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Description Notes	Items were filed together under the label, "Herbicide/TCDD Analysis TA C-52A." Includes picloram and arsenic sampling.

DEPARTMENT OF THE AIR FORCE
AIR FORCE ARMAMENT LABORATORY (AFSC)
EGLIN AIR FORCE BASE, FLORIDA 32542

REPLY TO
ATTN OF: ATMA (Lt Young/882-3431)

14 JAN 1970

SUBJECT: Penetration of Picloram in Soils of Range C-52A

TO: TA C-52A Environmental Pollution Control and Monitoring System Task Team

1. The following data refer to concentrations of picloram detected in soil samples obtained from the test grid on 21 Nov 69. The soil samples represented a composite of four 5-foot soil cores statistically obtained from a 1-acre area.

Soil Core Increment	ppm Picloram	Pounds Picloram per Acre	Pounds White Formulation per Acre
0-6 inch	--	--	--
6-12	2.76	5.52	54.1
12-18	0.83	1.66	16.3
18-24	0.46	0.92	9.1
24-30	--	--	--
30-36	--	--	--
36-42	--	--	--
42-48	0.81	1.62	15.9
48-54	--	--	--
54-60	0.27	0.54	5.3
			Total = 100.7

2. Approximately 3,300 gallons of White have been applied to the test grid. This represents 8,184 pounds of White formulation or 1,782 pounds picloram formulated as the triisopropanolamine salt. If White had been applied uniformly at the rate of 100.4 pounds formulation per acre, then 81.5 acres would have received White. Since this is greater than the 64-acre test grid, the location of the soil samples did not correspond to the area of heaviest application. Further core sampling is suggested.

B. C. Wolverton

B. C. WOLVERTON
Chief, Assessments Branch

ALVIN L. YOUNG

REPORT NUMBER 8

A STUDY OF DEFOLIANT RESIDUE IN SOILS OF TEST AREA C-52A EGLIN AFB RESERVATION, FLORIDA

The soil persistence of military defoliants has recently received considerable attention by both the general public and the scientific community. In particular, interest has centered on the persistence of the herbicides 2,4,5-trichlorophenoxyacetic acid (2,4,5-T), 4-amino-3,5,6-trichloropicolinic acid (picloram), and dimethylarsinic acid (cacodylic acid). The military defoliants are formulated from herbicides that have been commercially used in this country for many years; however, because of the rate of application and repetitive spray missions, applied herbicide concentrations not typical of commercial use are encountered. Defoliant agent Orange contains the herbicides 2,4,5-T and 2,4-dichlorophenoxyacetic acid (2,4-D), while defoliant agent White contains 2,4-D and picloram. Agent Blue contains cacodylic acid and its sodium salt, sodium cacodylate. The controversies on soil persistence of the three defoliant agents are: (a) Agent Orange has been shown to contain a highly teratogenic (fetus deforming) contaminant known as dioxin(s). Because of the low concentration of dioxin(s) in Orange, data on soil persistence is extremely difficult to obtain; nevertheless, a potential environmental hazard may exist. (b) Agent White has received criticism because of its lengthy persistence in soil. Picloram has been shown to persist in soil for periods in excess of one year. (c) Agent Blue contains 15.4% arsenic in its formulation. Despite the fact that this arsenic is in the pentavalent organic form of cacodylic acid or sodium cacodylate (forms that are only midly toxic to animals), fears on its

movement and eventual degradation to a more toxic product have been expressed.

The Eglin AFB Reservation serves various military uses, one of them being the developing and testing of aerial spray equipment. Insofar as possible, this equipment must be tested under realistic yet controlled conditions. For this purpose a testing installation has been established on the Eglin Reservation with the place of direct aerial spray application restricted to an instrumented area approximately one mile square in the southeastern part of the reservation and known as Test Area C-52A (TA C-52A). During a period of eight years (1962-1970) the one-square-mile test grid received approximately 165,325 pounds active ingredient 2,4-D, 158,197 pounds active ingredient 2,4,5-T, 2,252 pounds active ingredient picloram, and 13,180 pounds active ingredient cacodylic acid-sodium cacodylate. The last mission involving the use of Orange was in December 1970. White was last disseminated in June 1970 and Blue in September 1970. The soil of TA C-52A is a Gulf Coast Flatwood soil consisting of 93.9% sand, 3.7% silt, 2.4% clay, and 0.3% organic matter. The annual precipitation (as rainfall) for this area is 60.98 inches.

It was recognized that TA C-52A offered a unique opportunity to study the soil persistence of repetitive applications of operational quantities of the military defoliants Orange, White, and Blue. Studies on herbicidal persistence and soil leaching were, therefore, initiated in the spring of 1970 by an analysis of the vegetation density of the test grid and by bioassay and analytical studies of soil cores removed from the grid.

In order to properly evaluate herbicidal persistence and soil leaching, a vegetation chart of the test grid was prepared on 26 March 1970. The greatest amounts of vegetation were found near the water sources of the grid. There were two areas that supported very dense vegetation. A terracing effect of diminishing amounts of vegetation away from these two areas was apparent. The effects of repeated spray could be seen along the flight paths most frequently used in test programs. In these strips, vegetation occurred only near the water sources and even there it was scant. By considering the flight paths, the water sources, and the terracing effects, it was possible to divide the test grid into 16 vegetation areas. These areas formed the basis for the random selection of soil samples for a bioassay experiment investigating the phytohormonal herbicides 2,4-D, 2,4,5-T, and picloram. The statistical null hypotheses that were investigated were (a) there were no herbicide content differences among the soils of the various vegetation areas; (b) there were no differences in herbicide content among soil depths down to three feet; and (c) there was no interaction between the vegetation areas and the soil depths. In order to conduct an experiment that would provide reliable evidence with respect to these three concerns, it was recommended that three random soil cores three feet deep be taken from each of the vegetation areas and also from a control area. These cores provided the replications for the experiment. Because of the time involved in taking the soil cores and the possible effects of soil drying out if left unplanted for several days, it was necessary to apply the technique of "blocking" over the days of the soil core removal and planting. On each of

three days, one three-foot soil core was taken from each of the vegetation areas and from a control area. One cup of soil from each six-inch increment of depth for each core was planted with five soybean seeds (Glycine max (L.) Merr.) and placed randomly on a greenhouse bench. After a two-week growth period, the primary root length and the total length of each plant was measured.

A study of the effects of the defoliant Orange upon the growth of soybean plants was made in order to gain an estimate of the amount of Orange present in the soil of the test grid. Six concentrations of Orange (0, 0.25, 0.50, 0.10, 0.20, and 0.40 ppm) were mixed with control soil and following the growth period, standard curves of plant growth versus Orange concentration were prepared.

In summary, the results obtained from the field experiment indicated that there were evidences of herbicidal persistence in the areas of the test grid that had been sprayed repeatedly with defoliants. Soil leaching was more prevalent along the dissemination flight paths than in the other areas of the test grid. There were no statistical evidences of differences between wet and dry soils that received approximately the same amounts of herbicide. By considering that all phytotoxic effects on the bioassay organism were from Orange (an assumption that was not valid), approximate strengths of Orange residue on the test grid for various soil and residue conditions were obtained. In general, the dry soils with high herbicide residue (top six inches of soil core) had average concentrations of 3.16 ppm Orange (average of four cores), while analysis of wet soils with high herbicide

residues contained 2.43 ppm Orange (average of 4 soil cores). Soil leaching was most prevalent among samples from dry soils. Of the 48 soil cores collected and bioassayed, 21 cores were not significantly different from control cores (95% probability level).

Sixteen of the soil cores (one from each vegetation type) were subsampled for arsenic concentration (hence, a measure of Blue). The arsenic was extracted by a cold-acid extraction technique and analyzed by atomic absorption spectrophotometry. Four of the 16 locations contained arsenic levels above 1.0 ppm in the top six inches of soil. Further analysis for arsenic in the soil profile indicated that arsenic readily (and almost uniformly) leached throughout the soil profile. In areas receiving repetitive applications of Blue, the top six inches of soil contained arsenic levels of 1.4 ppm, and the additional six-inch increments down to five feet contained from 0.70 to 1.2 ppm arsenic.

From the bioassay study, it was evident that some areas of the test grid contained high levels of phytohormonal herbicide residue (i.e., residue showing plant responses similar to those caused by 2,4-D, 2,4,5-T, and picloram). Thus, five-foot cores were collected from two areas (dry soils) exhibiting highest herbicide residue. Each core was divided into six-inch increments, placed in amber bottles, and immediately shipped to the United States Department of Agriculture, Pesticide Investigations Laboratory, Beltsville, Maryland, for analysis of dioxin(s). No dioxin(s) was found in either soil core (the Pesticide Investigations Laboratory reported a detection limit capability of 0.0005 ppm dioxin(s)).

ARSENICAL STUDIES
MR HAMME
23 March 1970

DETERMINATION OF ARSENIC IN SOIL CORE SAMPLES

Sample No	Sample Depth	Peak Height in cm		Average Peak Height in cm	ppm As in Aliquot	ppm As in Sample XZ	Average ppm As in Sample
1	0- 6"	.30	.31	.31	.3	.6	< .6
2	0- 6"	.32	.28	.30	.3	.6	
3	6-12"	.28	.28	.28	.3	.6	< .6
4	6-12"	.32	.30	.31	.3	.6	
5	12-18"	.28	.38	.33	.35	.7	< .8
6	12-18"	.39	.35	.37	.4	.8	
7	18-24"	.32	.30	.31	.3	.6	< .7
8	18-24"	.34	.34	.34	.35	.7	
9	24-30"	.44	.38	.41	.5	1.0	<1.0
10	24-30"	.34	.46	.40	.5	1.0	
11	30-36"	.30	.36	.33	.35	.7	< .8
12	30-36"	.40	.40	.40	.5	1.0	
13	36-42"	.40	.50	.45	.55	1.1	<1.1
14	36-42"	.35	.41	.38	.5	1.0	
15	42-48"	.38	.32	.35	.38	.8	< .8
16	42-48"	.36	.32	.34	.36	.7	
17	48-54"	.38	.30	.34	.35	.7	< .7
18	54-60"	.38	.46	.42	.5	1.0	<1.0

Soil Cores Collected from grid on 21 NOV 69

Standards ppm As	Peak Height in cm		Average Peak Height in cm
0.5	.38	.42	.40
1.0	.80	.82	.81
2.0	1.28	1.20	1.24
3.0	1.90	1.88	1.89
5.0	2.80	2.72	2.76
10.0	4.72	4.80	4.76
20.0	7.90	8.08	7.99
40.0	11.88	11.98	11.93
60.0	14.32	14.34	14.33
100.0	16.52	16.54	16.53

Arsenical Studies
Mr Hamme
23 June 1970

Determination of Arsenic in Silt Samples

Sample Date	Sample No	PPM Arsenic
8 April 1970	G-1	0.2
8 April 1970	R-1	0.2
8 April 1970	T-1	0.2
12 May 1970	M-1	0.2
12 May 1970	T-1	0.1
12 May 1970	T-2	0.05

Arsenical Studies
Mr Hamme
22 June 1970

Determination of Arsenic in Soil Core Samples

Sample No.	PPM Arsenic	Average PPM arsenic
C-9(1)*	0.6	0.70
	0.8	
C-9(2)	0.8	0.75
	0.7	
C-9(3)	0.7	0.65
	0.6	
C-9(4)	0.7	0.60
	0.5	
C-9(5)	0.6	0.55
	0.5	
C-9(6)	0.5	0.55
	0.6	
C-9(7)	0.4	0.50
	0.6	
C-9(8)	0.5	0.45
	0.4	
C-9(9)	0.4	0.45
	0.5	
C-9(10)	0.4	0.60
	0.8	
L-5(1)	0.6	0.50
	0.4	
L-5(2)	0.5	0.60
	0.7	
L-5(3)	0.5	0.50
	0.5	
L-5(4)	0.5	0.55
	0.6	
L-5(5)	0.4	0.50
	0.6	
L-5(6)	0.6	0.50
	0.4	
L-5(7)	0.4	0.45
	0.5	
L-5(8)	0.5	0.50
	0.5	
L-5(9)	0.5	0.45
	0.4	
L-5(10)	0.4	0.45
	0.5	0.45

*Location of cores is specified by core code (e.g., C-9 & L-5) and represents sampler number on the TA C-52A test grid. The depth of the core is specified by the number in parenthesis (e.g., 1-10) and indicates successive 6-inch increments. Both cores were collected on 27 May 1970.

TABLE

Results of Chemical Analysis for picloram, 2, 4-D, 2, 4, 5-T, 2, 3, 7, 8-Tetrachlorodibenzo-p-dioxin (TCDD) and arsenic in soil samples from Eglin AFB Test Area C-52-A.

DATE COLLECTED	SAMPLE	DEPTH	PECLORAM ppb	2, 4-D ppb	2, 4, 5-T ppb	TCDD ppb	ARSENIC ppm
Nov. 1969	K-9N	1	34, 21	1.2	2.8	1.0	2.84
		2	21,11	7.0	2.0	1.0	0.86
		3	10,6	0.1	0.8	1.0	0.90
		4	10,5	0.1	0.6	1.0	0.52
		5	10, 5	0.1	0.9	1.0	0.62
		6	10, 5	0.1	0.3	1.0	0.54
Apr. 6, 1970	J-3NE	1	ND	1.7	1.2	1.0	0.55
		2	ND	1.7	1.0	1.0	0.34
		3	ND	0.1	1.0	1.0	0.41
		4	ND	0.1	1.0	1.0	0.41
		5	ND	ND	ND	1.0	ND
		6	ND	0.1	0.7	1.0	0.62
Dec.10,1970	J-3NE	1	10	0.1	2.4	0.1	4.70
		2	10	0.1	1.8	0.1	1.30
		3	10	0.1	1.1	0.2	0.90
		4	10	0.1	0.7	0.2	0.55
		5	10	0.1	1.0	0.2	1.33
		6	10	0.1	0.3	0.2	0.90

TABLE continued--

DATE COLLECTED	SAMPLE	DEPTH	PICLORAM ppb	2, 4-D ppb	2, 4, 5-T ppb	TCDD ppb	ARSENIC ppm
Apr. 6,1970	J-9 SE	1	ND	1.6	5.9	1.0	3.21
		2	ND	1.1	0.1	1.0	0.48
		3	ND	0.1	0.7	1.0	0.20
		4	ND	0.1	0.4	1.0	0.27
		5	ND	0.1	0.3	1.0	0.27
		6	ND	0.1	0.4	1.0	0.20
Apr. 6,1970	L-6 SW	1	ND	ND	ND	1.0	ND
		2	ND	ND	ND	1.0	0.86
		3	ND	ND	ND	1.0	0.55
		4	ND	ND	ND	1.0	0.48
		5	ND	ND	ND	1.0	0.62
		6	ND	ND	ND	1.0	0.62
Dec. 10,1970	B-14 SW	1	10	0.1	0.6	0.1	0.55
		2	10	2.4	0.4	0.1	0.55
		3	10	0.6	0.4	0.1	0.55
		4	10	0.1	0.2	0.2	0.58
		5	10	0.1	0.1	0.3	0.41
		6	10	0.1	0.1	0.1	0.48
Dec. 10,1970	C-9 SW	1	10	0.1	1.8	0.4	1.64
		2	10	0.1	1.2	0.3	0.88
		3	10	0.1	0.3	0.2	0.48
	4	4	10	0.1	0.3	0.1	0.48
		5	10	0.1	0.1	0.2	0.55
		6	10	0.1	0.4	0.4	0.52

TABLE continued--

DATE COLLECTED	SAMPLE	DEPTH	PICLORAM ppb	2, 4-D ppb	2, 4, 5-T ppb	TCDD ppb	ARSENIC ppm
Dec. 10,1970	M-8 SW	1	10	5.6	7.0	0.2	0.90
		2	10	5.8	1.4	0.2	0.48
		3	10	7.6	2.8	0.2	0.34
		4	10	15.0	5.6	0.1	0.41
		5	10	5.0	2.8	0.5	0.34
		6	10	13.2	6.8	0.8	0.55
Dec. 10,1970	M-12 SW	1	10	7.6	2.2	0.2	1.86
		2	10	10000	1.0	0.2	1.86
		3	10	11.8	1.2	0.2	0.76
		4	10	4.8	1.4	0.3	0.69
		5	10	6.0	2.6	0.2	0.69
		6	10	7.0	3.0	0.2	0.76
Dec. 10,1970	0-5S	1	ND	0.8	1.2	0.1	0.90
		2	ND	0.6	0.6	0.1	0.80
	3	3	ND	0.8	1.2	0.2	0.76
		4	ND	0.6	0.6	0.2	0.41
		5	ND	0.1	8.4	0.7	0.69
		6	ND	0.6	1.4	0.1	0.55
Dec. 10,1970	0-7 NE	1	10	ND	6.6	0.2	1.52
		2	10	2.8	0.2	0.3	0.76
		3	10	3.6	0.4	0.2	0.76
		4	10	0.8	2.6	0.2	0.62
		5	10	5.6	2.6	0.2	0.62
		6	10	1.2	0.2	0.6	0.62

TABLE continued--

DATE COLLECTED	SAMPLE	DEPTH	PICLORAM ppb	2, 4-D ppb	2, 4, 5-T ppb	TCDD ppb	ARSENIC ppm
Dec.10, 1970	O-B S	1	ND	0.1	1.2	0.1	2.70
		2	ND	0.1	2.6	0.1	0.58
		3	ND	0.1	0.8	0.1	0.62
		4	ND	0.1	0.6	0.1	0.20
		5	ND	0.1	1.2	0-1	0.41
		6	ND	0.1	0.6	0.2	0.07
May 13, 1970	K-9N	1[e]	ND	0.1	8.7	1.0	3.94
		1	ND	0.1	3.2	1.0	4.25
		2	17,7	ND	ND	ND	ND
		3	10,5	ND	ND	ND	ND
		4	11, 5	0.1	0.1	1.0	0.41
		4	ND	0.1	ND	1.0	0.41
		5	10, 5	ND	ND	ND	ND
		6	ND	001	1.0	0.08	0.48
May 13,1971	G-9N	2	92,160	ND	ND	ND	ND
Dec. 10,1970 or Apr. 8,1971[f]	CONTROL	1	10	0.1	0.1	0.2	0.55
		2	10				
		2	10	0.1	0.1	0.3	0.41
		3	10	3.0	0.1	0.2	0.55
		4	10	0.1	0.1	0.4	0.24
		5	10	0.1	0.1	0.4	0.41
		6	10	0.1	0.1	0.5	0.48

REFERENCES

a) Samples designated by the nearest permanent air smapler station on the one-square mile test grid. All samples were taken 50 feet from a certain air sampler, except control site was 0.4 miles from the one-square mile grid.

b) Samples taken with a core borer in 6 inch increments. Depth 1 = 0 - 6 inches, 2 = 6 - 12 inches, 3 = 12 - 18 inches, 4 = 18 - 24 inches, 5 = 24 - 30 inches and 6 = 30 - 36 inches. Each increment was uniformily mixed prior to sampling for chemical analysis.

c) Picloram Analysis was performed by International Research and Development Corporation and/or The Dow Chemical Company; Dow Chemical Company Method ACR 69.10, modified.

d) Analysis performed by E. A. Woolson, Pesticide Investigations Laboratory, U. S. D. A., Beltsville, Maryland.

e) Two samples from same depth were taken 10 feet apart.

f) Control soil for picloram analysis taken April 8, 1971 and other analysis performed after December 10, 1970 sampling.

TABEE --

Results of chemical analysis of water aamples for Picloram.

SAMPLE LOCATION	date COLLECTED	picloram[1] ppb.
Basin Creek, North of sampler station A-11 in NE corner of one-square mile grid.	June 11, 1971	11
Trout Creek, south of sampler station 0-11 in SE corner of one-square mile grid.	June 11, 1971	2.4
Basin Creek, same as June 1971 location.	Dec. 3, 1971	11, 9.4
Trout Creek, same as June 1971 location.	Dec. 3, 1971	1.4
Control; Long Creek approx. 3 miles feom onëesquare mile grid.	June 11, 1971	0.1

1) Analysis performed by the Dow Chemical Company; Method ACR 68.14.

Marginal Area 1

MA 2

B1

	1	2	3	4	5	6	7	8	9	10	11	12	13	14
A														
B	1	2	3	4	5	6	7	8	9	10	11	12	13	
C	14	15	16	17	18	19	20	21	22	23	24	25	26	
D	27	28	29	30	31	32	33	34	35	36	37	38	39	
E	40	41	42	43	44	45	46	47	48	49	50	51	52	
F	53	54	55	56	57	58	59	60	61	62	63	64	65	
G	66	67	68	69	70	71	72	73	74	75	76	77	78	
H	79	80	81	82	83	84	85	86	87	88	89	90	91	
J	92	93	94	95	96	97	98	99	100	101	102	103	104	
K	105	106	107	108	109	110	111	112	113	114	115	116	117	
L	118	119	120	121	122	123	124	125	126	127	128	129	130	
M	131	132	133	134	135	136	137	138	139	140	141	142	143	
N	144	145	146	147	148	149	150	151	152	153	154	155	156	
O	157	158	159	160	161	162	163	164	165	166	167	168	169	

MA 8

MA 3

MA 7

MA 4

MA 6

MA5

B2