminary experimentation was conducted to determine the relative solubility of the new compounds in water, acetone, and methanol.

When sample material was adequate, 1.8 g. of a given compound was dissolved or suspended in the selected solvent and the solution brought up to a volume of 60 ml base solution.

1 1b/A Rate - - - 8 ml of the original solution
was diluted further with the selected solvent, 0.04 ml
of Tween 20 surfactant was added and the solution
brought up to a volume of 40 ml.

5 1b/A Rate - - - To the remaining 52 ml of the base solution was added 0.052 ml Tween 20.

The above concentrations were caluclated to provide 1 and 5 1b/A rates when applied at a 20 gal/A volume. The surfactant level was equal to 0.1% on a volume basis.

### Spray Application

Application of the sample preparations was accomplished with a "Beltsville Type" spray apparatus employing a stationary spray nozzle and moving belt. The apparatus employed a T-Jet fan type nozzle with air pressure as the propellent. It was calibrated to apply a volume of 20 gal final spray solution per acre. Pots or plastic containers containing plant material to be sprayed were assembled in 14 X 20 inch flats for ease in loading on to and off of the moving belt. The belt moved at an approximate speed of 2 mph.

Sprayed plant material was held in the headhouse until completely dry prior to being moved to the "treated plant" greenhouse.

#### Evaluation

Observations were made of treated plants daily for two weeks rollowing application, and notes were made of any unusual response. Approximately two weeks after treatment final evaluations were made as to type and extent of response. A sample data sheet is attached as Page 1 of the appendix of this report. Response ratings for six (6) species of 7-day old plants and 14-day old Black Valentine bean plants were made on a 1 to 4 rating basis as follows:

- 1. Equals no effect
- 2. Equals slight effect
- 3. Equals moderate effect
- 4. Equals severe effect

A summary of the data resulting from the primary screening is found in Table I.

New compounds screened under this contract are listed by Ansul code numbers only. Two copies of the original data sheets providing the chemistry of each compound with its corresponding code numbers are being furnished Mr. J. Ray Frank, Project Officer, Plant Physiology Division (PROV.) Fort Detrick, Maryland. The ratings listed under the column headed 7-day plants are the sum of the individual ratings for respective rates of the six species involved.

Table I. Average ratings from two replicate treatments made two weeks following application of chemicals indicated using the 1 to 4 rating described above. Values under the "7-day Plants" Column represent the total of the average evaluations for six crops.

Code Numbers		•	Activity R	
Fort Datrick	Ansul	<u>Chemistry</u>	7-day Plants	14-day Plants
	AN-513	Confidential	17/20	3/4
	AN-514	Confidential	19/23	4/4
	AN-515	Confidential	20/22	4/4
	AN-516	Confidential	13/16	3/4
	AN-517	Confidential	11/12	2/3
•	AN-518	Confidential	12/12	3/3
	AN-519	Confidential	16/17	4/4
	AN-520	Confidential	9/11	3/3
	AN-521	Confidential	7/9	1/1
	AN-522	Confidential	8/12	1/1
	AN-523	Confidential	19/22	4/4
•	AN-524	Confidential	20/22	4/4
	AN-525	Confidential	6/7	1/1
	AN-526	Confidential	10/16	2/3
St.	AN~527	Confidential	14/20	4/4
	AN-528	Confidential	14/18	3/3
· · · · · · · · · · · · · · · · · · ·	AN-529	Confidential	18/21	4/4
	AN-532	Confidential	12/14	2/3
	AN-533	Confidential -	20/22	4/4
	AN-534	Confidential	24/24	4/4

			Activity 1#/	
Fort Detrick	<u>Ansul</u>	Chemistry	7-day Plants	14-day Plants
	AM-536	Confidential	12/20	3/4
	AN-537	Confidential .	17/18	3/4
	AN-538	Confidential	15/21	3/4
	AN-539	Confidential	18/24	4/4
	AN-540	Confidential	23/24	4/4
	AN-544	Confidential	22/24	4/4
	AN-545	Confidential	20/24	4/4
	AN-546	Confidential	23/24	4/4
	AN-547	Confidential	23/24	4/4
	AN-550	Confidential	15/20	3/4
	AN-551	Confidential	6/7	1/2
	AN-552	Confidential	12/15	2/3
	. AN-553	Confidential	14/18	3/4
	AN-554	Confidential	13/15	2/3
	AN-556	Confidential	9/11	1/2
	AN-557	Confidential .	16/19	3/3
	AN-558	Confidential	14/17	3/4
		Ansar 170 (Commercial)	16/20	4/4
,		Ansar 160 (Commercial)	21/23	4/4

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b. <u>Secondary Testing</u> - - No secondary screening was performed due to lack of manpower and reorganization.

#### Phase II

No work was performed on this section of the government contract during this period. This phase will be completed and reported on in the third semi-annual report.

# GOVERNMENT PRIMARY SCREENING

1	Screeni	ng No. t				Ţ	WT	A	ii tu	i ki i	ľ						
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11	Date App	plied:															
	~	y old cops Variety	Rate lbs/A	Abscission	Chlorosis	Contact	Curvature	Formative	lling .	Killing	Hecrosis	Abn. Pigment	Quilling	Advent. Rt.		Activity Rating	•
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	Bean	Black Valentine	1.0 5.0													_	•
П			1.0														
	Soybean	Chippewa	5.0														
П	Morning	Heavenly	1.0														
	Glory	B),ue	5.0		-											_	
17			1.0				_	_									
	Radish	Comet	5.0	_	-									_		_	
<b>.</b>			1.0														
	Oat	Lodi	5.0			_	_	_						_			
,		<b>Belli</b>	1.0 5.0								enches.						•
1	Rice	Patma	<b>3. 4</b>					ل	لـــا								
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	Bean	Valentine	5.0													_	
	Res	sponse: 1 -	None;	2	-	s1	<b>i</b> g!	ht	7	3	- ;	Mod	lor	ent	3;	4	4 - Severe

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Phase I - Thirty-eight (38) new screening against six plant species. monosodium acid methanearsonate were Preparation of 100g or greater quagainst gardenia and privet were prep This completes Phase I of the con	U. S. Army Biological Center Fort Detrick, Frederick, Maryland 217  arsenical compounds were tested in primary Commercial formulation of cacodylic acid and also tested for comparison purposes.  mantities of the more active compounds tested pared, and shipped to Fort Detrick.
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Title: Development of arsenic based defoliants

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Author: Charles A. Burleson

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#### **ABSTRACT**

Development of arsenic based defoliants--Third semiannual progress report

#### PHASE I

Seventy-one (71) new arsenical compounds were previously tested against six plant species. Nine (9) previously tested compounds and commercial formulations of cacodylic acid and MSMA were tested also. See semiannual progress report numbers one and two for the results.

The above and all other portions of Phase I were reported previously as complets.

#### PHASE II

Five (5) of the more active compounds in greenhouse tests were tested in field trials against willow, <u>Salix spp.</u> and mesquite, <u>Prosopia spp.</u>
They were compared to commercial formulations of cacodylic acid.

# Development of arsenic based defoliants--Third semiannual progress report

The third semiannual progress report will cover that research completed during the period of 1 May 68 through 31 October 68.

#### PHASE I

This portion of the contract has been completed and was reported in the first and second semiannual progress report.

#### PHASE II

- a. Five (5) of the more active compounds on the basis of Phase I evaluations were previously tested against red maple in field trials. Results of these trials were previously reported in the first semiannual report.
- b. Field trials were conducted on willow and mesquite using the five (5) compounds previously mentioned. These materials were compared to Phytar 160 and Phytar 138 (AN-425).

### PROCEDURE

<u>Plant Material</u> - - Three to five-foot naturally seeded willow and mesquite seedlings were selected for treatment in their original habital near Weslaco. Texas. At the

time of treatment, seedlings were growing vigorously.

7.24 inches of precipitation fell two months prior to spray application and 4.39 inches fell in the two months following application of the herbicides.

Chemical Sample Preparation - - A 2.46 g quantity of chemical, that was 100% active, was diluted to 205 ml with water or methanol, which comprised the base solution. The amount of chemical was adjusted accordingly if it was not 100% active.

5 1b/A Rate - - 0.35 ml. Multifilm X-77 surfactant was added to 70 ml of the base solution. This was then diluted with the appropriate diluent to 140 ml.

10 1b/A Rate - - 0.34 ml Multifilm X-77 surfactant was added to the remaining base solution.

#### Spray Application

A 5 sq. ft. quadrat was placed over the seedling to be treated. A 43 ml aliquot of respective final solution was then sprayed uniformly over the seedling and quadrat area, using a portable sprayer equipped with an 8001-E Tee Jet nozzle at 30 pounds pressure with  $\rm CO_2$  pressure. This gave a volume of application of 100 gal/A and a surfactant concentration of 0.25%. All treatments were made in triplicate.

The plots containing mesquite were sprayed on September 3, 1968. The temperature on that date was 88° F with 65% relative humidity. The plots containing willow were sprayed on September 4, 1968. The temperature on that date was 89° F with 68% relative humidity. The skies were partly cloudy and the wind speed was 8 - 12 mph on both days.

#### **Evaluation**

Seedlings were rated for degree of desiccation and defoliation at 7 and 14 days after application using a rating scale of 0 to 10, where 0 indicated no damage and 10 complete desiccation or defoliation. The data from these evaluations are tabulated in Table 1 and Table 2.

Table 1. Average ratings from three replicate treatments to mesquite seedlings made at indicated intervals following application on 3 September 68. Ratings are on a basis of 0 to 10 with 0 indicating no damage and 10 indicating complete desiccation or defoliation.

		Desico	ation	Defoli													
Chemical		Days	after		after				Day	s aft icati	er						
	Rate	App i 10	cation 14	Applic	14		28 1	7	ا لالك	42	OI)	90					
	16/A		14	<del></del>			Reps	4		Reps	<del></del>	<del></del>	Reps	<del></del>			
						T	11	111_	1	11	111	1	11	111			
Methyl (4-chlorophenyl)	5	10	10	5.3	8.7	×	×	×	×	×	x	×	x	x			
arsinic acid AN-394 D80033	10	9.7	10	5.3	7.5	×	x	x	x	×	x	x	x	×			
Ethylmethylarsinic acid	5	9.3	9.3	4	6	x		x	×	×	x	×	. X	, X			
AN-451 D15464	10	10	9.8	4.3	6.7	x	×	x	×	x	x	x s	2/ x 2	/ x 5			
Butylmethylarsinic acid	5	9.3	9.5	5.3	7.2	×	×	x	X	×	×	×	x	X			
AN-452 D 15465	10	10	10	3.2	5.2	×	×	x	x	×	x	×	x	X			
Methylpropylarsinic acid	5	10	10	6	7.7	×		X	x ·	×	x	X	×	X			
AN-453 D15466	10	10	10	2.2	4	x	×	x	x	x	x			x			
Reaction product of n-butyl alcohol and cacodylic acid	5	10	10	9	9	x	x	×	x	x	. <b>x</b>	×	×	x			
AN-499 D80166	10	10	9.8	6.5	7.7	x	x	x	X	x	×		×	X			
Cacodylic acid (Phytar 160)	5	9.3	9	10	10	x	x	x	X	x	x	X	x	X			
AN-481 D15675	10	10	10	8.7	9.3	x	x	x	x	x	x	X	X	×			
Cacodylic acid (Phytar 138)	5	10	9.2	9	10		x	x	x	×	x	x	x	X			
AN-471 D15678	10	10	10	6.7	8.8	x	x	x	x	x	x	x	x	X			

 $<sup>\</sup>underline{\mathcal{Y}}$  Individual reps are listed, x indicates regrowth

<sup>2/</sup> No regrowth on the upper 2 feet

Table 2: Average ratings from three replicate treatments to willow seedlings made at indicated intervals following application on 4 September 68. Ratings are on a basis of 0 to 10 with 0 indicating no damage and 10 indicating complete desiccation or defoliation.

		Desicca	2100	Defolia	tion				Re	rowt	h 1/			
		Bays	fter	Days	after				icati	after ation				
Chemical	Rate	Applica	14	Applica	14		8			42		90 2/ Reps		
•	16/A						eps II	111	<u> </u>	Reps II	III	1	II	II
Methyl (4-chlorophenyl)	5	4.5	9.3	8	8.3			x		×	x			
arsinic acid AN-394 D80033	10	8.5	10	6.8	7.7									
Ethylmethylarsinic acid	5	5.8	9	5.7	8				×	×	X			
AH-451 D15464	10	8.5	10	4	5									
Butylmethylarsinic acid	5	7.7	9.5	4.7	4.7				х.					
AN-452 D15465 AN-452 D15465	10	9.7	10	2.8	3.3									
Methylpropylarsinic acid	5	5	7.7	3.7	3.8	x			X					
AN-453 D15466	10	6.7	9.8	2	2						×			
Reaction product of n-butyl	5	4.8	7.8	2.2	2.7	X		X	X		<b>X</b>			
alcohol and cacedylic acid AN-499 D80166	10	6.3	8.8	2.3	2									
Cacodylic acid (Phytar 160)	5	5.5	9.3	2.2	2.3		X		X	X				
AN-481 D15675	10	7.3	10	3.2	2					x				
Cacodylic acid (Phytar 138) AN-471 D15678	5 10	5.5 6.7	8.7 10	4 2	5 2		x	x			x	<u>,</u>		

<sup>1/</sup> Individual reps are listed, x indicates regrowth

<sup>2/</sup> Plots were destroyed prior to 90 day ratings