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Item ID Number	03947 ☐ **Not Scanned**
Author	Young, Alvin L.
Corporate Author	
Report/Article Title	Notes, Data, and Photographs: Ecological Studies/Gulfport, Mississippi, 1-2 July 1974
Journal/Book Title	
Year	0000
Month/Day	
Color	☐
Number of Images	60
Descripton Notes	All items were filed together in a folder labeled, "Ecological Studies/Gulfport, Mississippi, 1-2 July 1974."

DEPARTMENT OF THE AIR FORCE
THE DEAN OF THE FACULTY
USAF ACADEMY, COLORADO 80840

1 July 1977

Mr John Davidson
Ag-Organics Department
Dow Chemical USA
P.O. Box 1706
Midland, MI 48640

Dear John

Under separate cover, I have sent Mr Don Ervick, 24 soil sample for TCDD analysis (see attached). As you will note these samples are from our biodegradation plots or from Test Area C-52A. They were carefully selected to provide the maximum amount of data on (1) the soil persistance and/or degradation rate of TCDD in three soil types, (2) the leaching of TCDD in soil profiles, (3) an assessment of degradation potential of samples in storage in the absence or presence of phenoxy herbicides, and (4) the confirmation of analysis by another laboratory.

I sincerely appreciated your assistance in providing for the analysis of these samples. I will provide a brief report to you on the results.

As of 15 July 1977, I will be affiliated with the USAF Occupational and Environmental Health Laboratory, Brooks AFB, TX 78235. I will call you when I get settled on station. Again, my thanks to you for all your assistance to me during my tenure at the Air Force Academy.

Sincerely

Alvin L. Young

ALVIN L. YOUNG, Capt, USAF, PhD
Associate Professor of Biological Science
Dept of Chemistry and Biological Sciences

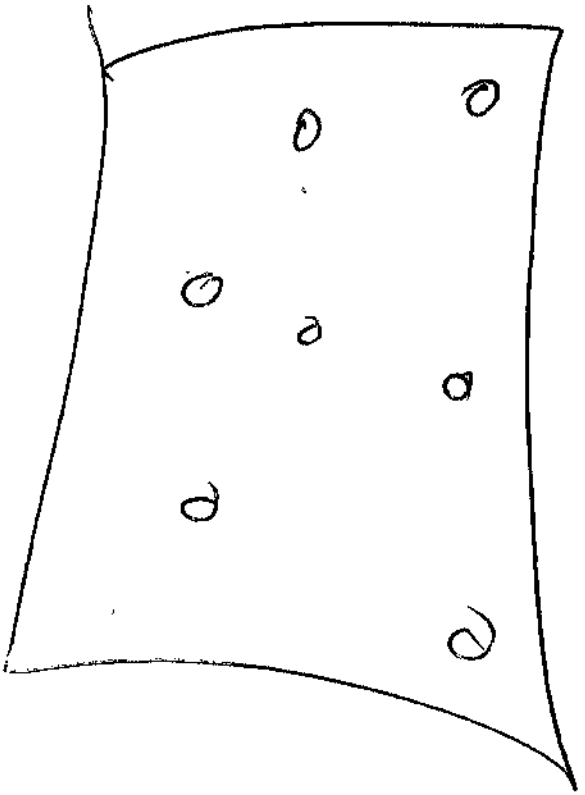
1 Atch
List of Samples

USAFA
TCDD BIODEGRADATION STUDY
SOIL SAMPLES
SUBMITTED TO DOW CHEMICAL USA
15 JUNE 1977

10 MAY 77

USAFA SAMPLE #	DESCRIPTION
1	Garden City, KS, 22 Mar 74, 4000 lb/A, 0-6
2	Garden City, KS, 14 Jan 75, 4000 lb/A, 0-6
3	1.5 ppm TCDD, Kansas soil, formulated 22 Jan 75
4	1.5 ppm TCDD plus 10,000 ppm Orange, Kansas soil, formulated 22 Jan 75
5	H-13, AFLC Test Range, UT, 5 Nov 76, 1000 lb/A, Rep I, Hole 3, 6-12
6	H-14, AFLC Test Range, UT, 5 Nov 76, 1000 lb/A, Rep I, Hole 3, 0-6
7	H-25, AFLC Test Range, UT, 5 Nov 76, 2000 lb/A, Rep I, Hole 2, 6-12
8	H-26, AFLC Test Range, UT, 5 Nov 76, 2000 lb/A, Rep I, Hole 2, 0-6
9	H-37, AFLC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 4, 12-18
10	H-38, AFLC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 4, 6-12
11	H-39, AFLC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 4, 0-6
12	H-44, AFLC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 2, 0-6
13	H-54, AFLC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep II, Hole 1, 0-6
14	0-6" Soil Core, Site 0-7, Eglin AFB, FL, collected 31 Mar 73
15	Plot #6, 0-6", Herbicide and plowed, Eglin AFB, FL, 21 May 73
16	Plot #10, 0-6", Herbicide and amendments plus charcoal, Eglin AFB, FL, 21 May 73
17	E-10, Eglin AFB, FL, 18 Nov 76, plot 5, hole 2, 12-18
18	E-11, Eglin AFB, FL, 18 Nov 76, plot 5, hole 2, 6-12
19	E-12, Eglin AFB, FL, 18 Nov 76, plot 5, hole 2, 0-6
20	E-17, Eglin AFB, FL, 18 Nov 76, plot 6, hole 1, 0-6
21	E-32, Eglin AFB, FL, 18 Nov 76, plot 7, hole 3, 0-6
22	E-41, Eglin AFB, FL, 18 Nov 76, plot 9, hole 2, 0-6
23	E-44, Eglin AFB, FL, 18 Nov 76, plot 10, hole 2, 6-12
24	E-45, Eglin AFB, FL, 18 Nov 76, plot 10, hole 2, 0-6

UNITED STATES AIR FORCE ACADEMY



Date

ROUTING AND TRANSMITTAL SLIP

TO: (Name, office symbol, room number, building, Agency/Post)	Initials	Date
1.		
2.		
3.		
4.		
5.		

Action	File	Note and Return
Approval	For Clearance	Per Conversation
As Requested	For Correction	Prepare Reply
Circulate	For Your Information	See Me
Comment	Investigate	Signature
Coordination	Justify	

REMARKS

O-6	#19 ✓	280 PPS	#11 6600
6-12	#18 ✓	8	#10 200
12-18	#17 ✓	6	#9 14

DO NOT use this form as a RECORD of approvals, concurrences, disposals, clearances, and similar actions

FROM: (Name, org. symbol, Agency/Post)	Room No.—Bldg.
	Phone No.

TCDD ANALYSIS, LIQUID ORANGE SAMPLES

Analysis Performed by ARL/LJ, WPAFB, Ohio

Samples submitted: 1 February 1975

Data Received: 11 March 1975

Sample Source	Sample Number	Date Sampled	TCDD PPM	
*Johnston Island	1	1 Aug 74	< 0.25	(a)
" "	2	"	1.3	(a)
" "	3	"	0.3	(a)
" "	4	"	< 0.07	
" "	5	"	< 0.07	
" "	6	"	0.07	
" "	7	"	4.6	
" "	8	"	4.6	
" "	9	"	5.3	
" "	10	"	0.28	
**Eglin AFB	1	1 Jan 70	< 0.04	
***Eglin AFB	2	"	< 0.04	

(a) TCDD peak appeared on top of large interference peak.

* Samples collected from Drums that were to be re-barrelled.

** Sample routinely used at USAFA for laboratory experiments.

*** Samples used in Biodegradation Plots, Eglin AFB, Florida, April, 1972.

UNITED STATES AIR FORCE ACADEMY

15 ppb = 51.77 $\mu g/m^2$

5% = $\frac{390\ \mu g/m^2}{}$ $\frac{5}{390} = \frac{100}{x}$

93 $\mu g/m^2$ A-3 325 ppt

54 $\mu g/m^2$ A-4

55 $\mu g/m^2$ A-7

OVA = 7800 $\mu g/m^3$

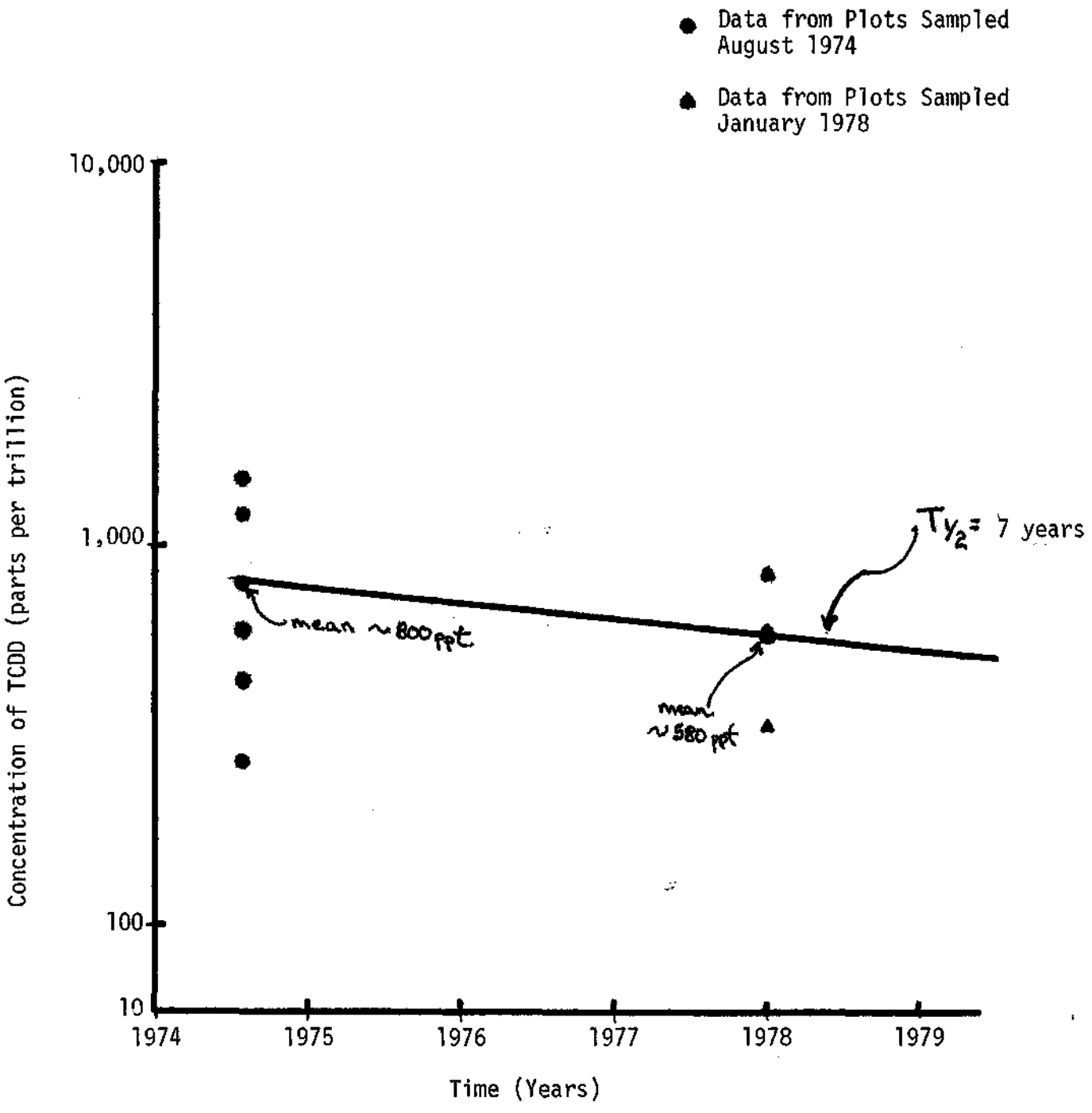
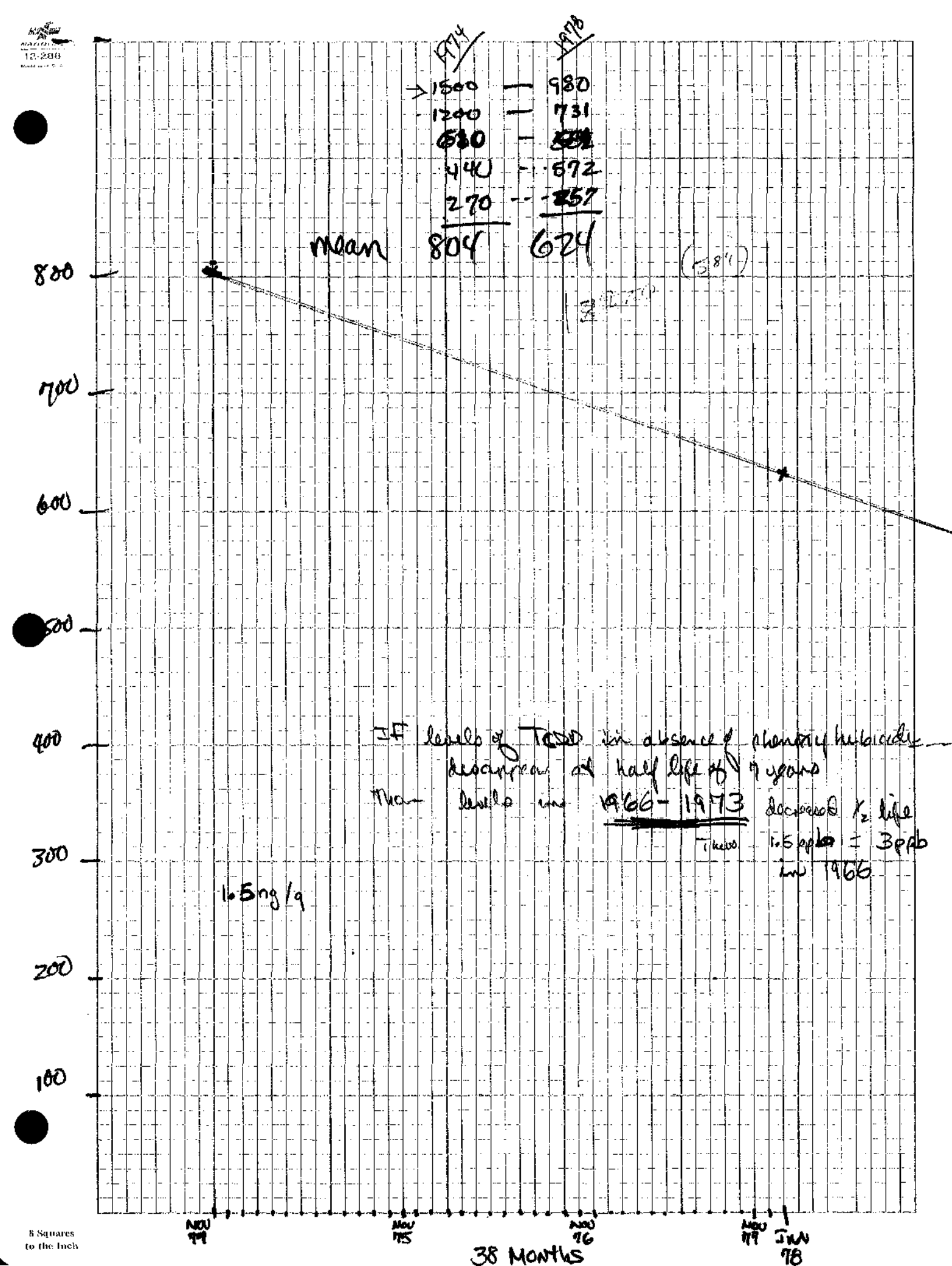


FIGURE 1. Semi-logarithmic Plot of Soil Concentration (0-15 cm) of TCDD in a Field Site Aerially-treated with 40,000 Kg 2,4,5-T, 1962-1964. Data for Each Date Represent Analysis of Five $1m^2$ Plots Established August 1974. (Unpublished Data, A. L. Young, USAF Occupational and Environmental Health Laboratory, Brooks AFB, Texas 78235.)


1974
1978
1500 — 980
1200 — 731
680 — 538
440 — 672
270 — 357
mean 804 624
(538)
800
700
600
500
400
300
200
100
If levels of TCDD in absence of chemical incidents decayed at half life of 7 years
Mean levels in 1966-1973 decayed ½ life
1.5 ppb = 3 ppb in 1966
1.5 ng/g
NOV 74
NOV 75
NOV 76
NOV 77
JAN 78
38 MONTHS
5 Squares to the Inch

EGLIN AFB TCDD DATA (GRID 1) A.C. Young

ppb

800

700

600

500

400

200

100

1 2 3 4 5 6 7

NOV 74 JAN 75 NOV 76 NOV JAN 78 78 NOV 78 JAN 79 NOV 78 NOV 79 NOV 80 NOV 81 APR 81

∴ $T_{1/2}$ TCDD in absence of 2,4-D & 2,4,5-T = 7 YEARS

K+E 8 X 8 TO THE INCH 10 X 15 INCHES KEUFFEL & ESSER CO. MADE IN U.S.A. 47 0540

K-M (Miglia)

DEPARTMENT OF THE AIR FORCE
USAF ENVIRONMENTAL HEALTH LABORATORY (AFLC)
KELLY AIR FORCE BASE, TEXAS 78241

REPLY TO ATTN OF: VT

SUBJECT: Trip Report - USAF Academy CO and Pullman WA

TO: Chief, OL AA USAF OEHL
Commander, USAF OEHL
IN TURN

1. Place: Department of Chemistry and Biological Sciences, USAF Academy CO and Department of Soils and Agronomy, Washington State University, Pullman WA.

2. Inclusive dates of travel: 25-30 Sep 1977.

3. Person making trip: Capt Alvin L. Young.

4. Primary mode of transportation: Commercial air.

5. Purpose of trip: To attend Herbicide Orange Conference at USAF Academy and review Herbicide Orange biodegradation contract at Washington State University.

6. Persons contacted: (See attached list).

7. Comments and Observations:

a. The objectives of the Air Force Academy Conference on Herbicide Orange were to (1) review recent laboratory and field data on the fate of TCDD and 2,4-D/2,4,5-T herbicides and to (2) define the direction of FY78 research. Recent data on TCDD and the phenoxy herbicides from USAF studies suggest the following:

(1) The degradation of TCDD within a soil profile (i.e., not on the soil surface) in a field environment is apparently a first order reaction, contrary to our earlier opinions (USAFA-TR-76-18). This means that at any time, the rate of TCDD loss is proportional to its concentration in the soil.

TCDD, the most favorable conditions for its activity will be defined. Attempts will then be made to culture the species in sufficient quantity to use for enriching field soil samples. The Department of Chemistry and Biological Science have on hand an excellent research staff, facilities, and equipment for this project. It is estimated that in addition to the $12,000 programmed for FY78 for the microbe study, an additional $8,000 would be necessary for labeled compounds and technician support. The Academy team is submitting a proposal for our evaluation and support.

c. From 28 - 30 September, I visited with Dr H. H. Cheng, Dr T. J. Mizuk and Mr J. T. Makja, Department of Agronomy and Soils, Washington State University, Pullman, WA. Dr Cheng is currently completing Phase II of an Orange Biodegradation Study (see Atch 1). Much of the chemical analyses of 2,4-D, 2,4,5-T and phenols is being done by a graduate study, Mr Makja in support of his Ph.D. program (Atch 2). Discussions were held on a proposed continuation of the project (Atch 3) for FY78. Details of this entire program were discussed in a previous trip report outlining FY78 Herbicide Orange Research (26 Aug 77). While at WSU, I was invited to lecture to Dr Cheng's Environmental Science (caps) Course and to Dr Muzik's WEED Science (caps) Course. Both lectures were on the "Dilemma for Disposal of Herbicide Orange." With the exception of a few final remarks on the actual incineration (e.g., efficiency, dates of incineration) the presentations were from a paper, same title, cleared by SOFOI and given in Helena, Montana, 16 Sep 75 at a conference on "Advancements in Pesticides."

d. This TDY gave me an opportunity to oversee the herbicide work by both the USAF Academy and Washington State University. Although, Orange has been incinerated, the projects by these two universities will provide valuable data in support of the reclamation of the Gulfport (NCBC) and Johnston Island storage sites. I recommend continuation of both projects during FY78.

ALVIN L. YOUNG, CAPT, USAF, Ph.D.
Chemist/Biologist Consultant

4 Atchs
1. Phase II Report
2. Rsch Report for HQ
3. Phase III & IV
4. List of Persons Contacted

	2,4-D	2,4,5-T	
H-13	.23	2.3 ppm	
H-14	T, 69.8, 83.2, 72.6	142.8, 325, 353, 327	
H-25	7.2		
H-26	60.6, 45.2, 40.2, 45.7	447.9, 223.7, 258.2, 637.4	
H-37	14	9	= 23
H-38	17	21 T	= 38
H-39			

	2,4-D	2,4,5-T	TOTAL
H-39	1040	1303	
H-44	1495	2531	3791
H-54	847	1865	2712

H-39 H'-39	1040	1303
H-44 H'-44	1495	2531
H-54 H'-54	657	1613

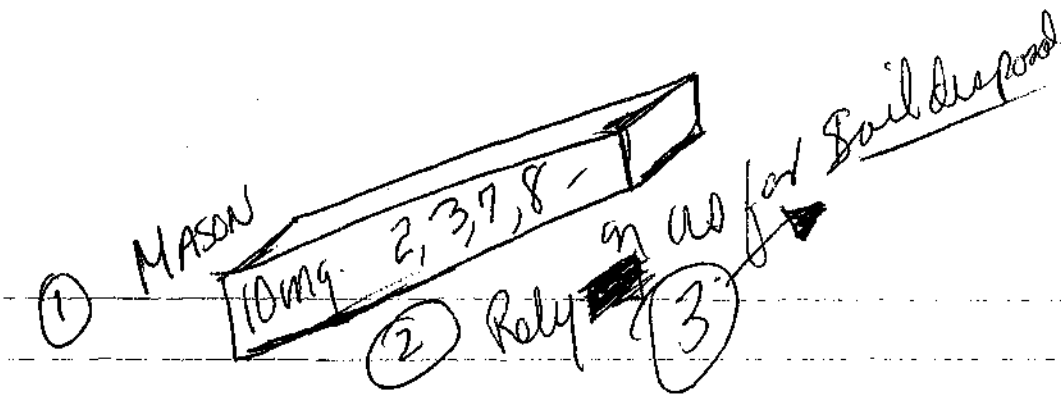
.04
40 ppb
long-term

TABLE 3. INFLUENCE of room temperature storage ON the TCDD Content of Ulysses silt loam soil with and without Herbicide

JAN 1975

TCDD
plus 10,000 ppm
2,4-D & 2,4,5-T

	TCDD plus Orange Herbicide	TCDD Without Herbicide
JAN 1975	1.5 ppm TCDD + 10,000 ppm Herbicide ***	1.5 ppm TCDD
Oct 1977	1.6 + 6,800 ppm Herbicide	



	TCDD	HERBICIDE
JAN 1975		
TCDD [redacted]	1.5	n.d.
TCDD + HERBICIDE	1.5 [redacted]	10,000*
OCT 1977		
TCDD	1.5	16
TCDD + Herbicide	1.6	6,800

Soils

225
105
170

10k-B 17.
29%
.84

16.66

16.7

7800
7300
14.6
32%
29²

14.60
.29
14.31

15.0

14.3

10,000 ppm
1 ppm TCDD
700 ppb 15 700

cc ANN. 574

NOV. 1976

	17, 19, 21, 23	16, 18, 20, 22
	0-6	6-12
PLOT 6	5	0.4
	<1	N.D
	13	N.D
	13	N.D

	34, 36	33, 35, [redacted]
PLOT 8	[redacted],	4
	11	N.D.
	N.D	2.0
	5.5	

	43, 45, 47	42, 44, 46
PLOT 10	300	<1
	1082	
	427	
	1809/3=	
	603 ppm	21 ppm

(2) Concentrations of TCDD in soils from field plots treated with a 3.7 ppm TCDD formulation of Herbicide Orange degraded five half-lives during a four year period (1972 - 1976). Therefore, the $T_{\frac{1}{2}}$ for TCDD in alkaline desert soils was approximately 290 days.

(3) Soils from field plots exposed to TCDD were brought into the laboratory, dried thoroughly at 30°C and treated to contain 1.5 ppm TCDD. Half of the soil was spiked with 10,000 ppm Herbicide Orange. Both samples were maintained for two years in the open at a constant temperature of 24°C and with no water. Less than 15% herbicide degradation occurred and no appreciable TCDD degradation occurred in either sample.

(4) Aspergillus leoporis, isolated from herbicide biodegradation plots in Utah, was evaluated for its ability to degrade ^{14}C-TCDD. Although the organism rapidly removed the TCDD from the media, no apparent metabolism occurred. Recovery of the ^{14}C-TCDD required disruption of the cellular membrane and exhaustive chloroform extraction. Thin-layer chromatographic separation suggested that the recovered ^{14}C-compound was the parent molecule.

(5) Analysis of Gulfport and Johnston Island soils for actinomycetes, fungi and bacteria indicated increased populations of microbes with increasing concentrations of herbicide. In soils from areas of herbicide spills, the populations of microbes were frequently two orders of magnitude (10^2) greater than in control soils.

(6) The movement of TCDD in a soil profile is negligible. Note that in Table 1, 98% of all TCDD detected is in the 0 - 15 cm increment of soil. The herbicide was originally subsurface injected in an 8 cm band at the 10 - 15 cm level. Thus, even in the 4,480 kg/ha plots, the TCDD detected in the 15 - 30 cm increment may have been there because of the mass movement of the herbicide at the time of application. Notice also in Table 1 the close association between rate of herbicide application

and the level of TCDD found four years after treatment. This is another indication that the rate of degradation is probably a first-order reaction.

(7) A question that has remained unanswered until recently was "How long can soil samples remain in a freezer without significantly affecting TCDD concentration?" Two soil samples have been reanalyzed after being stored for four years in a freezer in glass jars having aluminum cap liners. Table 2 give the results of the analysis. Recognizing that at this level of detection, an error of 20% is acceptable, the data support the conclusion that soil samples can be stored for at least four years with no appreciable loss of TCDD.

(8) Biodegradation plots that were treated with activated coconut charcoal prior to subsurface injection of Herbicide Orange still have significant herbicide and TCDD levels four years after treatment when compared with non-charcoal containing plots. Chemical recovery studies in the laboratory have shown that soils containing charcoal must be extracted for eight hours with benzene rather than the usual procedure of a 30 minute extraction with hexane/acetone in order to recover the TCDD.

b. The Air Force Academy Herbicide Staff have indicated their interest in continuing their program on Herbicide Orange through FY78. They propose supporting the Gulfport - Johnston Island Storage Site Study through the analyses of all soil samples for microorganisms. In addition they are very interested in a program to determine the feasibility of enriching selected field sites with microbes capable of degrading TCDD. Captain Cairney is a microbiologist and is currently the project leader. Under his direction a preliminary study of microorganisms in the soil of Gulfport and Johnston Island has been completed. He has isolated a number of species that could be evaluated for their ability to degrade TCDD. LtCol Bainter and Maj Bomar, both Ph.D. radiation biologists, are interested in examining TCDD degradation by these organisms (using tritium labeled TCDD and ^{14}C-labeled herbicides). When an organism is identified that can degrade

Microorganisms Common to Soils Exposed to TCDD

Measure Concentrations of 2,4-D, 2,4,5-T and.

Aspergillus leoparis

Species of Aspergillus have been isolated from herbicide biodegradation plots in Utah, Florida and a Florida site in Mississippi. One of the species Aspergillus leoparis was evaluated for its ability to degrade ^{14}C-TCDD. Although Aspergillus rapidly removed the TCDD from the media, no apparent metabolism occurred. Recovery of the ^{14}C-TCDD required sonication and exhaustive extraction of the cells. Thin layer chromatographic separation suggested that the recovered ^{14}C-TCDD was the parent molecule.

Soil samples

Eglin biodegradation plots were treated with a mixture of 2,4-D and 2,4,5-T containing 0.01 ppm TCDD. The theoretical concentration of TCDD should have been approximately 500 ppt based on a soil concentration of 5000 ppm herbicide. The analyses of soil cores at day 0 resulted in levels of 375 ppt. Degradation rate of TCDD in the core was as noted in the table.

	Day			
	0	414	513	709
DEPTH				
0-15cm	375	250	75	46
15-30cm	<25	50	<10	<10

$$\frac{X \text{ ppm TCDD}}{5000 \text{ ppm Orange}} = \frac{0.5}{1{,}000{,}000}$$

$$X = \frac{(0.5)(5)(10^{3})}{1 \times (10^{6})(10^{3})}$$

$$\frac{2.5}{1} \times 10^{-3}$$

.0025 ppm 0.5

2.5 ppb

(4897)

$$(.01)(5) =$$

$$1000 \overline{)\ .05000}$$

.00005 500 ppT

The Eglin Flight line sample was found to contain 0.01 ppm TCDD, Therefore the concentration of ~~the~~ TCDD at Day 0 should have been approximately ~~#0~~ ppt. Actual value determined was 375 ppt.

7 April 1972

2365
4/04
1462/8
8/14

0800 Meeting
USAF Academy

1. Status of Project

2. Visit to Switzerland & Seveso (See Outline)
   Microbial Data by Zeyer
   Biodegradation - May Option

3. Present Efforts
   Biodegradation
   Fate of TCDD
   SHE Monitoring
   Microbial Studies

4. Role of DFCBS
   [redacted] Present
   Future

EGLIN

Animal Study

① Placed-d

1	1500	P-6 225 SouthEast
2	640	P-7 200 South
3	1200	P-7 210 South
4	270	P-7 200 South
5	440	P-9 200 South

Plant Study

Soil Study

			Mean	1974	1978
1	200	440	804		
2	420	610	454		
3	420	710			
4	530	1200			
5	700	[illegible]500			

2, 7 - dichlorodibenzo dioxin

2,3,7,8

Associated

Herbicide Orange & Its Dioxin

Toxicology and Environmental

Fate.

PLOT #		2,4-D	2,4,5-T	TOTAL	
162	H-13	1	3	4	(11)
	H-14	57	287	344	(650)
394	H-25	1	7	8	(90)
	H-26	48	392	440	(1600)
586	H-37	14	9	23	(14)
	H-38	17	21	38	(200)
	H-39	1040	1303	2343	(6600)

Herbicide

H-39	2343	(6600)
H-44	4026	(7400)
H-54	2270	(6900)

VT 21 October 1977

Trip Report - USAF Academy CO and Pullman WA

Chief, OL AA USAF OEHL
Commander, USAF OEHL
IN TURN

1. Place: Department of Chemistry and Biological Sciences, USAF Academy CO and Department of Soils and Agronomy, Washington State University, Pullman WA.

2. Inclusive dates of travel: 25-30 Sep 1977.

3. Person making trip: Capt Alvin L. Young.

4. Primary mode of transportation: Commercial air.

5. Purpose of trip: To attend Herbicide Orange Conference at USAF Academy and review Herbicide Orange biodegradation contract at Washington State University.

6. Persons contacted: (See attached list).

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(8) Biodegradation plots that were treated with activated coconut charcoal prior to subsurface injection of Herbicide Orange still have significant herbicide and TCDD levels four years after treatment when compared with non-charcoal containing plots. Chemical recovery studies in the laboratory have shown that soils containing charcoal must be extracted for eight hours with benzene rather than the usual procedure of a 30 minute extraction with hexane/acetone in order to recover the TCDD.

b. The Air Force Academy Herbicide Staff have indicated their interest in continuing the program on Herbicide Orange through FY78. They propose supporting the Gulfport - Johnston Island Storage Site Study through the analyses of all soil samples for microorganisms. In addition they are very interested in a program to determine the feasibility of enriching selected field sites with microbes capable of degrading TCDD. Captain Cairney is a microbiologist and is currently the project leader. Under his direction a preliminary study of microorganisms in the soil of Gulfport and Johnston Island has been completed. He has isolated a number of species that could be evaluated for their ability to degrade TCDD. LtCol Bainter and Maj Bomar, both Ph.D. radiation biologists, are interested in examining TCDD degradation by these organisms (using tritium labeled TCDD and ^{14}C-labeled herbicides). When an organism is identified that can degrade TCDD, the most favorable conditions for its activity will be defined. Attempts will then be made to culture the species in sufficient quantity to use for enriching field soil samples. The Department of Chemistry and Biological Science have on hand an excellent research staff, facilities, and equipment for this project. It is estimated that in addition to the $12,000 programmed for FY78 for the microbe study, an additional $8,000 would be necessary for labeled compounds and technician support. The Academy team is submitting a proposal for our evaluation and support.

c. From 28 - 30 September, I visited with Dr H. H. Cheng, Dr T. J. Mizuk and Mr J. T. Makja, Department of Agronomy and Soils, Washington State University, Pullman, WA. Dr Cheng is currently completing Phase II of an Orange Biodegradation Study (see Atch 1). Much of the chemical analyses of 2,4-D, 2,4,5-T and phenols is being done by a graduate study, Mr Makja in support of his Ph.D. program (Atch 2). Discussions were held on a proposed continuation of the project (Atch 3) for FY78. Details of this entire program were discussed in a previous trip report outlining FY78 Herbicide Orange Research (26 Aug 77). While at WSU, I was invited to lecture to Dr Cheng's ENVIRONMENTAL SCIENCE Course and to Dr Muzik's WEED SCIENCE Course. Both lectures were on the "Dilemma for Disposal of Herbicide Orange." With the exception of a few final remarks on the actual incineration (e.g., efficiency, dates of incineration) the presentations were from a paper, same title, cleared by SOFOI and given in Helena, Montana, 16 Sep 75 at a conference on "Advancements in Pesticides."

d. This TDY gave me an opportunity to oversee the herbicide work by both the USAF Academy and Washington State University. Although, Orange has been incinerated, the projects by these two universities will provide valuable data in support of the reclamation of the Gulfport (NCBC) and Johnston Island storage sites. I recommend continuation of both projects during FY78.

ALVIN L. YOUNG, CAPT, USAF, Ph.D.
Chemist/Biologist Consultant

6 Atchs
1. Phase II Report
2. Rsch Report for HO
3. Phase III & IV
4. List of Persons Contacted
5. Table 1
6. Table 2

22 Dec 1977

Mr. John Davidson
Technical Advisor
Ag-Organics
Dow Chemical USA
P.O. Box 1706
Midland, MI 48640

Dear John:

Henry Dishburger sent me the results of the 24 soil samples that Dow so graciously analyzed in support of our research program on TCDD. Thank you so much for your assistance in this project. Permit me to share with you the significance of some of data.

dissipation of ~~f~~ many acids follows a first-order reaction, meaning that at any time the rate of herbicide loss is proportional to its concentration in the soil.

Microbial degradation is known to play a significant role in the dissipation of most herbicides in soil. However, other processes such as volatilization, adsorption, leaching, etc. may limit the availability of the herbicide for biodegradation.

3. Concentration of TCDD in field soils treated with a 3.7 ppm TCDD formulation of the n-butyl esters of 2,4-D or 2,4,5-T degraded 5 half-lives during a 4 year period (1972-1976). Therefore, the ~~TCDD~~ $T_{1/2}$ for TCDD in Alsea dosed soil was ~~300 days~~ approximately 290 days.

2360
4
14C

4

Rapid degradation for 2-3 weeks

C^{14}-TCDD

Removal from media but no breakdown

207
188
340

Storage Time
1973 – 30 100
1977 – 24 110

Chicago – Vet. Administration
Col Lanely

CHARCOAL

517-636-5177
Dick Hummel

F5 B52 A10

FATE OF HERBICIDE ORANGE IN THE SOIL

Interim Report-Phase II

May, 1977

Most of the research activities during the past six months have been in the laboratory, with the initiation of experiments on the adsorption-desorption, leaching, and degradation of 2,4,5-T in the soil. There was, on the other hand, very little action related to the field lysimeter study. Because of the prolonged dry period over the winter months, plus the lack of severe weather, little had happened in the field. We delayed the sampling of the lysimeters until April and decided to postpone the initiation of the summer series of lysimeters until August in order to characterize the seasonal factors better. Reason for this modification of research plan will become obvious from the discussion of the laboratory data.

The purpose of the initial series of laboratory studies was to become acquainted with the various research techniques, to compare our findings with existing literature values for verification of our procedures, and to discover any discrepancies or gaps in the existing information. Since we already have a wealth of background information on 2,4-D, most of the experiments in this initial series were conducted with 2,4,5-T at normal to low levels of treatment concentration. Several reports by O'Connor et al. (Soil Sci. Soc. Am. Proc. 38:433, 1974; J. Environ. Qual. 5:375, 1976) have been particularly pertinent in our comparison studies, since they used the Palouse soil in their studies. The Freundlich k adsorption constants we obtained for the Glendale soil were comparable to those by O'Connor et al., but those for the Palouse soil were higher than theirs. Whereas 2,4,5-T was desorbed readily from the Glendale soil (with 67, 74, 100,and 100% desorbed at 0.2, 0.7, 10.2, and 45.2 ppm after 5 desorptions), desorption was much less reversible in the Palouse soil (with 21, 20, 32, and 33% desorbed at the same concentrations). The predictive model by O'Connor et al. worked well for the Glendale soil, we were interested in the applicability of this model to a soil with very different desorption pattern. Preliminary leaching study on the mobility of 2,4,5-T in a column of Palouse soil under saturated conditions showed that more than 50% of the 2,4,5-T in the soil, after leaching of 10 pore volumes of water, remained in the top 3 cm of the soil column. Even though unsaturated flow may improve the leaching efficiency, the mobility of 2,4,5-T in the Palouse soil appears to be limited. We will be developing more data to test the validity of the model used by O'Connor et al.

We have also been conducting an incubation experiment to determine the degradation of 2,4,5-T in the soil at two concentrations under saturated or field capacity conditions. The pattern of 2,4,5-T degradation appeared to differ from that of 2,4-D in that there was no exponential take-off of the degradation rate of the former. Whereas it was almost indistinguishable between the degradation rates of chain-labeled vs. ring-labeled 2,4-D, the side-chain of 2,4,5-T appeared to degrade faster than the ring. Also more 2,4,5-T degradation occurred in soil at field capacity than at saturation.

The preliminary data from the laboratory already indicate that some modification of our research plan may be necessary. We will need to obtain more data on the adsorption-desorption of both 2,4,5-T and 2,4-D in all three soils, particularly at high rates of application. Similarly, we need to characterize the mobility of these two herbicides in the three soils under study. Particular focus of our attention will be the mobility of 2,4-D and the persistence of 2,4,5-T.

On our agenda for research this summer include the following activities:

1. Continued verification and improvement of procedures for extraction and analysis of HERBICIDE ORANGE in the soil.

2. Periodical sampling of the lysimeter soils and determination of the herbicide contents.

3. Preparation of ^{14}C-labeled butylesters of 2,4-D and 2,4,5-T for the incubation studies.

4. Assess the degradation of HERBICIDE ORANGE in the three soils under laboratory conditions using ^{14}C tracer technique.

5. Conduct additional experiments on the adsorption-desorption and mobility of HERBICIDE ORANGE in the three soils.

6. Initiate the summer series of outdoor minilysimeters in August.

7. Initiate studies on the extractability and identification of herbicide metabolites -- e.g., 2,4,5-trichlorophenol.

It is the goal of Phase II to develop the methodology and to define the research emphasis. We anticipate that much of this goal will be realized by September, 1977.

HERB

HERB/LIME OR FRUIT

HERBS + CHILI POWDER

0-15cm 315 ppt

120

375 ppt

RESEARCH REPORT FOR HERBICIDE ORANGE

Most of the summer work was devoted toward developing a reliable and sensitive chemical analysis for Herbicide Orange. The present status may be evaluated in terms of the analytical technique itself.

Extraction: We have been experimenting with a Sephadex anion exchange gel for extracting herbicide Orange from both water and soil from a 0.2 N NaOH water or soil extract. Presently, extraction efficiencies for both 2,4-D and 2,4,5-T have been greater than 70% for the Palouse silt loam, although we believe improved extraction efficiencies are still possible. To monitor the efficacy of the Sephadex before anion exchange, 2,4-D and 2,4,5-T can be spectrophotometrically characterized via ultra-violet light absorption in both acid and alkali media. Results presently indicate that a cleanup step before esterification will probably not be necessary as in most chemical analyses.

Esterification: To esterify the acid forms of 2,4-D and 2,4,5-T to the n-butyl ester form for determination on the gas chromatograph, two techniques were tested, diazoalkylation and boron trifluoride/n-butyl alcohol. At this time, diazoalkylation appears to be better adapted for routine analyses, although both techniques appear to give approximately equal yields. Within a few weeks we plan to esterify our stock solutions of C-14 labelled 2,4-D and 2,4,5-T for use in a laboratory incubation study scheduled for November. Thirty four individual incubation apparatuses have been constructed for the above experiment.

Gas-liquid chromatographic determinations: GLC analyses were performed on

ATCH 2.

columns packed with 5% SE-30 on Chromosorb W/DMCS, which gave satisfactory separations of the 2,4-D and 2,4,5-T butyl esters from our supply of Orange. Due to difficulties experienced with our tritium GLC detector, we are in the process of upgrading our system by installing a $2000 Ni-63 electron capture detector, which has the advantages of an enhanced linear range and resistance to herbicide overloading. This addition should improve our GLC capabilities considerably.

Field experiments: We are continuing our field studies as last reported. A third soil sampling was recently taken from the Palouse-Wyoming mini-lysemeters installed last winter, while a summer set of 12 mini-lysemeters for the Palouse-Mississippi soils were installed and herbicides applied. Soil samples were collected from these plots two days later, and stored. We are also preparing the water extraction equipment for the mini-lysemeters. Hopefully, there will be sufficient moisture in the early spring for water samples after winter precipitation.

Joseph T. Majka
H. H. Cheng

September, 1977.

Phase III - 1 October, 1977 to 31 July, 1978.

Work plan:

1. Complete the second year minilysimeter study of the fate of Herbicide Orange under field conditions.

2. Complete the laboratory degradation study using ring-^{14}C or chain-^{14}C 2,4-D or 2,4,5-T butyl esters in the three soils.

3. Initiate a new series of degradation study under controlled environment on Mississippi soil only with emphasis on metabolite identification and possibly the rate of metabolite degradation.

Phase IV - 1 August, 1978 to 30 June, 1979.

1. Complete all analyses for the various degradation studies.

2. Conclude the metabolite identification and degradation study.

3. Complete a Ph. D. dissertation.

Attach 2.

PERSONS CONTACTED

LtCol Hugh Bainter, DFCBS, USAF Academy
LtCol Orwyn Sampson, DFCBS, USAF Academy
Maj John Bomar, DFCBS, USAF Academy
Capt William Cairney, DFCBS, USAF Academy
Capt Randall Gaseor, DFCBS, USAF Academy

Dr H. H. Cheng, Professor of Soils, Washington State University
Dr T. J. Muzik, Professor of Agronomy, Washington State University
Mr J. T. Makja, Graduate Student, Dept of Agronomy and Soils, Washington State University.

Corn
+
Sorgum silage 25-30 Ton / Acre

Wheatgrass - 1 Ton/Acre

Native grass - 3/4 Ton/Acre

Alfalfa - 1 3/4 - 1 1/2 - 1 - 3/4 Ton/Acre

TABLE 1. Concentrations of TCDD, parts per trillion, in the Herbicide Orange Biodegradation Plots, AFLC Test Range, Utah, four years after application*.

DEPTH (CM)	ORIGINAL RATE OF ORANGE HERBICIDE APPLIED		
	1,120 kg/ha	2,240 kg/ha	4,480 kg/ha
0-15	650	1600	6600
15-30	11	90	200
30-45	-**	-**	14

* Samples collected 6 November 1976. Plots established Oct 1972.

** Samples not analyzed

TABLE 2. Influence of long-term freezer storage on TCDD content of two Eglin AFB, FL soil samples

COLLECTED, DESCRIPTION	ANALYZED MAY 1973	ANALYZED OCTOBER 1976
GRID 1, TA-C52A (0-15 cm)	30	24
Biodegradation Plots, Plot 6 (0-15 cm)	100	110

TABLE 1. Mean Concentration, ppm, of Herbicide in Metal Scrapings Collected From M/T Vulcanus, 3 Oct 77, Panama.

Ship Location	2,4-D Herbicide			2,4,5-T Herbicide			Total Herbicide
	Acid	Ester	Sub Total	Acid	Ester	Sub Total	
2C/Cover	43,000	7,250	50,250	41,500	8,500	50,000	100,250
3C/Wall	2,070	660	2,730	1,700	910	2,610	5,340
3C/Bottom	1,670	510	2180	1,480	700	2,180	4,360

TABLE 2. Theoretical concentration, μg/gm, of TCDD in metal scrapings as calculated from levels of Orange Herbicide detected in samples.*

Ship Location	Total Orange Herbicide, PPM	Theoretical Conc. TCDD, μg/gm Metal
2C/Cover	100,250	0.2
3C/Wall	5,340	0.01
3C/Bottom	4,360	0.009

TND

0.4 μg/gm

1.5 μg/gm

WSH 0.075 – 0.3 μg/gm

* Assumption: Mean TCDD concentration in Orange Herbicide = 2 ppm.

1 July 1977

Mr John Davidson
Ag-Organics Department
Dow Chemical USA
P.O. Box 1706
Midland, MI 48640

Dear John

Under separate cover, I have sent Mr Don Erwick, 24 soil sample for TCDD analysis (see attached). As you will note these samples are from our biodegradation plots or from Test Area C-52A. They were carefully selected to provide the maximum amount of data on (1) the soil persistence and/or degradation rate of TCDD in three soil types, (2) the leaching of TCDD in soil profiles, (3) an assessment of degradation potential of samples in storage in the absence of presence of phenoxy herbicides, and (4) the confirmation of analysis by another laboratory.

I sincerely appreciated your assistance in providing for the analysis of these samples. I will provide a brief report to you on the results.

As of 15 July 1977, I will be affiliated with the USAF Occupational and Environmental Health Laboratory, Brooks AFB, TX 78235. I will call you when I get settled on station. Again, my thanks to you for all your assistance to me during my tenure at the Air Force Academy.

Sincerely

ALVIN L. YOUNG, Capt, USAF, PhD
Associate Professor of Biological Science
Dept of Chemistry and Biological Sciences

1 Atch
List of Samples

USAFA
TCDD BIODEGRADATION STUDY
SOIL SAMPLES
SUBMITTED TO DOW CHEMICAL USA
15 JUNE 1977

USAFA SAMPLE #	DESCRIPTION
1	Garden City, KS, 22 Mar 74, 4000 lb/A, 0-6
2	Garden City, KS, 14 Jan 75, 4000 lb/A, 0-6
3	1.5 ppm TCDD, Kansas soil, formulated 22 Jan 75
4	1.5 ppm TCDD plus 10,000 ppm Orange, Kansas soil, formulated 22 Jan 75
5	H-13, AFTC Test Range, UT, 5 Nov 76, 1000 lb/A, Rep I, Hole 3, 6-12
6	H-14, AFTC Test Range, UT, 5 Nov 76, 1000 lb/A, Rep I, Hole 3, 0-6
7	H-25, AFTC Test Range, UT, 5 Nov 76, 2000 lb/A, Rep I, Hole 2, 6-12
8	H-26, AFTC Test Range, UT, 5 Nov 76, 2000 lb/A, Rep I, Hole 2, 0-6
9	H-37, AFTC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 4, 12-18
10	H-38, AFTC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 4, 6-12
11	H-39, AFTC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 4, 0-6
12	H-44, AFTC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep I, Hole 2, 0-6
13	H-54, AFTC Test Range, UT, 5 Nov 76, 4000 lb/A, Rep II, Hole 1, 0-6
14	0-6" Soil Core, Sites 0-7, Eglin AFB, FL, collected 31 Mar 73
15	Plot #8, 0-6", Herbicide and plastic, Eglin AFB, FL, 21 May 73
16	Plot #10, 0-6", Herbicide and activated carbon plus charcoal, Eglin AFB, [illegible]
17	H-[illegible] Eglin AFB, FL, [illegible] 76, [illegible] 5, hole 3, 12-18
18	H-11, Eglin AFB, FL, 15 Nov 76, plot 5, hole 2, 6-12
19	H-12, Eglin AFB, FL, 15 Nov 76, plot 5, hole 2, 0-6
20	H-17, Eglin AFB, FL, 16 Nov 76, plot 6, hole 1, 0-6
21	H-[illegible], Eglin AFB, FL, 18 Nov 76, plot 7, hole [illegible], 0-6
22	H-41, Eglin AFB, FL, 18 Nov 76, plot 9, hole 2, 0-6
23	H-41, Eglin AFB, FL, 18 Nov 76, plot 10, hole 2, 6-12
24	H-[illegible], Eglin AFB, FL, 16 Nov 76, plot 10, hole 2, 0-6

SAMPLING DATES

JOHNSTON ISLAND —

6 NOV — 10 NOVEMBER

DEPART COS - 6 NOV (SUNDAY)

ARRIVE JI - 7 NOV

WILLIAM J CALDWELL

153-34-3903

CAPTAIN USAF BSC

USAFA / DFCBS

SECURITY DATA - SECRET

DEPART JI - 10 NOV

ARRIVE COS - 11 NOV.

28 NOV — 2 DEC ALSO AVAILABLE.

DIURON - 2%/90 DAYS

2,4,5-T - O'CONNER - New Mexico

Adsorption Constants.

Trichlorophenol —

PLASTIC TUBES - 2,4,5-T Bibliography TCP Articles

Historians

Palouse

Grain, Field Crop.

Age of Spill?

Feasibility of the Soil environment for selected Microbes for degradation of TCDD

Proposed Microbial Uptake Study of TCDD.

Selected Organisms, chosen on the basis

LTCol Hugh T. Bantle
Maj John Bonner

Confirm the degradation of TCDD by microorganisms. Synthesize labels for each compound. —

300 curies/mole — ∴ Tritium label
Carbon label

Prefer the tritium label on the herbicide and the C^{14} labell on TCDD

300 millicuries/mole

Liquid Chromatographic unit with fraction collector and strip chart recorder + columns packing.

∴ Equipment	5,000
Label compounds	4,000

Temporary overhire

Technician	12,000

TCDD - PPT Oct '72
UTAH Nov 76

	1000 lb/A	2000 lb/A	4000 lb/A
0-6"	650	1600	6600
6-12"	11	19	200
12-18"			14

FIELD Replication

6600
7400
6900

7.1 ppm

FREEZER Samples

	1973		1977	1977
EGLIN	30	07	24	x
	100		110	—

Standards

	D	I
~~500ppb~~		
~~1ppm~~		
2 ppm	19.0 ±3.7	62.3 ± 6.8
1.5ppm	12.7 ±2.9	47.3 ± 6.6
1ppm	7.9 ±2.4	29.9 ± 5.9
.5ppm	3.6 ± .8	16.9 ± 3.3
.25ppm	1.3 ± .6	7.7 ± 1.2

4-11

1.93

2.4
5000
11.0000

.47000

TABLE 1. Mean Concentration, ppm, of Herbicide in Metal Scrapings Collected From M/T Volcanus, 3 Oct 77, Panama.

Ship Location	2,4-D Herbicide			2,4,5-T Herbicide			Total Herbicide
	Acid	Ester	Sub Total	Acid	Ester	Sub Total	
2C/Cover	43,000	7,250	50,250	41,500	8,500	50,000	100,250
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TABLE 2. Theoretical concentration, mg/gm, of TCDD in metal scrapings as calculated from levels of Orange Herbicide detected in samples.*

Ship Location	Total Orange Herbicide, PPM	Theoretical Conc. TCDD, mg/gm Metal
2C/Cover	100,250	0.2
3C/Wall	5,340	0.01
3C/Bottom	4,360	0.009

* Assumption: Mean TCDD concentration in Orange Herbicide = 2 ppm.

24" – Buildings –

CLAY - Mont.

Impact

SAND

1944-45] SITE PREPARED

Dam

Mr. Boland

Chief Engineer

PASCA

Silt Sandy loam

LAKELAND

Geology – Soil Analysis 4 ft. to water

Hydrology –

Soil Compaction – [aquifers –]

(2) Ease of working soil. (compaction)

(3) Reduction of area by piling

(4) Do you increase hazard of contamination by disruption of site

(5) What is the water level

(6) what are the microbial levels on the hard pan:

Glass Jars — Bill Carney

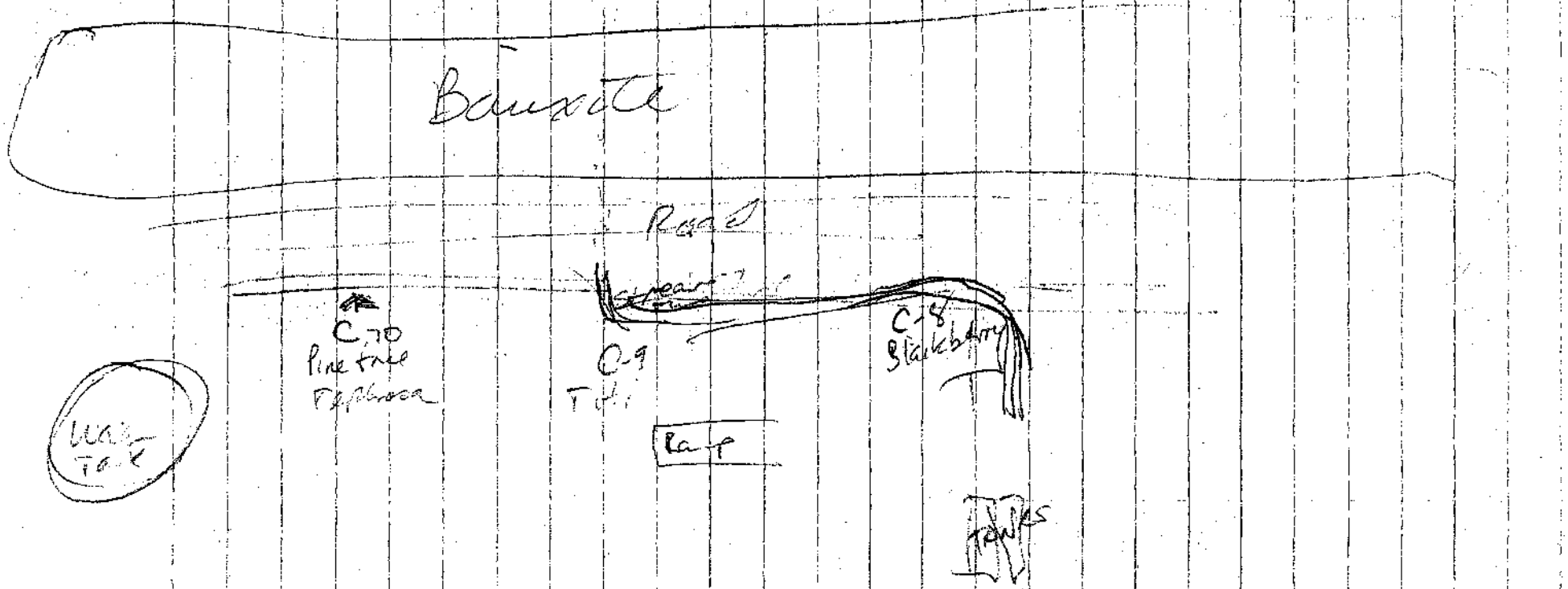
How much leaking?
How much can we expect to be spilled?

(A) Public health considerations with/without site decontamination

(B) Legal aspects.

IF alternate locality, how much area should be fenced?

Till & establish vegetation
to ground water
Contam


Road
C-10
Pine tree
C-9
C-8
Blackberry
Raft
TWIGS

A-1 Plantain

A-2 Thistle

A-3 Blackberry

A-4 Euphratorium

A-5 Dandelion

A-6 Milia

A-7 Spurge

A-8 Small Thistle C

A-9 Euphratorium

A-10 Horseweed

TRACK

B-1 T.H.

B-2 Longleaf Pine

B-3 Composite Seedlings (Opuntia Cactus)

B-4 Euphorbia

B-5 Fern (By last row of Trees)

B-6 ~~Horse weed~~ Nettle (west of Tank Trail Trees).

B-7 (Ramp) Dayflower/Nettle

B-8 - Heliconia (~~Ragweed~~ Ragweed Post 1994)

B-9 Croton (North of Fire on A Road)

B-10 Blackberry (Nettle) B road south of Barn

C-7 (Greenwood & Banado intersection) Plantain
Blackberry

C-6 Blackberry – Halfway down on
west side of Co. Campus

C-5 Ilex bush (curled leaves)
Plantain Near Bldg 8

C-4 Composite. Guide wire post near
2024 – 7th & McKinney

C-3 Blackberry on slope SE of Bld 221

C-2 Bottom end 7th Bear Neg
Young blackberry & cotton

C-1 Giant curl dock – intersection of Truck
& west end 7th
End of Bench

Degradation of Orange

1. Method of Solubilizing
2. Method of Degradation - Enzymatic
3. Conditions of Degradation
   - Sewage } pH
   - Vat } Water
4. Sequence / Time of Degradation
5. Products - Biological Evaluation
   - Phytotoxic ?
   - Animal ? - Aquatic ?
     - Rats ?
6. Pilot Plant Operation ?
   OR Immediately Available Plant Operation.
   Time of gear-up ?

Houston Oaks Hotel

Galleria-Post Oak, Houston, Texas 77027 (713) 623-4300. Cable: Westhotels, Houston

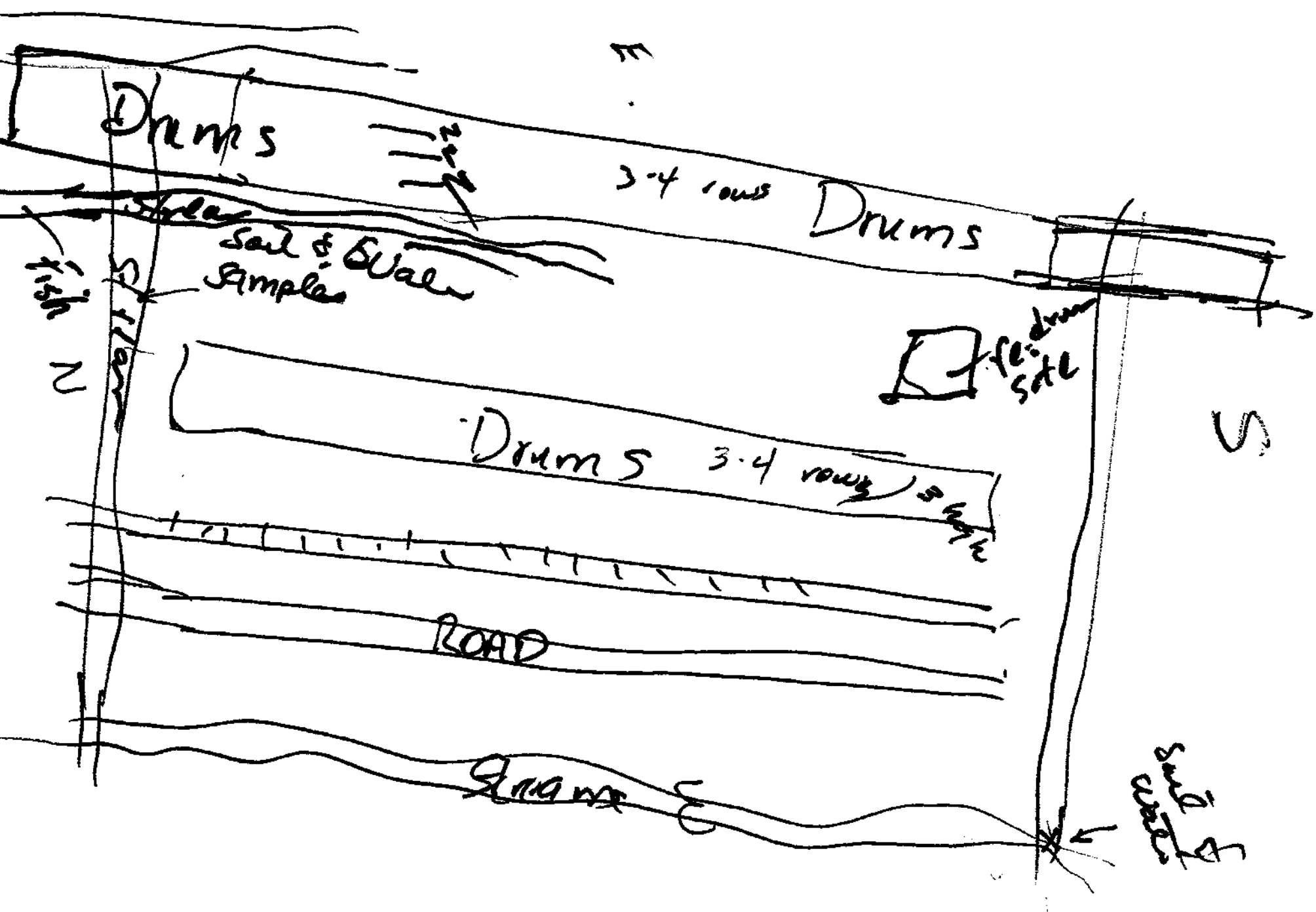
Dear Dr. Tierney

The ~~enclosed~~ analytical reports outline procedures for the analysis of TCDD in biological ~~tissues~~. Also, attached please find a current list of all USAF samples submitted to Wright State University from 2 Sept 1975 – 7 Sept 1976. Note that our records agree with yours except for ~~six~~ five (5) Eglin AFB soil samples collected 5 Apr 75 and submitted to you on 2 Sept 75. Please check your records on these samples.

I will be at WPAFB on 21 – 22 Oct 76 and look forward to discussing analysis of our samples with you at that time.

Many thanks &

A. Young


Drums
3-4 rows
Drums
Soil & Water
Samples
Drums
3-4 rows
LOAD
Soil & Water Samples

Vegetative Survey
Soil Survey — Fire House

(1) How large of an area must we be concerned with —
A. Alternative as to site ?

(2) How deep do we sample.

(3) What levels of herbicide are acceptable.

(4) What levels of TCDD are acceptable

(5) Is the water & soil leaving the area contaminated, IF so, how far.

(6) ON site contamination of animals — Not Likely
Fish down stream ?.

(7) MASTER PLAN
Long term Bio - monitoring programs.

IF SITE CONTAMINATED ABOVE ACCEPTED LEVELS
What then:

(1) Area Ten Acre Feet

6,000,000 lbs/Acre Ft = 60,000,000 lbs
30,000 TONS

440,000 cu Ft
16,300 cyd^3

138 lbs/ft^3 = 60,000,000 60.7810^6

[600 Railcars] 30,000 TON 30.4 X10^6 TON