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# TECHNICAL

## Report

INCINERATION OF ORANGE HERBICIDE

July 1972

EHL(K) 72-7

USAF ENVIRONMENTAL

HEALTH LABORATORY

KELLY AFB, TEXAS



USAF ENVIRONMENTAL HEALTH LABORATORY (AFLC)

UNITED STATES AIR FORCE

KELLY AFB, TEXAS 78241

INCINERATION OF ORANGE HERBICIDE

July 1972

EHL(K) 72-7

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## I. INTRODUCTION

The USAF EHL/K was first requested in 1970 to provide guidance concerning the disposal of excess herbicides in USAF inventories by the Aeronautical Systems Div/AFSC at Wright-Patterson AFB OH. The various methods of disposing of agents Orange, Blue and White were investigated in detail. The Laboratory recommended disposal by incineration in June 1970 (Appendix C-1, pg. 38). SAAMA/SF was directed in August 1971 to write an environmental statement and a statement of work for the disposal of Orange by incineration; this Laboratory was asked to assist (Appendix F-1, pg. 95). Assistance by the Laboratory has included assessing the capabilities of various commercial facilities to destroy Orange herbicide; review, with comments, the content of two draft environmental impact statements and a statement of work; the development of a protocol for the monitoring of the normal butyl esters (NB) of 2,4-D, 2,4,5-T and TCDD (2,3,7,8-tetrachlorodibenzo-p-dioxin) discharged during incineration; and, as a by-product of these latter studies, preliminary data concerning the efficiency of incineration as a means of destroying Orange. The Laboratory has also assisted HQ USAF, the University of Mississippi and the USAF Rocket Propulsion Laboratory, Edwards AFB, CA, in interpreting and planning investigations concerning the pyrolysis of Orange.

## II. DISCUSSION

### 1. Monitoring the emissions of incinerators burning Orange herbicide:

a. The necessity of monitoring the emissions of incinerators burning Orange was recognized in August 1971. This recommendation was repeated in September, October and November of that year (Appendices C-2, 3 and 4, pgs 44, 46, 53). SAAMA/SF was provided with a protocol suitable for monitoring incinerators burning Orange in February 1972 (Appendix B).

b. The scope of the investigation which generated this protocol included:

(1) Determining the feasibility of using Gas Liquid Chromatography (GLC) alone to analyze effluent combustion gases and scrubbing blowdown water for the herbicide esters and TCDD-contaminant in Orange herbicide.

(2) Development of efficient methods of extracting the NB esters and TCDD from gaseous and water discharges as they emerge from an incinerator.

c. A small continuous burning, flow-through incinerator was built which closely approximated the fuel/air aerosol injection method, dwell times, air/fuel ratios and temperatures of the commercial facilities under consideration. Orange herbicides containing both high (~14 ppm) and low (~0.1 ppm) of TCDD was incinerated in this apparatus. The protocol for monitoring the discharge of large incinerators was developed using the discharges of this model under various conditions of incineration. This protocol is contained in Appendix B, pg. 29.

d. Data concerning the efficiency of Orange and TCDD pyrolysis was necessarily generated in acquiring the above information. These data are of special interest since, at this time, they are the only pyrolysis data of Orange available which approximates the combustion conditions in the commercial incinerators considered in the candidate impact statement.

e. A detailed discussion of this incineration data is presented in Appendix A, pg. 8. Some of the significant results include:

(1) Simple extraction and cleanup procedures, coupled with GLC analysis were successful in monitoring the NB esters and TCDD discharged from incinerators in part per billion concentrations. The actual cost of this monitoring program will be dependent on contractor capability, equipment on hand and other variables. The simplicity of the monitoring protocol (Appendix B, pg. 29) will result in relatively reasonable costs.

(2) The destruction of the NB esters and TCDD in the model incinerator at 920°C, 2-3 second dwell times, and 150-180 percent stoichiometric air exceeded 99.999 percent for the esters and 93 percent for the TCDD. Total discharges of the combined esters ranged from 8.0-50.0 ppb (parts per billion) in the untreated gas discharges. The TCDD discharged when burning Orange containing high concentrations of TCDD were 3.0 and 18.0 ppb. Detailed data is presented in Appendix A, pg. 8.

2. Written consultations concerning the Draft Environmental Impact Statement:

a. Various AF agencies have been provided with guidance concerning the incineration of agent Orange since June 1970. Table 1 documents those suggestions and policies made which concerned the preparation of the draft environmental impact statement and the subsequent comments of State and Federal agencies requiring these actions. The individual written consultations are contained in Appendices C and E. The State and Federal comments are contained in Appendix D.

3. Consultations concerning research performed by other laboratories investigating the pyrolysis of Orange herbicide:

a. Consultation support has been given to SAAMA/SF concerning ongoing research at Mississippi State University (MSU) and an unsolicited research proposal of the Transvaal Corporation, Jacksonville, AR. Similar consultation was given to the USAF Rocket Propulsion Laboratory (USAFRPL) concerning contract research with the Marquardt Corporation, Van Nuys, CA. Written summaries of these consultations are contained in Appendix E.

b. The Environmental Assessment Statement concerning the incineration of Orange herbicide should contain three additional types of information to allay public and scientific concern about this action:

(1) Laboratory data indicating that the NB esters and TCDD will be thoroughly degraded during the incineration process (Appendices C-2 and C-3).

(2) A monitoring program throughout the burning operation which demonstrates adequate pyrolysis is actually occurring (Appendices C-2 and C-3).

(3) Laboratory data identifying the major intermediate pyrolyzates of Orange and TCDD incineration (Appendices C-3, E-1 and E-3). These intermediate pyrolyzates are not to be confused with the ultimate pyrolyzates of CO<sub>2</sub>, HCl and H<sub>2</sub>O.

c. The data and protocols contained in Appendices A & B satisfy the need for a monitoring program (assuming toxic intermediate pyrolyzates which require additional monitoring are not discovered). The pyrolysis data, also contained in Appendix A, coupled with data from the MSU studies, indicate pyrolysis of Orange esters and TCDD is feasible. However, none of the contract studies will supply the needed intermediate pyrolysis data. Such data is currently beyond the instrumentation of this Laboratory to provide as was stated in December 1971 (Appendix F-2, pg. 96). The MSU and AFRPL research efforts will not supply this information (Appendices E-5, pg. 86 and E-6, pg. 92).

4. Appendix F contains communications authorizing this Laboratory to support SAAMA/SF and defines the limits of this support.

### III. CONCLUSIONS

These data indicate:

1. Incineration of Orange herbicide at 920°C with 3-4 second dwell times efficiently destroys the NB esters of 2,4-D and 2,4,5-T and TCDD (Appendix A, pg. 9). Disposal of Orange by commercial incineration is feasible and safe if performed in a suitable facility under strictly monitored conditions. A suitable monitoring program for the herbicide esters and TCDD has been developed (Appendix B, pg. 30).

2. The emissions of NB 2,4-D and 2,4,5-T and TCDD resulting from the incineration of Orange can be monitored economically in the ppb range. Incineration of Orange, therefore, becomes a controlled destruction process. If emissions, at any time, exceed EPA and/or State Standards, disposal can be stopped until such standards are met (Appendix B, pg. 30).

3. The two USAF funded contract studies, which are known to the principal investigator, will not provide data identifying the chemical nature of the intermediate pyrolysis products resulting from the incineration of Orange herbicide. These data may be crucial in future negotiations with EPA and State agencies. (See Appendices E-5 & E-6, pgs. 86 and 92).

a. HQ USAF must decide whether to reapproach the concerned Federal and State agencies, without these data, or to obtain it from other sources for inclusion in the final impact statement.

4. The widely held view that the "political" actions and requirements of Federal and State agencies and civilian groups concerning environmental matters cannot be predicted in advance is not necessarily true. Such requirements and reactions are frequently predictable and were predicted. (Table 1). They may be routinely forecasted if the environmental implications of the actions involved are properly understood and evaluated.

#### IV. RECOMMENDATIONS

1. The Monsanto Chemical Company should be requested to re-examine its negative decision concerning the incineration of Orange herbicide. As stated in Table 1 and in October 1971 (Appendix E-1, pg. 72) the Krummrich Plant offers many advantages and should be given, again, every opportunity to bid for this contract and be included on an equal basis with all other potential contractors.

2. The States of Texas and Illinois should be reapproached concerning the incineration of Orange herbicide. Emphasis should be placed on the thoroughness of the combustion process as indicated by the above mentioned Laboratory research; that the actual incinerator will be monitored for herbicide and TCDD discharges, at the parts per billion level; and that all intermediate combustion products are innocuous at the emitted concentrations (as yet unproven); that ultimate incineration by-products will meet all State and Federal requirements as measured by Standard monitoring procedures.

3. The SUE Burner of Marquardt Corp should be considered as a possible destruction method on Johnson Island. See Appendix E-6, pg 92 for a description of this device and a discussion of its claimed capabilities.

Table 1

SUGGESTIONS OF THE ENVIRONMENTAL HEALTH LAB/KELLY,  
SUBSEQUENTLY CONCURRED IN BY FEDERAL AND STATE AGENCIES,  
CONCERNING THE DESTRUCTION OF ORANGE HERBICIDE

<u>EHL/K Suggestion</u>	<u>Appendix, pg.</u>	<u>Date</u>	<u>Confirming Action</u>	<u>Date</u>	<u>Appendix, pg.</u>
1. Incineration is a preferred method of destruction.	C-1 - 38	Jun 70	Federal EPA approved incineration in principle	8 Mar 72	D-1 - 56
2. The environmental impact statement must frankly discuss herbicide and TCDD emissions.	C-2 - 44	Sep 71	Federal EPA	8 Mar 72	D-1 - 56
	C-3 - 46	Nov 71	US Dept of Commerce	18 Feb 72	D-2 - 61
			US Dept of Agriculture State of Texas		D-3 - 63 D-4 - 65
Discussion of these areas provided.	C-3 - 46	Nov 71			
3. The statement must provide information and methods that assure safe emission levels will not be exceeded.	C-2 - 44	Sep 71	Federal EPA	8 Mar 72	D-1 - 56
	C-4 - 53	Oct 71			
	C-3 - 46	Nov 71			
Method for monitoring known Orange constituents and TCDD provided.	B-1 - 30	Feb 71			
4. Scrapping or recycling of drums superior to landfill disposal.	C-2 - 44	Sep 71	Federal EPA	Mar 72	D-1 - 56
	E-1 - 72	Oct 71			
	C-3 - 46	Nov 71			
5. Limits of discharges for NB 2,4-D; NB 2,4,5-T and TCDD in air and water necessary.	C-2 - 44	Sep 71	Monitoring program required by		D-1 - 56
	C-4 - 53	Oct 71	Federal EPA. 8 Mar 72 Limits of discharge for herbicide esters and TCDD not yet mentioned.		

Table 1 (contd)

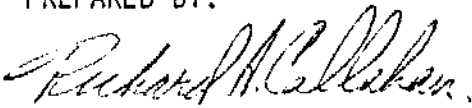
<u>EHL/K Suggestion</u>	<u>Appendix, pg.</u>	<u>Date</u>	<u>Confirming Action</u>	<u>Date</u>	<u>Appendix, pg.</u>
6. Careful preparation of the environmental impact statement is essential and should be favored over "crash programs."	C-4 - 53	18 Oct 71	Unfavorable publicity; adverse State and Federal reactions.	Feb 72	D-1 - 56 D-2 - 61 D-3 - 63
7. The Monsanto Krummrich plant's twenty years experience manufacturing phenoxy herbicides and unlimited resources in analytical chemistry offer distinct advantages over less expert and equipped facilities.	E-1 - 72	21 Oct 71	Statement of Mississippi State to Mr. Moseman concerning the limited analytical capabilities of Rollins Purle, Inc.	8 Mar 72	Oral Statement
			Statement of Ectyl Blair, Corporate Manager R&D Agricultural Chem Div Chemical Comp.	10 Nov 71	E-2 - 75
			State of Texas	14 Feb 72	D-4 - 65
8. Sauget, Illinois site should be considered as an equal facility in the statement not as an alternate facility.	E-1 - 72 C-3 - 46		ibid Para 7 confirming action #1	12 Apr 72	Oral Statement
			Congressional inquiry of Houston League of Women Voters as to why Sauget firm not given equal consideration as a site to burn Orange.		



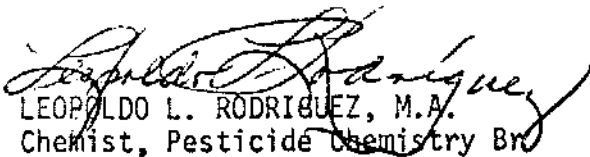
Appendix A

INCINERATION OF ORANGE HERBICIDE IN A  
LABORATORY SCALE INCINERATOR

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## LABORATORY INCINERATION OF ORANGE HERBICIDE

### I. INTRODUCTION

1. This Laboratory was requested by SAAMA/SF (in Nov 1971) to provide a protocol suitable for monitoring the normal butyl esters of 2,4-D and 2,4,5-T and 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) that might be present in gaseous or water discharges from an incinerator burning Orange herbicide. This protocol was provided to SF on 2 February 1972.

2. The scope of this investigation included:

a. The feasibility of using Gas Liquid Chromatography (GLC) alone to analyze combustion gases and scrubbing blowdown water for the herbicide esters and TCDD:

(1) Limits of GLC sensitivity to the ester and TCDD.

(2) Degree of interference with GLC analysis from partial combustion products which might be generated during pyrolysis of Orange.

(3) A protocol for preparing samples for EC, GLC analysis.

b. Development of efficient methods of extracting the NB esters and TCDD from gaseous and water discharges.

(1) Solvent extracting systems compatible with GLC analysis and which efficiently extract and hold the esters and TCDD.

(2) Scrubbing impinger trains which optimize extraction and minimize complexity.

3. A small continuous burning flow through incinerator was built which closely approximated the fuel/air aerosol injection method, dwell time, air fuel ratios and temperatures anticipated in the available commercial facilities. The unit was constructed so that these critical parameters could be varied, thereby producing a variety of incomplete pyrolysis products that might be produced in a large burner. These experiments tested the capability of GLC to detect the esters of TCDD in the presence of a host of contaminants.

4. Data concerning the efficiency of Orange and TCDD pyrolysis was necessarily generated in acquiring the above information. This data is of special interest since, at this time, it is the only pyrolysis data

available which approximates the combustion conditions in the commercial incinerators under consideration.

## II. MATERIALS AND METHODS

1. An all glass, Vycor-Pyrex, tube furnace with scrubbing system was constructed (Fig 1 and 2). The system functioned as follows:

a. Fuel (Orange Herbicide) was continuously delivered at a metered rate (via Hamilton 2.5 ml gas syringe mounted on a Sage Model 350M pump) (Fig 1-17 & 18) to the tip of a blunted stainless steel 22 gauge needle (Fig 2-19). The tip of this needle was sealed in a stainless steel Luer Lock syringe fitting. Compressed air was metered into this fitting via a 0-1 ml/min rotometer (Fig 1-16). The Orange was continuously aerosoled from the tip of the fuel probe (Fig 2-20) into the furnace. The air/fuel mixture was then deflected upwards by a dispersing cup (Fig 1-21). The combusting gases passed directly up and out of the furnace tube. The aerosol injection probe was a 20 gauge stainless steel pudental needle; the dispersing cup was also stainless steel. The Vycor tube within the furnace was 33 cm long with a volume of 156 cc (within the furnace).

b. Heat was provided by a Lindberg Heviduty tube furnace equipped with heating elements capable of operating at 1200°C. Temperatures were continuously monitored by thermocouples located within the furnace on the outside surface of the Vycor incinerator tube; and inside the incinerator tube, four centimeters below the top of the heating element. These thermocouples were used to prevent excessive temperatures from melting the Vycor tube and to monitor the gas temperature just prior to leaving the furnace.

2. All effluent gases passed through a three way ground glass fitting (Fig 1-4) where they were either vented directly to a hood flu or were passed through the scrubbing system.

a. Gases were forced through the system by positive pressure within the combustion tube. This pressure was monitored by a mercury manometer (Fig 1-1).

b. The scrubbing system included three midget impingers; two containing tapered air inlets a third a fritted gas diffuser. Impingers were charged with distilled water or nanograde benzene.

c. The fourth element of the system was a freeze condensing unit. This unit consisted of two pyrex tubes, one sealed within the other in such a way that a space approximately 1 cm wide separated the tubes. Gas entering the bottom of this space was forced to rise around the inner tube and exit through the top port (Fig 1-7). The inner tube was super cooled by filling it with dry ice and acetone.

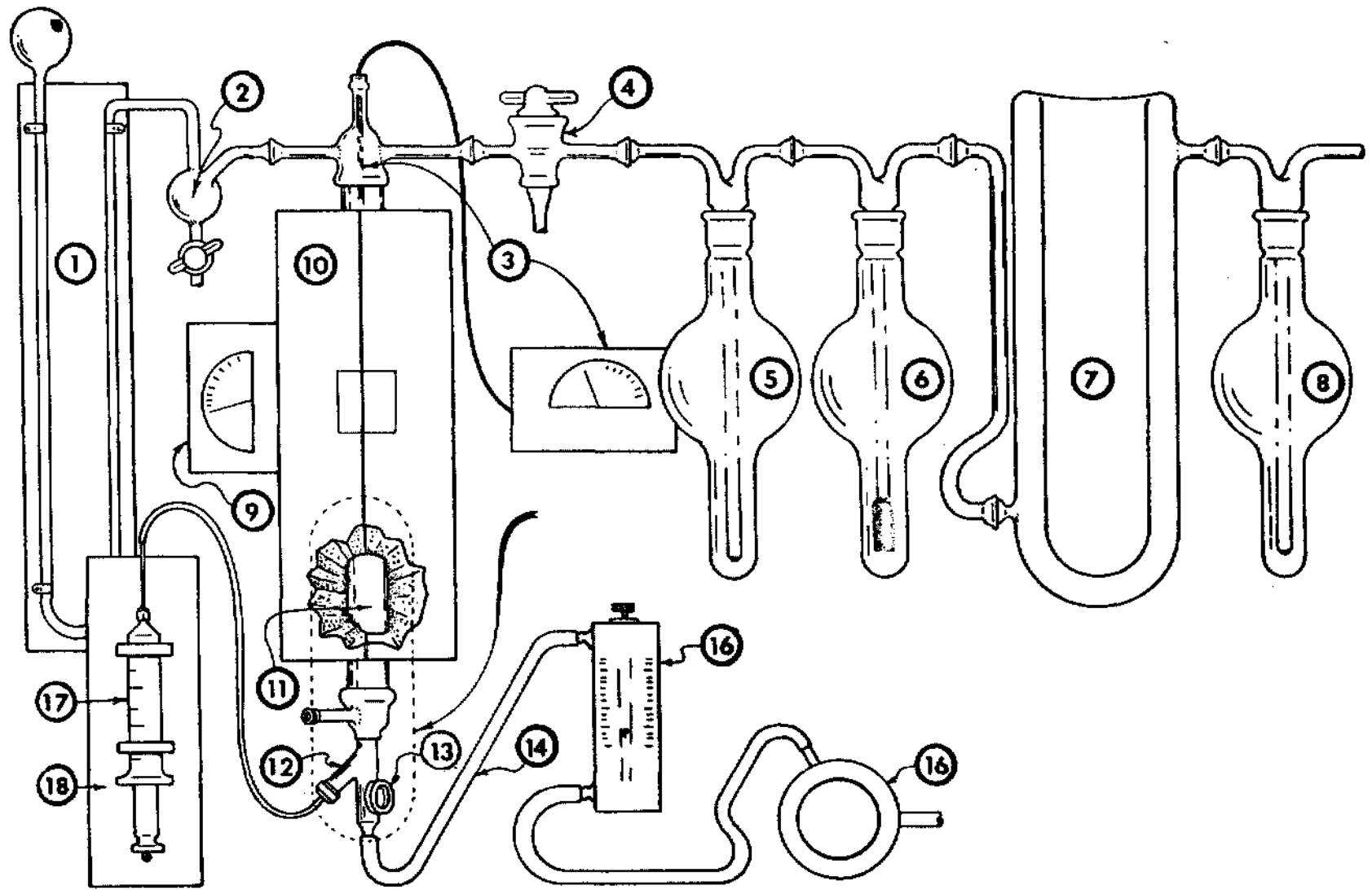


FIGURE 1. Incinerator Scrubbing System

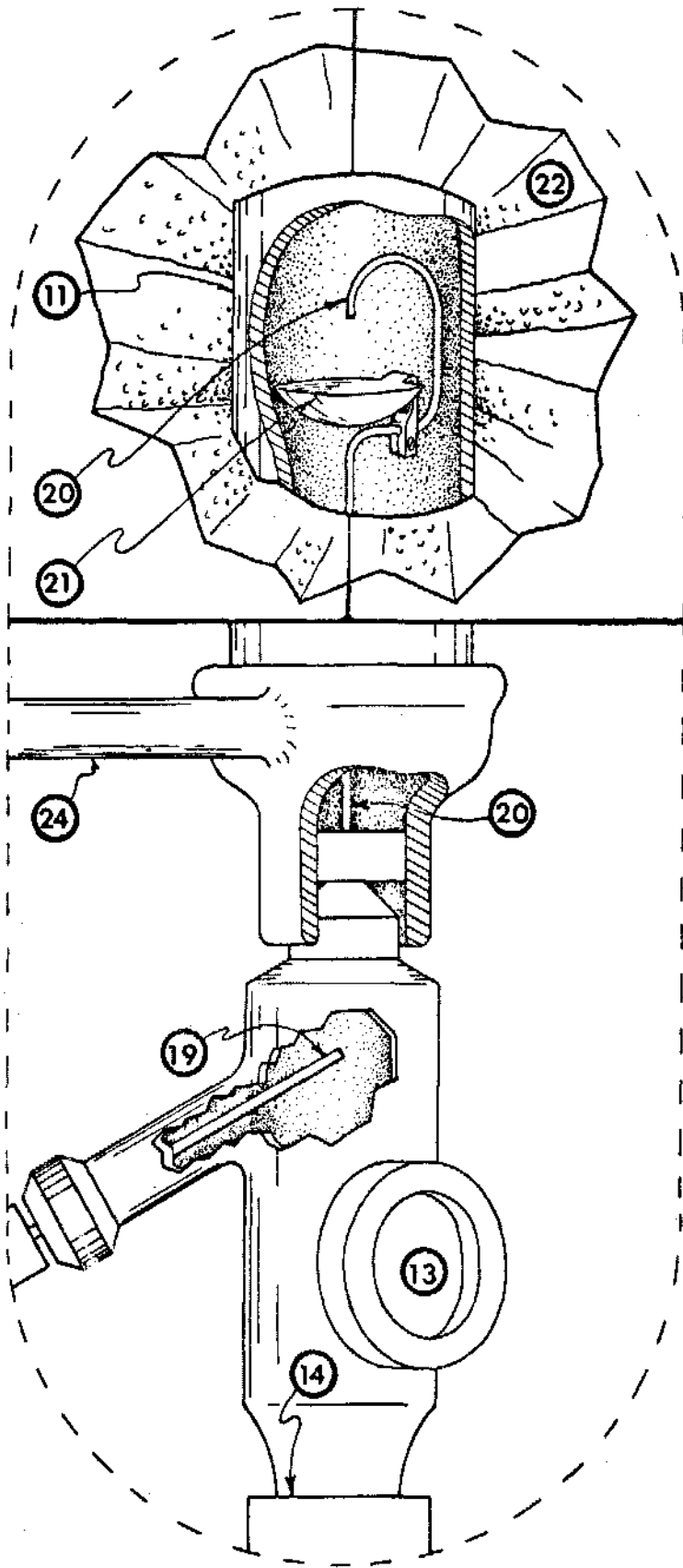


FIGURE 2. Incinerator Air-Fuel Injection System

PARTS IDENTIFICATION FIGURES 1 & 2

- 1 Mercury Manometer
- 2 Pressure Release
- 3 Chromel-Alumel Thermocouple (Gas Temperature)
- 4 Three Way Pyrex Stopcock
- 5 Tapered Impinger
- 6 Fritted Glass Impinger
- 7 Dry Ice Acetone Freeze Condenser
- 8 Tapered Impinger
- 9 Furnace Thermocouple
- 10 Lindberg Heviduty Tube Furnace
- 11 Vycor Glass Tube
- 12 Fuel (Orange) Injection Inlet
- 13 Injection Septum
- 14 Air Line
- 16 Rotometer and Compressed Air Tank
- 17 2.5 ml Hamilton Gas Syringe
- 18 Sage Model 350 Syringe Pump
- 19 18 Gauge SS Blunted Needle
- 20 20 Gauge Pudental Needle
- 21 SS Deflector Cup
- 22 Furnace Insulation

3. The total system was pressure tested for gas leaks before each test run by raising the internal pressure to over 20 cm Hg.

4. All incineration tests followed the following protocol.

a. The three-way valve was set in the bypass position.

b. The rotometer was set to deliver the desired volume of air and the furnace was adjusted so that the effluent air was at the desired temperature.

c. Orange herbicide was then introduced with the air by actuating the previously adjusted Sage pump. The incinerator was allowed to burn for approximately 10 minutes.

d. The volume of herbicide in the Hamilton syringe was recorded and the effluent valve switched so that all gases passed through the scrubbing system. This phase was timed with a stop watch.

e. The syringe pump was turned off, the effluent valve reswitched to the bypass position and the collection time recorded. Orange delivery rates were calculated as microliters ( $1 \times 10^{-6}$  liters; ul) of Orange delivered/time. These rates were reproducible to  $\pm 2.0$  ul/min.

5. The NB esters of 2,4-D and 2,4,5-T and TCDD were determined in each impinger separately by Electron Capture GLC (EC-GLC). The EC-GLC analyses were performed using standard techniques under the following conditions.

a. Clean up of benzene samples - two washes with glass distilled water containing a pinch of sodium bicarbonate followed by an additional water wash. The benzene was dried with sodium sulfate, brought to an appropriate volume of benzene and immediately injected into the GLC.

b. Water samples were extracted three times with equal volumes of benzene and then similarly treated.

c. EC-GLC conditions:

(1) Instrument - Microtek 220 GLC equipped with a Ni63 EC detector.

(2) Column - Six foot glass, packed with 1.5% OV210, 1.5% QF1 on Anakrom Q80/90 mesh.

(3) Temperatures - Injector 225°C, column 195°C, Ni63 detector - 345°C.

(4) Carrier Gas - Nitrogen at 50 ml/min.

### III. RESULTS AND DISCUSSION

#### 1. Limits of analytical sensitivity:

a. The linearity and sensitivity of the Ni63 detector to analytical standards of the NB esters of 2,4-D; 2,4,5-T and to TCDD was determined. The detector response was linear up to 50 picograms (1 pg =  $1 \times 10^{-12}$  gm) of the D and T esters and from 0 to over 100 pg of TCDD.

b. The practical sensitivity of the detector to standard solutions is less than 3.0 picograms of the esters and 7.0 picograms of TCDD.

2. Tables 1 and 2 contain data concerning the physical parameters of the incinerator - scrubbing system during the burning runs. Tables 3 and 4 document the scrubbing efficiencies under various conditions and the recoveries of spiked samples. Table 5 documents the effect of several air/fuel ratios and temperatures on the emission of the herbicide esters and TCDD.

#### 3. Interference of EC-GLC by pyrolysates of Orange herbicide.

a. Figs 3 & 4 present two typical chromatograms of the benzene scrubber solution. The first chromatogram is of the first impinger from Run 8 (8,1), the second is similarly from run 7,3. The first chromatogram is clean - only residues of the NB, T and D esters and TCDD are apparent. Run 8 was a 30 minute burn at 181% air and 945°C effluent gas temperature. The second chromatogram detected five chlorinated pyrolyzates not present in Run 8. Run 7 was supplied with only 71% of the stoichiometrically required air with an effluent temperature of 795°C.

b. This data indicates that at incineration temperatures approaching 1000°C and air/fuel ratios of approximately 150 percent, incomplete combustion products will not interfere with EC-GLC analysis.

(1) This data also strongly indicates that dehalogenation occurs rapidly during the pyrolysis process of the NB esters of 2,4-D and 2,4,5-T. The emission of even ppb concentrations of chlorinated organic pyrolysis products appear remote and would be detected by the monitoring system.



FIGURE 3

EC-GLC Chromatogram

Run 8 Impinger No. 1

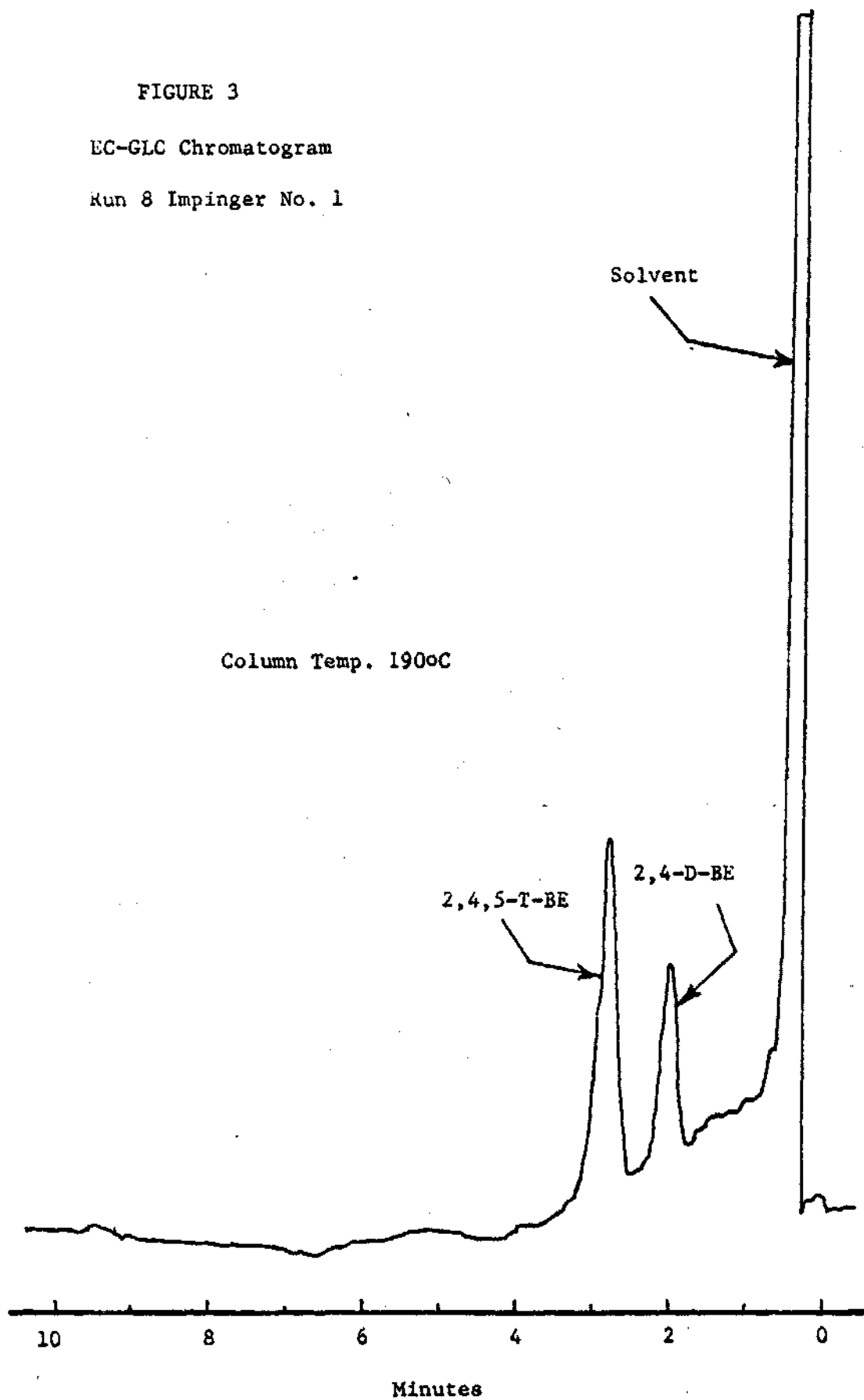


FIGURE 4  
EC-GLC Chromatogram  
Run 7 Impinger No. 3

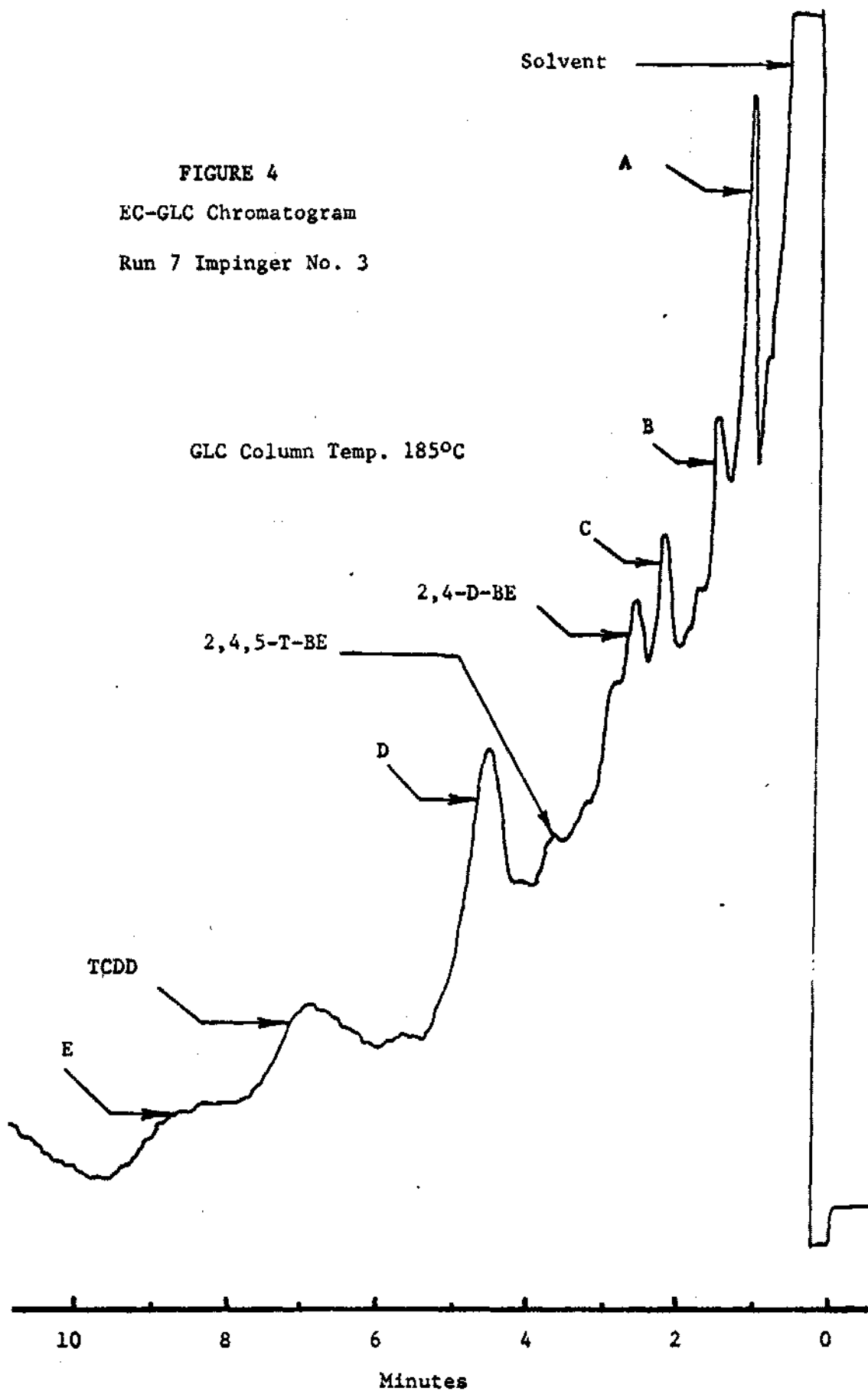


Table 1  
INCINERATION CONDITIONS DURING VARIOUS TEST RUNS

Run	Orange I Burnt		TCDD Content/ ppm	Liters/ min Air	Collection Time/ min	Furnace Temp °C	Exit Air Temp °C	Pressure (Cm Hg above Ambient)	Pump Settings			Calculated Dwell Times/Sec	
	ul/min	Total ml							Syringe Used cc	Syringe Setting cc	Volume Setting ml/min	X Temp 600°C	800°C
3	37.1	1.01	<0.1	0.66	27.2	1005	780	1.2	2.5	10	0.15	4.5	3.5
4	60.0	0.6	<0.1	0.65	10.0	1005	790	1.2	2.5	5	0.15	4.3	3.3
5 <sup>1</sup>	120.0	1.2	<0.1	0.65	10.0	1008	790	6.0	2.5	5	0.3	4.1	3.2
6	110.6	1.5	<0.1	0.65	11.8	1018	795	1.5	2.5	5.0	0.3	4.2	3.2
7	125.0	1.25	14.0	0.60	15.0	1010	795	1.4	2.5	5.0	0.3	4.5	3.5
8 <sup>2</sup>	42.5	0.51	<0.1	0.2	12.0	1125	740	1.8	2.5	30	0.4	8.1	5.8
9	59.6	0.72	<0.1	0.65	12.0	1100	910	2.2	2.5	5.0	0.15	4.3	3.3
10	59.3	0.71	<0.1	0.65	12.0	1120	915	2.3	2.5	5.0	0.15	4.2	3.3
11	58.0	0.87	<0.1	0.65	15.0	1110	920	1.7	2.5	5.0	0.15	4.3	3.3
12	58.3	0.70	<0.1	0.65	12.0	1119	925	1.8	2.5	5.0	0.15	4.2	3.3
13	no	fuel burned		0.65	31.00	1111	925	1.8					
15 <sup>3</sup>	58.3	1.45	<0.1	0.68	25.0	1120	920	1.8	2.5	5.0	0.15		
16	58.3	1.05	<0.1	0.65	18.0	1140	950	1.2	2.5	5.0	0.15	4.2	3.3
17 <sup>4</sup>	no	fuel burned		0.6	45	1100	910	1.5					
18	63.3	0.950	<0.1	0.65	15	1060	900	1.8	2.5	5.0	0.15	4.2	3.3
19	53.3	1.6	14	0.65	30	1110	945	1.8	2.5	5.0	0.15	4.3	3.3
14 <sup>5</sup>	no	fuel burned		65	30	1110	930	1.8					

NOTE:

- <sup>1</sup>Impinger #4 occluded
- <sup>2</sup>Reversed burner input at top
- <sup>3</sup>Spiked burner gas with 8.4 ug/TCDD
- <sup>4</sup>Retention and recovery of TCDD and Orange
- <sup>5</sup>Recovery of NB 2,4-D

Table 2  
 SCRUBBING TRAIN CONFIGURATIONS DURING VARIOUS RUNS

Run	Spiked		Impinger 1	Solvent	Impinger 2	Solvent	Impinger 3	Solvent	Impinger 4	Solvent
	ug	Location								
3	0	-	TI	Benzene	FI	Benzene				
4	0	0	TI	Benzene	FI	Benzene				
5	0	0	TI	Benzene	FI	Benzene	F.Coil.	-		
6	0	0	FI	Benzene	TI	Benzene	FC	-		
7	0	0	FI	Benzene	TI	Benzene	FC	-		
8	0	0	TI	H <sub>2</sub> O	TI	Benzene	FI	Benzene	FC	-
9	0	0	TI	H <sub>2</sub> O	TI	Benzene	FC	-	FI	Benzene
10	0.06 TCDD	Imp #1	TI	H <sub>2</sub> O	TI	Benzene	FC	-	FI	Benzene
11	0	0	TI	H <sub>2</sub> O	FC	-	FI	Benzene	TI	Benzene
12	0	0	TI	H <sub>2</sub> O	FC	-	FI	Benzene	TI	Benzene
13	1400 Orange 0.06 ug TCDD	Imp #1	TI	H <sub>2</sub> O	FC	-	FI	Benzene	TI	Benzene
14	2.4 NB 2,4-D 2.4 NB 2,4-D	Imp #1 Imp #2	TI	1:1 H <sub>2</sub> O: Benzene	FI	Benzene	FC	-	-	-
15	8.4 TCDD	Incinerator gas	TI	Benzene	FI	Benzene	FC	-	TI	H <sub>2</sub> O
16	0	0	TI	Benzene	FC	Benzene	FI	Benzene		
17	4200 Orange 20.4 TCDD		TI	Benzene	FI	Benzene	FC	-	TI	Benzene
18	0	0	TI	Benzene	FI	Benzene	FC	-	TI	Benzene
19	0	0	TI	Benzene	FI	Benzene	FC	-	TI	Benzene

TI = Tapered Impinger  
 FI = Fritted Impinger  
 FC = Freeze Condenser

Table 3  
RECOVERY OF THE NB ESTERS OF 2,4-D and 2,4,5-T and TCDD FROM SPIKED SAMPLES (micrograms)

Run	Note	Collector 1	%/ Collector	Collector 2	%/ Collector	Collector 3	%/ Collector	Collector 4	%/ Collector	Total ug Spiked	Total ug Recov.	% Spiked
10	Orange D <0.1 ppm	TI:H <sub>2</sub> O		TI		FC		FI				
		ND	0	0.2	52.6	0.18	47.3	lab accident		none	0.38	-
		ND	0	0.2	44.4	0.25	55.6			none	0.45	-
	TCDD, burned	0.063	100	ND	-	ND	-			0.06	0.063	105
13	spike, D no fuel,	TI:H <sub>2</sub> O		FC		FI		TI				
		147	91	8.6	5.3	5.0	3.1	0.6	0.3	720	161.2	22.0
		125	96.5	2.5	1.9	1.6	1.2	0.5	0.3	720	129.5	18.0
	TCDD, burned	0.06	100							0.05	0.06	120.0
14	no burn D Imp 1&2	TI 1:1		FI		FC		TI				
		H <sub>2</sub> O:Ø						Trace	<1.0	2.4 ug	4.3	90.0
	each spiked w/ 2.4 ug NB 2,4-D	1.8	41.8(75) <sup>1</sup>	Imp 2&3; 2.5 ug; 58(104)						in Imp 1&2		
15	Orange D <0.1 ppm	TI		FI		FC		TI				
		10	69.0	4.5	31.0	Trace	1.0	ND		none	14.5	-
		15.6	43.8	18.11	50.9	1.9	5.3	Trace	1.0	none	35.6	-
	TCDD, burned	1.1	56.4	0.67	34.3	0.18	9.2	ND	0.1	8.4	1.95	-
					[78]					into inci- nera- tor		
17	spike, D no fuel,	TI		FI		FC		TI				
		1560	96	65	4.0	ND	0	ND	0	1664	1625	97.6
		1200	99.9	0.95	1.0	ND	0	ND	0	1536		78.1
	TCDD, burned	17.8	100	ND		ND		ND		20.4	17.8	87.3

1. ( ) = % spiked into that impinger.

2. [ ] = % recovery of material passing through impinger.

Table 4  
ORANGE CONSTITUENTS RECOVERED FROM VARIOUS IMPINGERS SCRUBBING COMBUSTION GASES

Run	Note	Collector 1	%/Collector	Collector 2	%/Collector	Collector 3	%/Collector	Collector 4	%/Collector	Total ug Recovered
3	<0.1 ppm TCDD	TI		FI						
D		3.7	68.5	1.7	31.8					5.4
T		2.9	65.9	1.5	34.1					4.4
4	"	TI		FI						
D		0.7	58.3	0.5	41.6					1.2
T		0.7	46.6	0.8	53.3					1.5
5	"	TI		FI		FC				
D		0.8	66.7	0.4	33.3	ND				1.2
T		1.2	66.7	0.6	33.3	ND				1.8
6	"	FI		TI		FC				
D		Trace	<1.0	0.3	100	Trace				0.3
T		Trace	<1.0	0.3	100	Trace				0.3
7	14 ppm TCDD	FI		TI		FC				
D		Trace		Trace		Trace				Trace
T		Trace		Trace		Trace				Trace
	TCDD	0.48 ug	32.8	0.55 ug	37.6	0.43 ug	29.5			1.46
8	burner inverted <0.1 ppm TCDD	TI:H <sub>2</sub> O		TI		FI		FC		
D		5.7	11.2	1.2	2.3	43.0	84.9	0.7	1.4	50.6
T		5.1	9.0	1.0	1.7	50.2	89.0	0.1	0.1	56.4
9	<0.1 ppm TCDD	TI(H <sub>2</sub> O)								
D		Trace	<1.0	0.15	27.2	0.20	36.4	0.20	36.3	0.55
T		Trace	<1.0	0.47	37.3	0.39	30.9	0.40	31.7	1.26

Table 4 (contd)  
 ORANGE CONSTITUENTS RECOVERED FROM VARIOUS IMPINGERS SCRUBBING COMBUSTION GASES

Run	Note	Collector 1	%/ Collector	Collector 2	%/ Collector	Collector 3	%/ Collector	Collector 4	%/ Collector	Total ug Recovered
12		TI (H <sub>2</sub> O)		FC		FI		TI		
D		0.18	28	0.15	23	0.15	23	0.17	26	0.65
T		0.15	23	0.16	25	0.17	27	0.16	25	0.64
16		TI		FC		FI				
D		1.1	64.7	0.35	20.6	0.25	14.7			1.7
T		0.88	79.2	0.12	10.8	0.11	9.9			1.1
18		TI		FI		FC		TI		
D		2.1	56.9	1.59	43.1	Trace	<1.0	Trace	<1.0	3.69
T		1.57	55.3	1.27	44.7	Trace	<1.0	Trace	<1.0	2.84
19		TI		FI		FC		TI		
D		4.32	82.7	0.9	17.2	Trace	<1.0	ND	<1.0	5.22
T		0.76	81.7	0.17	18.2	ND	<1.0	ND	<1.0	0.93

TI = Tapered Impinger  
 FI = Fritted Impinger  
 FC = Freeze Condenser  
 ND = None Detected

Table 5  
HERBICIDE AND TCDD EMISSIONS FROM INCINERATION OF ORANGE HERBICIDE

Run	Feed Rate <sup>1</sup> ul/min	% Air <sup>2</sup> Stoi- chio- metric	Dwell Times Assum- ing 600°C/ 800°C	Eff Gas Temp °C	Total ug NB-D Disch	ppb NB-D Disch	Total ug NB-T, Disch	ppb NB-T Disch	Total ppb Herb. Disch	Total ug TCDD Input	Total ug TCDD Disch	ppb TCDD	Total Burn Time/ min	H <sub>2</sub> O <sup>4</sup> in First Impinger
3	37.1	248	4.5/3.5	780	5.4	28.3	4.4	20.5	48.8				27.2	
4	60.0	161.2	4.3/3.3	790	1.3	17.6	1.5	18.1	35.7				10.0	
9	59.6	162.3	4.3/3.3	910	0.55	6.2	1.26	12.69	8.35				12.0	*
10	59.3	163.9	4.2/3.3	915	0.38	4.3	0.47	4.73	9.0				12.0	*
11	58.0	166.8	4.3/3.3	920	0.5	<4.5	0.5	<4.0	<8.5				15.0	*
12	58.3	166.8	4.2/3.3	925	0.8	8.3	0.8	8.1	16.3				12.0	*
16	58.3	165.9	4.2/3.3	950	1.7	12.8	1.11	7.5	20.4				18.0	
18	63.3	152.8	4.2/3.3	900	3.7	33.4	2.84	22.9	56.3				15.0	
19	53.3	181.5	4.3/3.3	945	5.2	23.6	0.93	3.7	27.4	29.4	0.8	3.15	30.0	
5	120.0	80.6	4.1/3.2	790	1.2	16.3	1.8	21.8	39.0				10.0	
6	110.6	87.5	4.2/3.2	795	0.3	3.5	0.3	3.1	6.5				11.8	
7	125.0	71.4	4.5/3.5	795	<0.4	<5.9	<0.4	<5.2	11.1	21.0	1.46	18.7	10.0	

<sup>1</sup> 1.0 μl = 1x10<sup>-6</sup> liters (microliter)

<sup>2</sup> 100% air = air stoichiometrically required for complete combustion

<sup>3</sup> ug = 1x10<sup>-6</sup> grams = 1 microgram

<sup>4</sup> ppb =  $\frac{24,450 \times \text{ng/ml}}{\text{MW}}$



(2) Washing the benzene scrubbing solution with water and sodium bicarbonate is an adequate cleanup procedure.

4. Table 3 contains data concerning the recovery of the esters and TCDD when spiked into benzene (Run 17), water (10 & 13), and a benzene: water mixture (Run 14). In all of these runs one or more of the esters or TCDD were spiked into the first impinger (Run 14 was spiked with 2.4 ug NB-D in impingers 1 and 2) and the incinerator run without that constituent being burned (Table 2). These results indicate:

a. TCDD was recovered well from both water (112%) and benzene (87%). TCDD did not "bleed" down the impinger train; 100 percent of the amount recovered was always found in the impinger into which it had been introduced. This data indicates that once scrubbed from the gas phase TCDD does not re-enter it to any detectable degree.

5. The esters of 2,4-D and 2,4,5-T present a more complex situation. Recoveries of esters spiked into water as described above average about 20 percent (Run 13). However, collectors downstream from the water filled impinger yielded small and rapidly diminishing concentrations of herbicide esters (Tables 3 & 4); indicating the system was efficiently scrubbing the "bleeding" esters and that the amount bled was small (3-9 percent). This suggests that either the esters were not being efficiently extracted from the water or that they were being degraded in aqueous solution.

a. The partition coefficient of Orange esters between glass distilled water and benzene was determined using standard laboratory techniques and found to be over 99 percent in favor of the benzene; for both esters. The recovery of the NB esters of D and T from spiked distilled water samples, extracted as described above was 98-102 percent. Recoveries of D and T esters from spiked benzene charged impingers was 97 and 78% respectively (Run 17). This data indicates the low recovery of esters from Run 13-1 were due to breakdown of the NB esters in aqueous solution.

b. This hypothesis was further tested in Run 14. Impinger 1 was charged with a 1:1 mixture of benzene and distilled water; impinger 2 with benzene only. Both impingers were spiked with 2.4 micrograms of NB-2,4-D and air passing out of the incinerator was passed through the impinger train. Recovery of NB-2,4-D in the first and third impingers combined 105%; only a trace of ester was detected in impinger four. This data also indicates NB ester degradation is occurring in the first impinger but at a lower rate than in Run 13. The 1:1 benzene:H<sub>2</sub>O mixture apparently protected the spiked esters to some extent from degradation. This data therefore confirms the observations of Run 13 and also strongly suggests a hydrolysis degradation reaction. This mechanism has appeal because hydrolysis of organic esters in aqueous systems

is known to occur, and to proceed rapidly when catalyzed by either acids or bases. Further investigations into the mechanism of this phenomenon was considered beyond the scope of this study.

c. The above data has the following significance:

(1) The NB esters of 2,4-D and 2,4,5-T were recovered satisfactorily from benzene solutions; although a small amount of the esters did "bleed down" the impinger train; very little, if any, "bled" out of the train in a thirty minute period.

(2) Degradation of the esters evidently proceeded rapidly in aqueous solutions scrubbing incinerator effluent air. Benzene protects the esters. The comparatively small volume of water:benzene (about 1:10) that will be present in the stack samples will adequately protect the esters from significant degradation after extraction from the air stream.

(3) Undegraded herbicide esters are quantitatively recoverable from neutral water samples.

(4) This data indicates that herbicide esters may be significantly hydrolysed within the incinerator scrubber prior to contact with the benzene. Such a hydrolysis mechanism if present would result in the release of 2,4-D and 2,4,5-T acids. These materials are potent herbicides and if formed would not be monitored in this system. Determining the presence or absence of such intermediate degradation products is being accomplished by contract and is beyond the scope of this project.

6. The data above indicates that TCDD does not "bleed" from impinger to impinger and is easily and accurately chromatogrammed in benzene solution. The following experiment demonstrated that TCDD is also efficiently collected from the gaseous phase into benzene solutions. Eight and four tenths micrograms of TCDD was injected through a septum directly into the incinerator tube, four centimeters below the exit port (Run 15). The benzene solution of TCDD immediately evaporated and combusted prior to leaving the burner. Undegraded TCDD was then swept out of the incinerator into the impinger in a gaseous state.

a. Approximately twenty-five percent of the dioxin injected into the upper incinerator was recovered in the impingers. Recoveries rapidly diminished up the impinger train indicating efficient scrubbing. The fritted glass impinger was the most efficient scrubber removing about 78.0 percent of the TCDD coming to it.

7. Data indicating that the NB esters of 2,4-D and 2,4,5-T are efficiently scrubbed from gases are presented in Table 3 and 4. Runs 5,

15, 16, 18 and 19 indicate efficient scrubbing across the benzene charged impingers, especially the fritted impinger. Runs 9, 10, and 12 indicate that scrubbing efficiency was lowered by first saturating the air stream with water. However, Run 8 indicates a series of fritted impingers may overcome this problem. This potential problem of water induced scrubber inefficiency should not particularly prove serious because inefficient scrubbing is readily apparent, and can be eliminated by taking the gas samples just prior to the incinerator scrubber, or by using a desiccant scrubbing medium in the first impinger, such as ethylene glycol, and extracting the herbicide esters from it. Drying the stack gases with cartridge type filters charged with sodium sulfate is not feasible since pesticides are known to bind to this compound.

8. Table 5 contains data concerning the emissions of herbicide esters and TCDD under various temperatures and air to fuel ratios at dwell times of 3 - 4.5 seconds.

a. The time deadline imposed by this project prevented procurement of a thermocouple probe of sufficient length to empirically measure the temperature profile of the combusting gases along the length of the incinerator. The average temperature of molecules passing through the furnace (time basis) was therefore estimated to be between 600-800°C.

b. The mean dwell time (the mean time a molecule spent in the incinerator tube) was then estimated at these temperatures by dividing the effective volumes of the incinerator at 600 and 800°C by the calculated volume of gas generated by the total combustion (CO<sub>2</sub>, H<sub>2</sub>O and HCl) of the metered quantities of Orange herbicide and air combusted per second.

c. Incineration of Orange at 165% air, 950°C, and dwell times of 3-4 seconds resulted in efficient pyrolysis of the NB esters of 2,4-D and 2,4,5-T. Less than 60.0 parts per billion of combined esters were emitted from the burner at any time. Greater than 99.999 percent of the herbicide esters were destroyed under all experimental conditions of pyrolysis.

(1) The emissions of NB herbicide esters was not significantly increased when the temperature was reduced to 800°C and/or the air:fuel ratios were reduced to approximately 70 percent of the stoichiometrically required quantity. This data indicates that operational variations above and below the temperature and air:fuel ratios found to be optimal in the successful commercial facility will not result in fluctuating emissions of NB herbicide esters.

(a) This data does not indicate that pyrolysis of the NB esters is as complete at lower temperatures and air:fuel ratios. In

fact visual analysis of the chromatograms indicates that the emission of chlorinated pyrolyzates increases under these conditions (Fig 4). The emissions of nonhalogenated pyrolyzates, though not measured, undoubtedly increase to a much greater extent, especially under conditions of less than the stoichiometrically required air.

d. The very limited data concerning the pyrolysis of TCDD indicated less efficient percent destruction (97% at 945°C) but a lower gas effluent concentration (3.2 ppb) because of its much lower initial concentration in the herbicide.

(1) Lowering the effluent gas temperature 150°C apparently increased the effluent emissions of TCDD sixfold (Table 5, 19 & 7). Increasing the temperature of the burner to 1000°C (instead of 950°C, Run 19) is expected to substantially increase the efficiency of TCDD pyrolysis.

#### IV. CONCLUSIONS

1. Monitoring the NB esters of 2,4-D and 2,3,5-T and TCDD in water and gas effluents resulting from commercial incineration appear feasible.

2. Limits of detection for each of the NB esters in effluent gas and water are 2.0 and 1.0 ppb respectively. The corresponding limits for TCDD are 3.0 and 1.0 ppb respectively.

3. Interference from other pyrolyzates will be negligible at temperatures of 1000°C, dwell times of 3 seconds and stoichiometric air/fuel ratios of 150%.

4. The very high water content of the gas samples taken from the incinerator stacks may interfere with the benzene charged fritted impinger extraction system. This condition is readily detectable. Substituting ethylene glycol for benzene in the first impinger should overcome this potential problem. Other alternatives are available.

5. Emissions of the NB esters of 2,4-D and 2,4,5-T and TCDD when burned at 1000°C with 150% air and a dwell time averaging 3 seconds will be very low and safe to all forms of life. Incineration in tandem with the monitoring program developed above and outlined in detail in Appendix 6 of this report will offer negligible risks to the environment or human health from emissions of NB herbicide esters or from TCDD.

#### V. RECOMMENDATIONS

1. Identification of other pyrolyzates formed during the incineration of Orange herbicide should be accomplished as soon as possible. Pyrolysis of herbicide in such experiments should be accomplished in a continuously burning liquid injection incinerator as described herein to provide valid results.

2. The impact statement be modified as previously recommended by this Laboratory to include the monitoring program outlined in Appendix B.

**Appendix B**

**STATEMENT OF WORK INCLUDING MONITORING  
PROGRAM FOR ORANGE HERBICIDE**

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



REPLY TO  
ATTN OF: CC

18 May 1972\*

SUBJECT: Protocol for Monitoring Incinerators Burning Orange Herbicide

TO: SF

1. Attached is the protocol originally submitted to you on 2 Feb 1972.
2. The document has been declassified as requested in your letter of 5 April 1972.
3. This letter also requests that changes be made as necessary to reflect information gained by the research at Mississippi State University (MSU). The first three MSU reports contained no information bearing on this monitoring program. The latter report was received in this Laboratory 15 May 1972. Additional delay in submitting this protocol appears unwarranted.
4. Some changes have been made in the protocol refining stack sampling techniques and increasing the maximum allowable recommended limits of herbicide and TCDD discharges.

FOR THE COMMANDER

A handwritten signature in cursive script, reading "Richard A. Callahan".

RICHARD A. CALLAHAN, Ph.D.  
Capt, USAF, BSC  
Chief, Biological Environmental Studies Branch

1 Atch  
Statement

\*Original submitted February 1971.

## Statement of Work-Monitoring Incineration <sup>1</sup> of Orange Herbicide

### 3. Guidelines for Incineration

a. Disposal of Orange and Orange II herbicides will be by incineration. The incinerator must be operated so that the temperature of the interior gases reach or exceed 1000°C with a mean dwell time of three seconds excluding the scrubbing processes.

b. The incinerator will be provided with safety features which automatically stop the introduction of herbicide into the incinerator if one or more of the following circumstances occur:

- (1) The operating temperature drops below 1000°C.
- (2) The air to fuel ratio drops below the value preset by the contractor.
- (3) The temperature of the emitted gases exceeds 200°F.
- (4) The emission scrubbing system fails.
- (5) The fuel feed rate exceeds the value preset by the contractor.

c. The rate of herbicide incineration will immediately be altered as directed in Paragraph 6 if the discharge of n-butyl 2,4,5-T, n-butyl 2,4-D, isooctyl 2,4,5-T or 2,3,7,8-tetrachlorodibenzoparadioxin (TCDD) exceed those limits described below for either air or water discharges. The sampling frequencies and volumes of air and water samples will be those described below. Alternate methods of sampling stack emissions and water discharges as well as alternate analytical techniques may be used provided they meet the standards of accuracy and precision outlined below and are approved by the USAF and all other municipal, state and federal regulatory authorities. Empirical proof of such alternate techniques will be required prior to the granting of Air Force approval.

### 4. Permissible Discharges of Incineration Products

a. At no time shall the concentration of any one of the normal butyl esters of 2,4-D or 2,4,5-T, or the isooctyl ester of 2,4,5-T exceed 0.1 ppm in the effluent gases, when calculated on a volume basis and corrected to 10% O<sub>2</sub>, 70°F and 760 mm Hg (STP). The combined herbicide emissions shall never exceed 0.3 ppm when calculated on a volume basis and corrected to 10% O<sub>2</sub>, STP.

<sup>1</sup> Paragraphing numbered for insertion into a draft SOW prepared by SAAMA/SF.



b. The concentration of TCDD shall not exceed 0.01 ppm in the effluent gases when calculated on a volume basis and corrected to 10% O<sub>2</sub>, STP.

c. The combined (sum) concentration of the N.B. 2,4-D, 2,4,5-T and the isooctyl ester of 2,4,5-T shall not exceed 0.1 mg/l in the scrubber blow down water.

d. The concentration of TCDD in the blow down H<sub>2</sub>O shall not exceed 50.0 ppb (50 ug/l).

e. Dilution of water discharges to meet these criteria is unacceptable.

f. The discharge of all other combustion products, including the oxides of carbon and nitrogen and hydrogen chloride shall meet with all applicable federal, state and municipal requirements.

g. The overall discharge of herbicide to the environment shall never exceed one hundredth of one percent of the input to the incinerator during any monitoring period.

5. Calibration and Testing of Monitoring Program Prior to Incineration.  
Prior to Incineration:

a. Prepare analytical standards of the Normal Butyl esters of 2,4,5-T and 2,4-D and the isooctyl ester of 2,4,5-T in nanograde benzene. Convenient concentrations should fall between 10 and 20 picograms ( $1-2 \times 10^{-11}$  grams) per  $\mu$ l benzene. Prepare similar analytical standards of 2,3,7,8-tetrachlorodibenzoparadioxin (TCDD). Caution - TCDD is EXTREMELY TOXIC - Special handling procedures must be followed for its safe use.

b. Pack a glass gas liquid chromatography (GLC) column with 1.5% OV210, 1.5% QF-1 on Anakrom Q 80/90 Mesh. This column may be prepared using standard techniques or obtained from commercial sources. Condition the column for use at 195°C.

c. The following GLC parameters have been found suitable for the analyses of Orange herbicide constituents. Temperature parameters: Injector-225°C, Column 195°C, Ni 63 Electron Capture Detector-345°C. Carrier gas: Nitrogen at 5.0 ml/min. flow. Under these conditions good separation of all herbicide esters and TCDD is obtained using a 6 ft. glass column.

d. Establish the response characteristics of the instrument to all three herbicide esters and TCDD. Quantitation of all residues must be made within the linear limits of detector performance.

e. The lower limits of sensitivity shall be 5.0 picograms ( $5 \times 10^{-12}$  grams) or lower for all herbicide esters and 10 picograms for TCDD.

f. Precision shall be no less than  $\pm 5\%$ .

6. The following gas scrubber systems have been tested using a laboratory scale incinerator and found to recover 90% NB 2,4-D; 80% 2,4,5-T and 95% TCDD present in effluent gases of a laboratory scale incinerator.

a. Probe: Use a Pyrex or equivalent glass probe constructed in such a way that the gas temperature is maintained at 150-200°C.

b. Train Assembly: Attach the probe to the impinger train assembly via a glass ball joint. The impinger train consists of four all glass midget impingers with fritted glass diffusers connected in series by glass ball joints. The impingers are filled with 30 ml of nanograde benzene.

c. Pump - Meter Assembly: A vacuum pumping system capable of pumping known quantities of air at known temperatures and pressure is used to draw gas samples through the impinger train. Suitable equipment includes rotary vane-type pumps, dry gas meters, orifice meter, and a rate meter. The temperature and pressure of the gas at the meter must be accurately recorded. Benzene and water are removed via a gas drier holding 50 grams of activated carbon and 10 grams of Silica Gel located just downstream of the impingers. Alternately 125g of Silica Gel and activated carbon may be placed in two separate impingers and placed downstream of the scrubber impingers.

## 7. Sampling Procedure

a. Determine the stack gas velocity and flow rate using methods I and II of "Environmental Protection Agency Regulations on Standards of Performance for New Stationary Sources (40 CFR 60, Appendix, Test Methods I and II)." Redetermine these parameters whenever the fuel or air feed rates are varied or each seven days.

b. Determine particulate sizes by the following modification of 40 CFR; 36 Part 60, "Standards of Performance for New Stationary Sources; Appendix Test Method 5." Place two, heat resistant filters in series. The first capturing particulates 0.5  $\mu\text{m}$ . Determine weights as described in the method. It is in the contractors advantage to capture as small particles as possible in the second filter. Sample for at least 10 minutes.

c. Insert probe and thermometer into the center of the stack at least two diameters from its top or some other convenient location suitable for stack sampling.

d. Sample at 0.6-0.8 l/min STP as dry benzene free gas.

e. Accurately record:

(1) Rate meter reading (read at 5 minute intervals).

(2) Gas temperature and pressure at dry gas meter.

(3) Sample collection period.

(4) Oxygen concentration in stack gas using Orsat or equivalent meter.

f. Sample collection period shall be thirty minutes or longer of continuous incinerator operation.

g. During the sampling period composite at least 3, 1 liter water samples of scrubber effluent at equal time intervals.

h. Quantitatively transfer benzene from impingers 1-3 into a 500 ml separatory funnel, wash twice with 100 ml H<sub>2</sub>O, adding several mg of sodium bicarbonate to the final wash. Discard H<sub>2</sub>O, dry with sodium sulfate.

i. Quantitatively transfer benzene from impinger 4 to 150 ml volumetric. Wash similarly as above using 50 ml H<sub>2</sub>O and 1 mg bicarbonate.

j. Quantitate the NB 2,4-D, NB 2,4,5-T, isooctyl 2,4,5-T and TCDD contained in the two samples using standard Electron Capture GLC techniques. Concentrate or dilute the volumes of benzene as necessary to obtain GLC responses in the linear range previously determined.

k. Resample if greater than 5% of any one of these four herbicide constituents is present in the final impinger.

(1) If greater than 5% continues to be found in the final impinger, proceed with the following technique. Charge the first impinger with 30 ml of analytical grade ethylene glycol. Sample using the four impinger train as above. Quantitatively transfer the glycol to a 500 ml separatory funnel containing 300 ml of water and extract three times with 25 ml of benzene. Composite the benzene extracts with impinger 2 and 3, dry and analyze as above.

(2) Document the recoveries of the NB and IO esters and TCDD by spiking 30 ml of ethylene glycol with 5-10 µg of ester and TCDD standards.

(a) If recoveries are less than 90% contact Air Force sources for aid.

l. Calculate the dry, benzene free volume of gas passed through the impingers at STP.

$$\text{Vol at STP} = \text{Vol metered} \times \frac{\text{meter absolute pressure (mm Hg)}}{760} \times \frac{273.16}{\text{Temp of metered gas } K^{\circ}}$$

m. Calculate the ppm by volume of each of the herbicide constituents corrected to 10% O<sub>2</sub>.

$$\text{ppm} = \frac{(24450) (\text{mg/l}) \times \% \text{O}_2 \text{ in stack gas}}{\text{M.W.} \quad 10}$$

8. If any constituent violates the emission limits - Cut feed rate of herbicide by 50% and resample immediately or find cause of violation. When probable cause found and corrected resume feed rate and resample immediately to verify correction. Each feed rate must be known to meet emission and effluent specifications within 1 1/2 hours after any rate change. Backup GLC equipment will be necessary to avoid incinerator shutdown due to analytical down time.

9. Calculate total herbicide emitted from stack during entire sampling period using standard stack sampling procedures.

10. Adjust the pH of the blow down effluent sample to 7.0 using HCl or NaOH and extract a one liter aliquot three times with 50 ml of benzene; compositing benzene extracts. Bring these extracts to a convenient volume, dry with a small amount of sodium sulfate and chromatograph.

11. Calculate the concentration of herbicide constituents in the blow down water. Follow the procedure listed in Paragraph 8, above if water discharge exceeds limit.

12. Calculate total amount of each constituent discharged in water during stack emission monitoring period. (Volume of blow down discharged during sample period x concentration of constituent/unit volume.)

13. Calculate percent herbicide discharged to the environment.

$$\frac{\text{oz of constituent discharged to air and H}_2\text{O} \times 100}{\text{oz of constituent fed to burner}}$$

14. If percent degradation is less than required follow procedure outlined in Paragraph 8 above.

15. Procedures to be followed for starting herbicide burn.

a. Bring incinerator to operating conditions as outlined above using natural gas as fuel.

b. Introduce herbicide at one half the rated capacity of the incinerator.

c. Ten minutes into burn take gas and water samples and analyze as prescribed above.

d. Two hours later take second set of samples and similarly analyze.

e. Increase herbicide input to rated capacity of incinerator only if both samples meet or exceed all removal specifications.

f. Take samples 10 minutes and two hours after increasing feed rate.

g. Determine stack gas velocity and flow rate.

h. Sample every 8-hours for the first 48-hours of incineration at fully rated capacity.

i. Sample every 12-hours from 48-120 hours after initiation of capacity burn.

j. Sample every 24-hours thereafter.

k. If other materials are incinerated with Orange herbicides the following parameters must be met:

1. The term full rated capacity refers to the total input into the incinerator regardless of the percent herbicide in the mixture.

2. The effluent gases and blow down water will be sampled ten minutes after any change in the chemical nature of the feeding solution is made. This sample will be in addition to the samples mentioned above.

3. GLC responses giving peaks  $\pm 1\%$  of the retention time of herbicide constituents will be considered as being such constituents until they are demonstrated not to be.

4. Particulate emissions must average (by weight) less than 0.5 microns in diameter.

1. All GLC results must be reviewed and validated by a graduate pesticide chemist, with at least two years experience with pesticide analyses, prior to the use of this data to continue the burning of herbicides.

APPENDIX C

WRITTEN COMMENTS CONCERNING THE ENVIRONMENTAL  
ASSESSMENT STATEMENT

	<u>Page</u>
C-1 Review of Methods to Destroy Tactical Herbicides - June 1970-----	38
C-2 Comments Concerning Draft Environmental Impact Statement - 13 Sep 71-----	44
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HKG

22 June 1970

Disposal and Handling of Herbicides (Yr Ltr, 18 May 70)

ASD (ASJT-1)

Wright-Patterson AFB OH 45433

1. Several typographical errors occurred in your letter of 8 May 1970. Orange consists of 50% n-Butyl 2,4,5-trichlorophenoxyacetate not 3,4,5-trichlorophenoxyacetate. Orange not Blue contains approximately 30 ppm of a dioxin compound (not dioxane). White or Tordon 101<sup>TM</sup> is 10.2% 4-amino-3,5,6-trichloropicolinic acid as the trisopropanolamine salt, not 3,5,6-trichlorophenoxyacetate, and 39.6% 2,4-dichlorophenoxyacetic acid as the trisopropanolamine salt.

2. Collection and disposal systems would necessarily need to be designed by engineers on site or with detailed information concerning the operations. Obviously, a method of disposal should be a prior consideration before collection systems are designed. General information concerning this subject is discussed below in paragraph 3.c.

3. Disposal techniques for concentrated pesticides are limited to biodegradation, chemical and thermal destruction.

a. Biodegradation:

(1) Biodegradation of large quantities of Orange is theoretically possible and is known to occur in soil after high rates of application. However, the following problems make this alternative impracticable as a disposal technique of excess or contaminated herbicides.

(a) Presently, the causative agent or agents of the widely reported teratogenic effects of 2,4,5-T<sup>(1)</sup> are unidentified. Therefore, biological systems that discharge effluents to waterways; i.e., lagoons, activated sludge units, etc., could not be considered safe for release to civilian potable water supplies. Similarly, surface run-off from soil containing 2,4,5-T would also be unsafe for consumption by women of child-bearing age.

(b) The toxicity of these materials to biological systems is still incompletely understood. For example, 2,4-D has been reported to have a delayed lethal effect under some conditions to at least several diverse species of bacteria<sup>(2)</sup>. 2,4-D is the most rapidly degraded herbicide and yet its toxicity to soil organisms is scantily understood.

(2) Agent White (Tordon 101<sup>®</sup>) contains Tordon<sup>®</sup> or picloram (10.2%) and 2,4-D (39.6%). Tordon is relatively stable in soils and microbial decomposition is slow. Bovey et al<sup>(3)</sup> has shown that picloram applied at 3 lbs/acre persists for at least 18 months. Clay soil exhibited phytotoxicity 18 months after application. Data is presented for 5 different soils, at 5 different depths under varying climatic conditions. All conditions<sup>(3)</sup> affect biodegradation. This paper illustrates the persistence of Tordon<sup>®</sup> and the variability of biodegradation under various conditions.

(3) I know of no studies specifically on the biodegradation of agent Blue (Phytar 560<sup>®</sup>). Bioassay studies indicate degradation is rapid (1-2 months).<sup>(4)</sup> Agent Blue is comprised of 27.2% sodium cacodylate (sodium salt of dimethylarsinic acid). Complete degradation of this material yields 15.3% elemental arsenic. This quantity of arsenic is incompatible with large scale disposal of this agent via biodegradation in soil or aqueous systems because the residual arsenic would be liberated into the environment - stay in the soil or be leached into ground water and streams.

b. Due to the covalent bond structure of all of these agents chemical techniques of destruction are theoretically possible. However, construction and operation costs of such a facility would be large, and a disposal problem of the resulting chemical by-products could be as big a problem as the disposal of the untreated agents. Operation of such a facility would require highly trained chemists and a basic chemistry laboratory. Such a solution is not practical.

#### c. Thermal Destruction:

(1) Thermal destruction of herbicide concentrates is the most feasible, safe and cheapest method of disposing of pesticide concentrates. Such an incinerator facility would have to be constructed so that emissions of phytotoxic residues would be impossible. Research progressing at the University of Mississippi<sup>(5)</sup> indicate incineration is technologically feasible for chlorinated hydrocarbon pesticides at temperatures of 1000°C. Complete pyrolysis of 2,4,5-T occurs at 500°C leaving no residue. At the former temperature picloram left a residue of 0.8% and disodium metharsonate left a residue of 18.8%. This latter residue would consist almost exclusively of arsenic oxides. Similar pyrolysis products would be left by agent Blue.

(2) A small incinerator equipped with a continuous feed pump would dispose of relatively large amounts of herbicide if operated 24 hrs/day - 5 gal/hr = 600 gal/5 day week.

(3) The combustion residue of agent Blue would consist largely of arsenic (as arsenic trioxide) which could be disposed of through an R&M facility or converted to an insoluble ammonium salt and placed in a suitable landfill. This latter alternative may not prove practical for moderate to large quantities of arsenic. An arsenic residue is going to be present no



matter what disposal technique is used for Blue. Incineration will yield a relatively pure, easily handled, perhaps marketable residue.

(4) A properly designed incinerator facility could be used for the destruction of other organic chemical wastes generated in SEA including DDT and Dieldrin. After use in SEA the incinerator could be shipped to the CONUS where the need for several such facilities is rapidly becoming critical. Therefore, the investment in such a combustion facility would provide a convenient safe method of destroying not only excess and contaminated herbicides, but, if properly designed, other persistent and toxic chemicals which presently constitute a major disposal problem. The design and construction of several such facilities is strongly recommended.

4. Accidental spills can best be coped with on surfaces that are impervious to the agent. For this reason, areas in which herbicides are stored, or routinely handled, should consist of a lipped concrete apron draining to a central sump. Large spills can then be recovered by pumping and rebarreling. Residual material can be soaked onto an absorbent material such as sawdust. Small spills or residues from large spills can be decontaminated by wetting the surface with a suitable solvent - diesel oil, kerosene, or isopropyl alcohol for Orange, H<sub>2</sub>O for White and Blue - and the solvent in turn picked up by the absorbent. The contaminated absorbent could then be mixed in a slurry and pumped into the incinerator described above.

#### 5. Herbicide Decay Curves in Soils:

a. Definitive decay curves of organic materials vary in a given soil because of fluctuations in temperature, moisture, and light and in different soils because of many variables, including the above, plus pH, organic content, endogenous flora and fauna, particle size, mineral content, etc. Recognizing these limitations most authors do not attempt to define generalized decay curves. The application of such data from one situation to another, without much more knowledge than is currently available, is fallacious.

#### b. Translocation of Herbicides:

(1) When herbicides are applied to soils they degrade with time. Prior to degradation they may be translocated from their point of application. Barnett et al<sup>(6)</sup> found that 27% of 2,4-D ester was washed from a sandy loam test plot by an artificial rain. Less than 3% of the water soluble amine was so lost. The authors explain this phenomena occurs because the water insoluble esters remain near the surface whereas the water soluble amine washes into the ground. Orange consists of the water insoluble esters of 2,4,5-T and 2,4-D.

(2) Trichell et al<sup>(7)</sup> found substantial herbicide removal occurred - the runoff containing 1.6-2.2 ppm herbicide - when dicamba,

2,4,5-T and picloram (Tordon<sup>®</sup> a constituent with 2,4-D of Tordon 101<sup>®</sup>, agent White) were applied at 2 lbs/acre. These experiments were performed on Irving Clay and loam soils. Surprisingly, the presence of a sod cover greatly increased the rate of runoff. Four months after application detectable residues were still washing from the plots.

(3) These studies indicate substantial quantities, especially of water insoluble Orange, can be expected to run off of areas to which it is applied. The previously cited teratogenic effects of 2,4,5-T (which comprises 50% of agent Orange) should therefore preclude the disposal of Orange by soil degradation.

#### c. Biodegradation:

(1) Orange, White and Blue are biologically degradable. Norris<sup>(8)</sup> found that the constituents of Orange degraded at different rates. Eighty-five percent of the 2,4-D degraded in 300 hours compared to less than 25% of the 2,4,5-T so applied.

#### (2) Oregon Studies:

(a) Currently, the Environmental Health Sciences Center of Oregon State University is conducting a Waste Pesticide Management Program under the direction of Dr. R. L. Goulding.<sup>(9)</sup> (Status reports of this study are published monthly and can be obtained from this Laboratory or from Dr. Goulding in Corvallis 97331.) A summary of these reports follows:

(b) A massive application of 2,4-D waste liquor (27.6% 2,4-D, 72.6% chlorophenols) equal to 300 lbs 2,4-D/acre, followed by a heroic water application (9" of H<sub>2</sub>O over a 10-day period) resulted in the bulk of the pesticide being located from 1 1/2 - 12" below the soil surface. Small quantities of 2,4-D could be found 4 ft below the surface. A 90-fold decrease in 2,4-D occurred in 155 days. It should be kept in mind that: The chlorophenol content undoubtedly modified the percolation properties (most likely held the 2,4-D near the surface); the 2,4-D present is in the acid form not the butyl ester (as seen above, esters of 2,4-D are insoluble in water, the acid is water soluble); previous papers have shown 2,4-D to be much more readily degradable than 2,4,5-T.

(c) Studies are also being conducted by the Corvallis group concerning the effects of covering similarly treated plots with black polyethylene; thereby, elevating the moisture concentration and temperature. Twenty-six days after treatment with the manufacturing waste, mentioned above (Ca 3:7 2,4-D:chlorophenols), concentrations of both chemicals were substantially higher under the polyethylene covered treatment. Two factors which undoubtedly affected these results were the inability of the chlorophenols to evaporate from under the tarp and the probability of a low concentration of oxygen in the covered soil. Therefore, no conclusions

concerning the effect of water content of soil on the biodegradation rate of 2,4-D can be made at this time from these studies.

#### 6. Cleaning Herbicides Storage Tanks:

a. The risks involved in cleaning storage tanks formally containing herbicides are primarily those encountered in cleaning any such tanks, anoxia and heat prostration; secondarily, toxicity of the herbicides or cleaning solvents could be a hazard.

b. Safety procedures for cleaning storage tanks should include the basic safety procedures utilized in cleaning fuel storage tanks (T.O. 00-25-235).

(1) Use the buddy system with one member outside the tank at all times.

(2) Personnel within the tank should be attached to his "buddy" by rope.

(3) Supply air to member inside tank through a face mask equipped with eye protection; such as Scott No. 21325 respirator, combination airline/self contained (pressure demand) with communication system. An additional air supply and mask should be available for emergency use.

(4) Wear rubber gloves, rubber boots, and coveralls.

(5) Change clothing and shower immediately after work.

(6) Beware of heat prostration, anoxia and if agent Blue has been stored in tanks monitor urine for lead.

c. Water is the preferred solvent for cleaning tanks formally holding Blue and White. Tanks contaminated with Orange can be cleaned with diesel oil, kerosene or isopropyl alcohol.

#### SIGNED

WALTER W. MELVIN, JR., Colonel, USAF, MC  
Commander

1 Atch  
Literature Cited

Cys to:  
AFLC (MCDPE/Maj Higgins)  
USAF Weapons Lab (Maj Eggert)

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



REPLY TO  
ATTN OF: CC

13 September 1971

SUBJECT: Comments on Environmental Impact Statement on the Disposition of  
Orange Herbicide by Incineration

TO: Commander, USAF Env Health Laboratory, Kelly

1. The possibility 2,4,5-T, Dioxin and to a lesser extent 2,4-D will be emitted to the environment via stack emissions and/or in scrub water discharges certainly constitute a situation " . . . highly controversial with regard to environmental impact" as stated in DoD Directive 6050.1. These possible emissions are not mentioned in the impact statement.

2. This Laboratory has routinely recommended discharge specifications to meet state and federal water quality standards for the past four years. Acceptable concentrations of dusts, solvents, vapors and pesticides in the home, on the job, and in the natural environment are also routinely recommended. Recent communications with Hq USAF indicate this role will shortly be expanded.

3. Recommend this policy be continued regarding this impact statement by:

a. providing information regarding the probable emissions of 2,4,5-T, 2,4-D and dioxin in air and water resulting from this incineration.

b. recommend permissible stack and effluent water discharge limits.

c. require that the contractor provide data that demonstrate these limits are met in his facility or -

d. receive written permission from all the state and local authorities concerned to dispose of the herbicide by incineration without such data.

**SIGNED**

RICHARD A. CALLAHAN, PhD, Capt, USAF, BSC  
Chief, Biological Environmental Studies Branch

1 Atch  
Recommendations

Insert pg 10, Line 3

The combustion temperature is maintained at approximately 2500°F. Combustion at 900°F has been shown to degrade these herbicides to CO<sub>2</sub>, H<sub>2</sub>O and (after scrubbing) HCl. Dioxin (2,3,6,7 tetrachloro-dibenzo-p-dioxin) is thought to degrade similarly. Combustion at 2500°F with sufficient air and incinerator retention times should discharge herbicide in the part per billion (ppb) concentration range, and dioxin at concentrations less than 1X10<sup>-12</sup>. Such minute discharge will have no effect on man, plants or animals.

Insert pg 12 after Line 4

The discharge of herbicide or Dioxin if existent at all will be so minute as not to have any measurable environmental effect. However due to the widely publicized alleged teratogenic effects of these products public concern relative to destruction of this material may possibly occur. This concern can be best circumvented by the adoption of discharge specifications for 2,4-D, 2,4,5-T and the Dioxin. Final specifications should be the results of the efforts of the concerned Federal, State and local agencies. Limits such as 0.1 ppm herbicide in stack emissions and 2 parts per billion (ppb) in water discharges are recommended. All Dioxin discharges should be undetectable in air and water.

Insert pg 15, Line 10 under D

Alternates for disposing of drums by landfill are:

1. Sell as reconditioned containers.
2. Sell as scrap metal for smelting.

The sale and reuse of these containers is possible. Standard reconditioning techniques will assure reconditioned drums are completely safe. Used drums often sell for about \$2.00 a piece yielding a possible savings of \$30,578, plus the avoidance of landfill disposal costs.

The sale of crushed decontaminated drums for scrap metal will yield less monetary return than reconditioning of the drums but avoids landfill costs.

Both of these alternatives are safe provided decontamination is thorough.

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



REPLY TO  
ATTN OF: CC

23 November 1971

SUBJECT: Review of Environmental Impact Statement on the Disposition of  
Herbicide "Orange"

TO: Director of Aerospace Fuels/Colonel A. D. Hagen  
Hq SAAMA  
Kelly AFB TX 78241

Attached for your review and any action which you may deem appropriate  
are comments from personnel of this Laboratory on the subject indicated  
above.

*Walter W. Melvin, Jr.*  
WALTER W. MELVIN, JR., Colonel, USAF, MC  
Commander

1 Atch  
Comments

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



REPLY TO  
ATTN OF CC

23 November 1971

SUBJECT: Review of Environmental Statement on the Disposition of Orange Herbicide

TO Commander, USAF Env Health Lab/CC  
Kelly AFB TX 78241

1. The draft of this environmental impact statement (Atch 1) has been reviewed.
2. Detailed changes in the prescribed document format have been prepared (Atch 2).
3. Additions, corrections or deletions fall into one or more of the following categories. Changes are numbered to facilitate coordination.

<u>Reason for Change</u>	<u>Change No. (Atch 2)</u>
a. Statement treats the Sauget, Illinois site as an alternate facility. Facilities should be treated equally in this statement.	1,7,8,9,15,21,22
b. Information presented is incomplete.	2,3,6,12,13,14,18
c. Wording awkward or word misspelled.	4,25,26,27,8,18
d. Organization awkward.	5,10,13,28
e. Information deleted is superfluous and/or inaccurately preferential.	9,16,21
f. Decontaminated drums should be disposed of by smelting not burying in a landfill. This method is cheaper and less controversial than drum burial. This recommendation has been made on three prior occasions. Burial in a landfill should be presented as an alternate.	11,15,17,19,20,21,22, 24
g. The public and scientific concern over this action necessitates the continuous documentation of its safety throughout the burning operation. Therefore, a monitoring program of all emissions and discharges for active herbicides and tetrachlorodioxins should be a requirement not an option as stated in this report.	14,18,23

*Richard A. Callahan*

RICHARD A. CALLAHAN, PhD  
Captain, USAF, BSC  
Chief, Biological Environmental Studies Br

2 Atch  
1. Environmental Impact Statement  
2. Changes



Changes Recommended for

"Disposition of Orange Herbicide by  
Incineration - Environmental Statement"

22 November 1971

Change

- 1 P2, line 2. Two commercial facilities, one in Houston TX, the other in Saugat IL are capable of incinerating Orange herbicide.
- 2 P4, line 12. Biological Properties: Orange is a systemic hormone type herbicide which interferes with photosynthetic, respiratory, growth regulatory and other physiological systems in plants. Herbicidal applications of Orange defoliate and often kill broad leafed vegetation. Exposure to lower concentrations of Orange cause leaf curl and other growth abnormalities. Orange is effective against all broad leafed vegetation, however, some plant species (tomatoes and beans) are much more sensitive than others.
- 3 P4, line 19. The low vapor pressure of Orange indicates that its volatility is very low. However, the phytotoxic properties of Orange are such that abnormal growth and defoliation may occur to plants exposed to Orange vapors.
- 4 P4, line 23. Toxicological data: The acute, oral toxicity, LD50, of Orange to the rat is 566 mg/kg.
- 5 P4, line 31. Recommend Section B "Ester ingredients from Orange contract terminations" through P5, line 15 "Total gallons - 2,336,565" be included under P4, line 11 "...rubber are resistant.
- 6 P6, line 4. This impurity was tested and found to cause the teratogenic results similar to those attributed to 2,4,5-T. Subsequent tests by HEW investigators, and other competent scientists have shown that extensively purified 2,4,5-T is teratogenic and/or fetotoxic to various animal species. Critics of restrictions placed on 2,4,5-T resulting from these test results argue that the tests do not relate to actual human use. Proponents of the restrictions argue that the experimental data demonstrate that use of 2,4,5-T may result in increased incidences of human terata and fetal deaths, and that the possibility justifies such restrictions.

Physical, Chemical and Toxicological Properties of Dioxin Compounds:

"Dioxin" as used in this paper refers to 2,3,7,8 tetrachlorodibenso-  
F-dioxin (TCDD). Many other isomers of di, tri, penta, hexa, hepta

Atch 2

Change

6 (contd) and octa chlorodibenzodioxins also exist. However, analysis of Orange indicates that only the 2,3,7,8 tetrachloro isomer is present in Orange. Dioxin is nonvolatile, stable when exposed to air but unstable in the presence of U.V. light. Experiments indicate that the degradation of TCDD is a first order reaction with a half life of approximately 30 minutes under laboratory conditions using a General Electric R.S. Sunlamp. TCDD is formed at high temperatures from the condensation of 2,4,5-trichlorophenol. This condensation reaction occurs only under alkaline conditions. The possibility that 2,4,5-T could condense into TCDD during incineration is precluded by the acidic environment generated by the pyrolysis of the chlorinated herbicide and by the temperature of the incinerator. TCDD is unstable at temperatures of 800°C or higher. More data concerning the rate of pyrolysis, and pyrolysis products of TCDD at various temperatures, are currently being gathered by the USDA and will be available before incineration of Orange herbicide is initiated.

TCDD is an extremely toxic substance. The oral LD50 to rats is 22 to 44 ug/kg ( $2.2 - 4.4 \times 10^{-5}$  grams/kg). The LD50 to guinea pigs is 0.6-2.0 ug/kg. Acute animal exposure is followed by prolonged illness, liver injury and death - up to 48 days after treatment. The acute lethal dose to rabbits is the same whether the material is applied dermally or orally, indicating efficient dermal absorption. The physiological mechanism by which TCDD kills is unknown.

TCDD is excreted primarily via the liver and is stored preferentially in the fat. Very little is known concerning the metabolism of TCDD.

TCDD is also a potent toxin at exposure levels many times lower than fatal dosages. Ten daily dosages of 0.125 ug/kg to the dam are fetotoxic to rat embryos.

Single dosages of ug quantities of TCDD cause severe chloracne in humans and animals.

7 P6, last line. Add after last line - similar savings can be expected to the Saugeit Illinois facility.

8 P7, line 1. Environmental Factors.

Both the government and industry have had extensive experience shipping Orange herbicide via rail and water. This experience has produced an excellent handling confidence factor. No restrictions have been levied on transportation of Orange or its ingredient materials relative to corrosiveness, toxicity, flammability or other hazardous conditions.

Change

- 8 (contd) Transportation by land (road-rail) would require considerations for spillage. Spills can be absorbed by any good sorbent such as diatomaceous earth, collected and burnt in the incinerator facility. Any spill of Orange is a serious threat to plant life in the immediate vicinity. A large spill would certainly result in the loss of plant life in the immediate area.

Such a spill would threaten human life only if the accident resulted in extreme exposure.

Transportation by water would result in the same potential spillage problem. Lost drums would sink, but would be recoverable. The herbicide is heavier than, and immiscible with water, so liquid spillage tends to sink to the bottom of any waterway. A large liquid spill would cause a fish kill and kill the aquatic vegetation in the area. Spilled herbicide would deteriorate over a period of several months to carbon dioxide, chloride and water. Orange and Orange II are no more toxic than thousands of tons of commercial products shipped by water daily.

The environmental areas considered for herbicide destruction are Deer Park, Texas (also referred herein as the Houston, Texas area) and Sauget, Illinois. The method of transportation to be used and the routes of travel have not been determined.

Commence Page 8 line 4 "A commercial incineration plant..."

- 9 PB, line 17 & following. The incinerator is fueled with natural gas and is equipped with a venturri and packed column scrubbing system. Emissions are discharged from a 100 foot stack. The combustion temperature would be maintained at 2500°P at all times. This contractor has stated he intends to burn Orange in conjunction with other liquid wastes.

A commercial facility at Sauget, Illinois (Atch 4) just across the Mississippi River from St Louis, Missouri has an incinerator capable of burning Orange and its ingredient materials. Road, rail and barge transportation facilities are available at the plant site for handling drum shipments. Adequate storage space exists within the plant for drum storage.

This plant is a large organic chemical manufacturing site for one of the nations largest producers of chemical products. The plant employs 1303 employees. This plant manufactured Orange herbicide for 20 years ceasing operation in 1969. The plant has expert personnel in all aspects of herbicide chemistry handling and hazards.

Resume Page 10 line 24 "The incinerator was installed..."

Change

- 10 P 11, line 12-15. Include this paragraph after discussing incinerator.
- 11 P11, line 12. The empty drums would be flushed with a nonpolar solvent, crushed and smeltered for scrap steel. The flushing residue would be incinerated with the herbicide.
- 12 P11, line 19. ...including a water wash system, a venturri scrubber and a stacked column of polypropylene saddles. These scrubbers remove over 99% of the hydrogen chloride from the emitted gases. The water effluent is treated in a commercial waste treatment plant, and discharged into the Mississippi River. The stack effluent water is diluted over  $10^6$  fold after mixing with the Mississippi River.

The incinerator does not currently convert hydrochloric acid to sodium chloride. This could easily be accomplished by chemical additions to the blowdown water. As mentioned above, alkaline conditions are to be avoided during combustion of Orange.

The PCB's currently being burned by this facility contain an equivalent amount of chloride as Orange. Current hydrochloric acid emissions and discharges from this facility are 25% those authorized in the existing permit.

- 13 P8, line 23 through page 10, line 1. Insert above page 12, line 9 as follows:

Data published in the scientific literature indicates that the acid form of 2,4,5-T and 2,4-D pyrolyze rapidly and completely at 500°C and higher. The esters of these acids are thought to burn similarly. Similar data indicates TCDD is much more stable; burning completely at 900°C (1908°F). A combustion analysis program is currently underway with the Dept of Agriculture, to provide detailed studies and pyrolysis data on combustion of Orange and TCDD.

Continue through page 9, line 8.

- 14 P9, line 9. Combustion stack emissions and liquid effluent monitoring systems and test methods are being developed for inclusion in the contract work statement. Discharge limits will be established for 2,4-D, 2,4,5-T and TCDD and included in the work statement along with the requirement that the contractor be able to demonstrate on a daily basis that the incineration operation is meeting these standards

Continue through page 10, line 1, see Changes 28-29.

## Change

- 15 P12, line 5. The emptied drums will be chemically decontaminated with a nonpolar solvent, then crushed and the metal smeltered as scrap. The contaminated solvent will be incinerated under the same conditions as Orange. Commercial incinerators capable of burning Orange at a rate of 2 to 5 thousand gallons per day are located in Houston, Texas and Sauget, Illinois. Incineration will take 468 24-hour days at a burning rate of 5000 gallons per day.

### B. Probable Impact of Proposed Action on the Environment:

The probable environmental consequences of the proposed action will be insignificant. The incineration of 2,336,565 gallons of Orange herbicide will produce an increase in atmospheric carbon dioxide and an increase in the chloride content of the receiving waters. The drums will be decontaminated, crushed and smeltered for scrap metal. The project will be accomplished without a detrimental impact on the environment.

- 16 P13, line 10. A total of 44,600,000 lbs of carbon dioxide and 7,525,000 lbs of chloride will be generated and released into the surrounding environment.
- 17 P13, lines 12-15. Drum disposal substitute. The disposal of 42,483 55-gallon drums will recycle 1,700,000 lbs of steel within the economy.
- 18 P13, line 18. The herbicide vapors are phytotoxic and will kill vegetation. However, proven handling methods are known and will be required by contract which will insure plant injury will not occur.

Emissions and discharges from the incineration operation will be continuously monitored to insure it will not adversely effect human health, aesthetically or culturally valuable surroundings, standards of living or other aspects of life. The recycling of 850 tons of scrap steel will have no adverse environmental impact.

- 19 P14, line 8. Dispose of drums by landfill.
- 20 P16, lines 7-12. The disposal of drums, after decontamination, could be accomplished by crushing and burying the drums in a proper landfill area. This alternative would require additional expenditures for land use. The drums would eventually convert to ferric oxide. Although this action would not pose a pollutional threat, it may cause concern among environmental groups and would be more expensive than recycling the steel.

## Change

- 21 P16, line 20. The incineration of Orange herbicide in facilities such as those described herein and the smelting of decontaminated drums do not involve the irrevocable use of resources, changes in land use of resources, changes in land use, destruction of archeological or historical sites, unalterable disruption of ecosystems or curtailment of existing beneficial uses of the environment.
- 22 P18, lines 5-9. The emptied herbicide drums will be decontaminated and smelted as scrap steel. Suitable commercial incineration plants are located just south of Houston, Texas and in Sauget, Illinois.
- 23 P18, line 10 and following:
4. The environmental impact of the proposed action will involve the discharge of 44,600,000 lbs of carbon dioxide into the atmosphere and 7,525,000 lbs of chloride to the receiving waters. Small amounts of hydrochloric acid will be released to the atmosphere. These discharges would be made over a period of not less than 468 days. The carbon dioxide, chlorine, and chloride discharges will be controlled to meet all Federal, State and Local pollution regulations. Limits of discharge will be set for herbicide and a TCDD in both stack emissions and blowdown scrub water. The contractor will be required daily to demonstrate that these discharge limits are not being exceeded. Methods for accomplishing this will be included in the statement of work.
- 24 P19, line 4. h. Dispose of drums by landfill.
- 25 P4, line 26. Inhalation misspelled.
- 26 P4, line 28. Change sanitary to safety.
- 27 P6, line 4. Change DIOXIN to TCDD.
- 28 P9, line 16. The incinerators are equipped with scrubbers which will efficiently remove the chlorine, liberated during pyrolysis of Orange, from the effluent gases. This chlorine will be disposed of in compliance with all laws binding the individual contractor.
- 29 P9, line 21. Change 12,400,000 lbs of sodium chloride to 7,525,000 lbs of chloride. "...daily rate of discharge ... 16,000 lbs of chloride and 95,000 lbs of carbon dioxide..."

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

CC

25 February 1972

Supplying Herbicide Orange Samples to Civilian Firms for  
Experimental Purposes

SAAAIA/SF

1. SF's unclassified message concerning the USAF supplying civilian agencies with Orange herbicide for experimental uses has been reviewed (Atch 1). The following comments are offered for your consideration.
2. Recent AF policy concerning the redistribution of pesticides states that the AF will be certain that pesticides will not be released from AF stocks unless it is certain that they will be used as federally registered. Supplying small quantities of herbicide for experimental uses in the laboratory does not conflict with this policy. However supplying large quantities of pesticides for application in the environment other than those applications for which the material was registered is contrary to AF policy. The requests from Transvaal Inc. and from the New Mexico Salt Water Disposal Co. Inc. are of this latter type.
3. The request from the latter company is of a very controversial nature and may require an impact statement and its detailed review prior to accomplishment.
4. Recommend that any SF request for blanket approval to fill industry needs for Orange herbicide be limited to supplying a maximum of 20 gallons of material per company. Larger requests should be reviewed by HQ AFLC to determine appropriate action.
5. Recommend request for Orange samples by New Mexico Salt Water Disposal Co. be refused pending further information since deep well injection of Orange constitutes a MASQUE as defined by recent AF instruction.

FOR THE COMMANDER

**SIGNED**

RICHARD A. CALLAHAN, Ph.D.  
Capt, USAF, ESC  
Chief, Biological Env Studies Br

1 Atch  
SF Message

APPENDIX D

COMMENTS OF OTHER GOVERNMENTAL AGENCIES CONCERNING  
THE DRAFT ENVIRONMENTAL IMPACT STATEMENT

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Appendix C-4

MEMO FOR THE RECORD - 18 OCT 71

FOR OFFICIAL USE ONLY

PAGES WITHDRAWN

ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

Mr. Aaron J. Racusin  
Acting Assistant Secretary  
of the Air Force  
Office of the Secretary  
Headquarters USAF/PREV  
Washington, D.C. 20330

MAR 8 1972

Dear Mr. Racusin:

We have reviewed the U.S. Air Force draft environmental impact statement on the disposal of Orange herbicide by incineration.

The proposed action calls for the incineration of 2,338,900 gallons of Orange (including Orange II) herbicide over a 468-day period at either Deer Park, Texas or Sauget, Illinois.

We concur that the process of incineration if properly carried out under the appropriate conditions can effectively reduce the components of Orange to carbon dioxide and hydrochloric acid. However, these two gaseous effluents must be disposed of in such a way that they pose essentially no hazard to the environment. The final impact statement must provide additional information if we are to determine whether or not this project will be carried out in a way which is protective of public health and the environment.

We offer the following specific comments to assist you in the preparation of the final statement:

1. Special precautions should be taken to assure that efficient combustion conditions (product intake, temperature, and retention time) are maintained throughout the operation. These precautions are necessary to insure that the original material plus any intermediate pyrolysis products are burned completely and are not present in the stack effluent. Since the natural combustion properties of the herbicide will provide the fuel required, there should be no mixing of this herbicide with other combustible wastes as suggested for the incinerator in Deer Park, Texas.

2. The estimate of 468 days for the complete incineration is based on a feed rate of 5,000 gallons a day. If incineration is carried out at Sauget, Illinois, this time period must be increased to over 810 days since the incinerator capacity is only 2,880 gallons per day. No calculations were presented for the total volume of the wash from 42,483 barrels and the time for incineration of that wash.

3. Proper disposition of the hydrochloric acid is necessary if there is to be no adverse effect on the environment. At the Sauget, Illinois, incinerator, the daily volume of hydrochloric acid discharge is not given. Consequently we cannot calculate the concentration of the acid and the pH of the waste water. To assess the ability of the municipal sewer system to handle such a discharge over a long period of time, consideration should be given to the disposal of this waste dilute acid by sale, or free of charge, to companies who have need for such acid rather than disposing of it by sewer system discharge.

On the basis of the documented calculation of  $12.4 \times 10^6$  lbs. of sodium chloride produced in Texas, it was calculated for these comments that there will be  $7.7 \times 10^6$  lbs. of hydrogen chloride carried off in the liquid effluent at Sauget. For each of the 810 days of operation, this is approximately 9500 lbs. of hydrogen chloride.

The document indicates that approximately 95% of the total hydrogen chloride evolved in the incineration will be scrubbed from the effluent gas, the remaining 5% being exhausted to the atmosphere. Based on the same calculations as were used in the preceding paragraph, this is approximately 500 lbs. per day hydrogen chloride emission. Since the Sauget source is slightly east of a line drawn directly south from downtown St. Louis, and because the document indicates a prevailing southeast wind, it appears likely that this daily emission of 500 lbs. would fall into the area of downtown St. Louis most of the time. Because the draft environmental impact statement has not provided enough operating data on the incinerator at the Illinois site to calculate the concentrations of the hydrogen chloride emissions, it is impossible to accurately determine the effect of this amount of emissions on the surrounding community. It is safe to say however, that such an amount of emissions over such a long period of time could present a potentially serious condition.

It is felt that a correctly sized and operated sodium hydroxide scrubber added to the Sauget system would eliminate the hydrogen chloride problem completely. The sodium chloride and sodium carbonate produced by the scrubber could be disposed of by controlled discharge into the sanitary sewer system or directly into the river. In Deer Park, Texas, the absorption solution will be discharged into Tucker Bayou. There is not enough information to compute the expected plant effluent concentration of salt or sodium carbonate produced by the reaction of sodium hydroxide and carbon dioxide. This is important because salt equilibrium can affect the biota of estuarine systems and especially that of Tucker Bayou which has a variable rate of flow. The release of carbon dioxide into the atmosphere should pose no danger to the environment. We emphasize the necessity and the importance of compliance with Federal, State, and local air and water pollution control regulations.

4. Proper monitoring of the incineration process must be put into effect by both the contractor and the U.S. Air Force. Frequent periodic analyses of the stack gases and liquid effluent for unburned Orange pyrolyses products, hydrogen chloride, carbon dioxide, and ash (if any) must be made to assure that complete combustion is taking place. A technical representative should be present at the incinerator facility throughout the operation to assure that all combustion controls and scrubbers are functioning properly and to check on the monitoring operation and proper operational practices. Any breakdown in control measures or devices must be cause for stoppage of the operation until the problem is corrected.

5. The empty drums should be decontaminated with kerosene and an alkaline detergent and should be allowed to dry before being handled further. The preferred treatment of the drums should be either salvaging for further shipping uses or for smelting as scrap metal. Their disposal in landfill is the least acceptable alternative. If, however, this method of disposal must be used, the landfill site should be located on property so that there is no chance of runoff into streams, lakes, or groundwater systems.

6. The physical movement of 2,338,900 gallons of Orange from its present locations to the ultimate site of disposal is potentially a serious threat to the environment and we feel the draft statement does not give sufficient information on movement details, such as mode of transportation, off-loading, storage at disposal site, spill containment, decontamination, etc. We recommend the following: (1) careful observance of Department of Transportation safety requirements in the transport of hazardous materials; (2) spelling out of specific modes and routes of transportation so as to plan for any contingency that might occur; (3) separate and individual contingency plans covering such items as immediate field detoxification, health and safety considerations of personnel who might be involved in cleanup; (4) a firm written commitment from the transportation contractor that containment equipment is located and available to the contractor during transportation; and (5) pre-designation of the on-scene coordinator prior to any shipment.

Off-loading areas should be equipped with materials and equipment necessary for rapid cleanup, and off-loading equipment should be checked thoroughly before the commencement of each loading or unloading in order to assure safe and dependable operation. Furthermore, responsible persons engaged in off-loading should be given complete instructions in cleanup techniques along with instructions on how to proceed in case of a spill.

While shipment by water is cheaper than land and there has never been a spill during water transport, it might be recognized that material spilled in a waterway would be distributed by the current. A land spill could be much more easily contained. If shipment is made by rail or truck, cleanup teams and equipment should accompany the transport vehicles.

7. If the drums are deteriorating, consideration should be given to either redrumming or transfer to tank-cars. As some of the Orange will be held for up to 2 1/2 years at the disposal site, there is question as to the advisability of storing the Orange in drums at all. If the site has suitable bulk storage tanks available, these should be used. Shipping in bulk and building several storage tanks at the site might prove cheaper and safer than redrumming, shipping and storing drums.

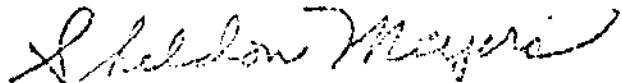
Because of the extensive precautions which should be taken during transportation and the possibility of contamination of other cargo in the event of leakage, we feel the use of Orange drums as filler cargo is inadvisable.

8. In the matter of storage, whether in bulk or in drums, only those areas especially designed for storage of hazardous materials should be used. Such areas should provide (1) structures to prevent surface water runoff from entering the area, (2) pavement and gutters to collect surface water runoff within the area, (3) drains to channel contaminated runoff to a holding facility, (4) materials and equipment necessary for rapid cleanup of spills, and (5) fencing to control admission to the areas. In addition, storage areas should be located remotely from occupied dwellings.

9. The alternative of building a new incinerator in a remote region should be examined in detail.

We appreciate the opportunity to review this draft environmental impact statement.

Sincerely yours,



Sheldon Meyers  
Director

Office of Federal Activities



February 18, 1972

Department of the Air Force  
HQ USAF/PREV  
Washington, D. C. . 20330

Dear Sir:

The draft environmental statement titled "Disposition of Orange Herbicide by Incineration," was received by the Department of Commerce for review and comment.

The Department of Commerce has reviewed the draft environmental statement and has the following comments to offer for consideration.

The key question is the completeness of combustion - i.e., the fraction not oxidized but carried up the stack. Once this can be estimated, then there is the atmospheric transport and diffusion problem to a point at the nearest habitation or unrestricted area around the disposal plant. There is a 100 foot stack. Combustion temperature is 2500°F (1400°C) for 3 second dwell (p. 11).

Tentative data show orange decomposes at 900° C. (but how long does it take at this temperature? e.g., water boils at 100° C, but a large pot of water must remain at this temperature for sometime before it boils away). See page 15 - the Illinois plant would release 5% of the HCl as a stack effluent.

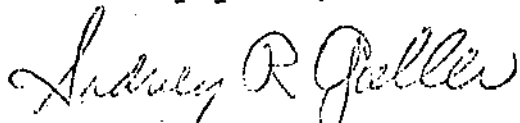
The combustion of gasoline in an auto engine is only partially complete - and hydrocarbons are emitted as wastes; i.e. unburnt gasoline. The combustion temperature of an auto is undetermined and the dwell time is about 4 millisecc, so the analogy may be poor.

The safety of this operation will also depend on how continuously reliable and constant are the actual temperature/pressure/dwell

conditions inside the burner - because it will take more than a year of continuous burning to complete the job. The constancy, uniformity and reliability of the contractors' facility are therefore important questions which probably should be treated in the statement.

We hope these comments will be of assistance to you in the preparation of the final impact statement.

Sincerely yours,



Sidney R. Galler  
Deputy Assistant Secretary  
for Environmental Affairs





DEPARTMENT OF AGRICULTURE  
OFFICE OF THE SECRETARY  
WASHINGTON, D. C. 20250

FEB 22 1972

Mr. Aaron J. Racusin  
Acting Assistant Secretary  
of the Air Force  
Washington, D.C. 20330

Dear Mr. Racusin:

We have reviewed the Draft Environmental Statement on "Disposition of Orange Herbicide by Incineration". Several questions have arisen that you may wish to consider.

The environmental statement does not contain data to show that effluent emissions would not contain biologically active dioxins. Data to show that dioxins are not emitted into the atmosphere must be provided. The statement does not provide for monitoring stack flume emissions from either of the proposed incinerators.

Information should be provided to assure that the orange herbicide remaining in the emptied containers does not have a higher concentration of dioxin than was present in the lot as a whole. Such a situation would arise if the dioxin settles to the bottom of a drum. If that happens, much of the dioxin would go into the soil instead of being combusted.

Damage to vegetation can occur from 2,4-D and 2,4,5-T in the vapor phase. Shipment of orange to the incineration site should be geared to incineration capacity so that large stocks are not kept in storage at the incineration site.

We believe the environmental impact statement must contain data on temperatures required for total combustion. The statement must also identify the effluent gases, and intermediate breakdown products. For example, incomplete combustion may occur when the incinerator is shut down. Intermediate combustion products may be potentially hazardous.

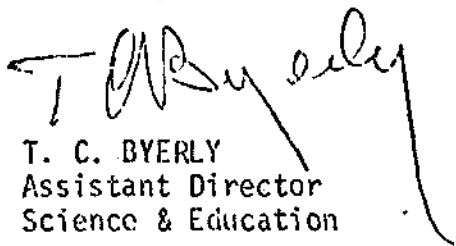
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The biological activity of the effluent gases must be documented and a scrubbing system specified that will assure safety.

The volume of water into which the sodium chloride is discharged and the rate and volume of fresh water inflow should be specified so that the increased salt content of the water can be determined.

We hope these comments are helpful to you.

Sincerely,

A handwritten signature in cursive script, appearing to read "T. C. Byerly", with a long, sweeping underline that extends to the right.

T. C. BYERLY  
Assistant Director  
Science & Education

Enclosure:  
1 copy of Environmental  
Statement



14. H. U.

EXECUTIVE DEPARTMENT  
DIVISION OF PLANNING COORDINATION

RESTON SMITH  
GOVERNOR

BOX 12428, CAPITOL STATION  
AUSTIN, TEXAS 78711  
PHONE 512 475-2427

ED GRISHAM  
DIRECTOR

February 25, 1972

*Orig to  
Air Staff - One Cart  
for info  
3-1-72*

Mr. Aaron T. Racusin  
Acting Assistant Secretary  
of the Air Force  
Headquarters USAF/PREV  
Washington, D.C. 20330

Dear Mr. Racusin:

The Office of the Governor, Division of Planning Coordination (State Planning and Development Clearinghouse), and affected Texas State agencies have reviewed the draft environmental impact statement for the disposition of Orange herbicide by incineration in Deer Park, Texas.

The Texas Air Control Board presently objects to the proposed project for several reasons which include insufficient technical information in the draft environmental statement and the possible harmful effects to the area by adding additional air pollutants to the atmosphere. The Texas Air Control Board (TACB) has statutory responsibility and authority in matters of air contamination.

The comments received from State agencies are enclosed.

Thank you for the opportunity to review this draft environmental impact statement.

Sincerely,

Ed Grisham  
Director

EG:gtt

Encl. (4)

cc: Mr. Charles R. Barden, TACB      Mr. Hugh C. Yantis, Jr., TWQB  
Mr. James U. Cross, TP&WD      Dr. James E. Peavy, TSDH  
Mr. A. T. Traynor, USAF

1100 WEST 49th STREET  
AUSTIN, TEXAS - 78756

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BERT W. WHITNEY, P.E.  
Vice-Chairman

February 14, 1972

Mr. Ed Grisham, Director  
Division of Planning Coordination  
Office of the Governor  
Capitol Station  
Austin, Texas 78711

Dear Ed:

Following are our comments on the Draft Environmental Impact Statement for Disposition of Orange Herbicide by Incineration, prepared by the Department of the Air Force in January, 1972:

"Information received since the draft environmental statement on the incineration of Orange herbicide from Kelly Air Force Base was written in October of 1971 makes it inadvisable to allow this operation to be conducted in the State of Texas at this time.

The following factors were considered in evaluating the proposal to incinerate the Orange herbicide in the Rollins Purle incinerator in Deer Park, Texas:

1. The information submitted in the impact statement does not indicate that alternate methods of disposing of the herbicide have been thoroughly explored, or that these methods will be more harmful to the environment than burning the herbicide would be.
2. Technical information submitted with the impact statement is insufficient to determine the feasibility of destroying great quantities of Orange herbicide by incineration. Although the impact statement indicates

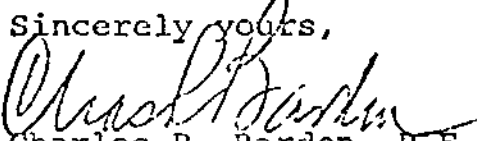
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that Rollins Purle, Incorporated will comply with air pollution control regulations, methods of compliance and technical data are lacking; and no mention is made of laboratory facilities or the analytical capability of the Rollins Purle facility. On page twelve of the impact statement, the gravity of the problem is indicated by reference to the need for complete destruction of the Orange material in order to avoid contamination of the environment with hazardous combustion materials or unburned herbicide chemicals. The next sentence reveals that combustion stack emissions and liquid effluent monitoring systems and test methods have not yet been developed. The ultimate responsibility for technical errors and accidents is not clear.

- 3. The area around the proposed site of incineration, Air Quality Region VII, is a highly industrialized area which has relatively high concentration of air pollutants. The addition of combustion products from the incineration of over two million gallons of Orange herbicide into the atmosphere of this area over a prolonged period could compound an existing problem and might very well prove harmful. It might be desirable to explore the possibility of incinerating the Orange in a federally-owned facility located in a relatively unpopulated area.

In view of the factors enumerated above, we feel that the destruction of Orange herbicide in the State of Texas, as outlined in the Air Force impact statement, would be imprudent at this time."

We appreciate the opportunity to comment on this project. If I may be of further service to you, please let me know.

Sincerely yours,  
  
Charles R. Barden, P.E.  
Executive Secretary  
Texas Air Control Board

cc: Mr. Jim Menke, Regional Supervisor, Baytown Regional Office



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JAMES U. CROSS  
EXECUTIVE DIRECTOR  
JOHN H. REAGAN BUILDING  
AUSTIN, TEXAS 78701

February 14, 1972

Mr. Ed Coker  
Division of Planning Coordination  
Executive Department  
Capitol Station  
Austin, Texas 78711

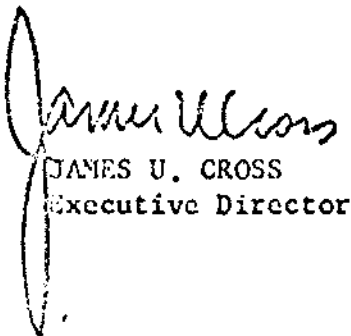
Dear Mr. Coker:

We have reviewed the draft environmental impact statement for the disposal of Orange Herbicide by incineration, and are in general agreement with the method of disposal and the draft statement.

We would recommend that stack gases be monitored for 2,4-D; 2,4,5-T and dioxin to prevent any escape to the atmosphere. If other precautions are taken to prevent loss or spillage of the barrels and if the empty barrels are disposed of properly, the Parks and Wildlife Department would not object to the incineration of Orange Herbicide.

We appreciate having had the opportunity to comment on this draft statement.

Sincerely,

  
JAMES U. CROSS  
Executive Director

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FEB 15 1972

WATER QUALITY BOARD



JAMES U. CROSS  
J. E. PEAVY, MD  
BYRON TUNNELL  
HUGH C. YANTIS, JR.  
EXECUTIVE DIRECTOR  
PH. 475-2651  
A.C. 512

DURALEIGH

314 WEST 11TH STREET 78701  
P.O. BOX 13246 CAPITOL STATION 78711  
AUSTIN, TEXAS

February 1, 1972

Mr. Ed Grisham, Director  
Division of Planning Coordination  
Office of the Governor  
Capitol Station  
Austin, Texas 78711

Dear Mr. Grisham:

In response to your memorandum of January 26, 1972, I would like to restate the comments of our letter of November 10, 1971, a copy of which is included in the Draft Environmental Statement for the Disposition of Orange Herbicide by Incineration by the Department of the Air Force.

In restating our previous opinion, I would like to suggest that insofar as water quality is concerned, no environmental statement or special permission is required so long as the disposal by Rollins Purle is carried out within the conditions of waste control order No. 01429 and so long as the final waste disposal of decontaminated drums is carried out pursuant to the statute. It should be understood that if the disposal of either the orange herbicide or the drums was proposed to be carried out contrary to the statute, then this office would decline to authorize the disposal.

Truly yours,

Hugh C. Yantis, Jr.  
Executive Director

go

All Board Members - Texas Water Quality Board  
Mr. Josiah Wheat, Legal Counsel, TWQB

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FEB 3 1972



# Texas State Department of Health

AMES E. PEAVY, M.D., M.P.H.  
COMMISSIONER OF HEALTH

AUSTIN, TEXAS

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ROYCE E. WISEDAKER, M. S. ENG.

J. B. COPELAND, M.D.  
DEPUTY COMMISSIONER

February 15, 1972

Honorable Preston Smith  
Governor of Texas  
State Capitol  
Austin, Texas 78701

ATTENTION: Mr. Ed Grisham

Dear Governor Smith:

The Draft Environmental Statement for the "Disposition of Orange Herbicide by Incineration," prepared by the Department of the Air Force has been reviewed by this Department.

In considering the proposal with regards to possible pollution of the waters of the State, we are in accord with the viewpoints expressed by Mr. Hugh C. Yantis, Jr., Executive Director of the Texas Water Quality Board, in his letter of February 1, 1972, to Mr. Ed Grisham, Director of your Division of Planning Coordination. Mr. Yantis stated that if the project is carried out in such a manner as to control the discharge so that the limitations set forth in Waste Control Order Number 01429 are not exceeded, no conditions will exist which will require special or extra permission.

However, when disposal by incineration is viewed from the standpoint of potential adverse air pollution conditions, we concur with the statements and recommendations offered by Mr. Charles R. Barden, Deputy Commissioner of the Air Control Section of this Department, who also serves as Executive Secretary of the Texas Air Control Board.

Respectfully,

J. E. Peavy, M.D.  
Commissioner of Health

RECEIVED

FEB 15 1972



APPENDIX E

TRIP REPORTS

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



REPLY TO  
ATTN OF: CC

21 October 1971

SUBJECT: Trip Report

TO: Commander, USAF Env Health Lab/CC, Kelly AFB TX 78241 ~~W.M.~~  
AFLC/SGPE  
IN TURN

1. Place: Monsanto Chemical Co., Krummrich Plant, St Louis IL
2. Inclusive Dates of Travel: 13 Oct 1971
3. Persons Making Trip: Capt Richard A. Callahan
4. Primary Mode of Transportation: Commercial Air
5. Purpose of Trip: To evaluate the capability of the Krummrich Monsanto Plant to dispose of large quantities of Orange herbicide by incineration.
6. Persons Contacted:

Mr. Wayne Vanderventer, SAAMA/SFQT

Mr. Chester Knowles, Jr., Monsanto Envir Chem, Chicago IL (Commercial Technology Director)

Mr. Gerry Bratsch, Organic Chemicals Div, Krummrich Plant

Mr. Art Leysi, General Superintendent of Production, Krummrich Plant

Mr. Michael Foresman, Pollution Control Engineer, Krummrich Plant

7. Findings and Observations:

a. The William G. Krummrich Monsanto Plant is located on a 134 acre tract in Sauget IL, a subdivision of East St Louis. The plant manufactures a diversified group of organic chemicals including isomers of nitrobenzene and organic and inorganic acids. Available freight facilities include rail, truck, barge and aircraft. The plant employs 926 wage and 377 salaried employees.

b. Monsanto is familiar with the problems associated with Orange herbicide. The Krummrich Plant manufactured this herbicide from 1950-1970, and is one of the few concerns in the nation with experience in analytically measuring dioxin compounds in herbicides. Plant personnel are experienced in the occupational health hazards associated with this material.

c. The Monsanto incinerator is located centrally in the Krummrich installation and has been in operation since May 1971. The surface surrounding the facility is curbed concrete with underdrains leading to sump pumps. Any spilled product could easily be washed into the sumps from which it is pumped to the incinerator. Herbicide incinerated in this facility would undergo the following sequence of events:

(1) Steam preheating of the drum contents to insure product is in liquid form.

(2) Contents of barrel dumped into chemical wet well.

(3) All barrel lifting accomplished by specially equipped fork lift.

(4) Product pumped from wet well to storage tank (capped with nitrogen blanket) to incinerator injector nozzle.

(5) Herbicide preheated in nozzle and atomized into primary combustion chamber of incinerator.

(6) Combusting gases then pass into a large combustion area venting through the floor to a three stage water scrubber. Retention time in the incinerator under present operating procedures is three to four seconds. Operating temperatures can be set up to 2,800°F (1,538°C).

(7) Combustion products pass through a water scrubber, a Venturri scrubber and a packed column (polyethylene) scrubber. Presently, the incinerator is burning polychlorinated biphenyls (PCB's); a controversial environmental contaminant containing a similar chlorine content as the herbicide Orange. Monsanto claims the chlorine content of stack emissions is only twenty-five percent of their State Permit.

(8) Acidic stack scrubbing water receives primary treatment at a municipally-owned industrial waste treatment plant prior to discharge to the Mississippi River. The primary safety factor afforded by this plant is dilution. The overall dilution of the incinerator scrub waters after passing through the treatment plant and mixing with the waters of the Mississippi will be approximately one millionfold. This is a substantial advantage over facilities offering low initial dilution. Discharges from the Monsanto incinerator to the waste water treatment facility are currently meeting all sewer codes of that facility.

(9) The Krummrich plant has no current means of decontaminating empty herbicide drums. Presently, the plant is shipping such drums to a local concern which decontaminates them with solvents and then either recycles the drum or sells them for scrap. The Krummrich plant incinerates the drum washings. The plant does have a certified landfill area available to bury decontaminated drums. Space in this landfill is at a premium.

d. Mr. Bartsch stated that Illinois State Law requires an individual permit for the incineration of such wastes as PCB's and herbicides. He stated the Krummrich incinerator stack emissions and waste scrub water are monitored daily for specified chemicals, including PCB's under the conditions of the PCB permit. The incinerator and stack have appropriate sampling ports installed. Analyses are done in the Krummrich Chemistry Laboratory which employs some 15 chemists. Monsanto enjoys a tremendous advantage over many companies who may be interested in disposal of Orange by having the facilities and personnel to run a complete monitoring program in-house. Additionally, Monsanto's Central Chemistry Research Facility is located twenty miles from the Krummrich Plant in West St Louis. Expert toxicological, chemical and engineering experience is therefore available to this facility on very short notice.

#### 8. Recommendations or Conclusions:

a. The Monsanto Krummrich Plant appears potentially capable of disposing of herbicide Orange by incineration. The incinerator operating temperature and retention times are adequate to decompose the herbicides. Monsanto's strengths, besides its physical facility, are the experience at Krummrich of manufacturing Orange herbicide and its in-house toxicological, chemical, engineering expertise.

b. The two outstanding weaknesses of this facility are its distance from Gulfport and its current inability to decontaminate large numbers of empty 55-gallon drums. The company may be able to successfully counter these disadvantages by savings in monitoring the incinerator discharges and by subcontracting drum decontamination or by installing such a capacity.

c. Recommend the Environmental Impact Statement be rewritten to include the East St Louis area.

d. This Laboratory previously recommended (Atch 1) that recycling of decontaminated drums, either for reuse (valued at \$30,000) or for scrap steel, is advantageous to burial in a landfill.

e. Recommend Impact Statement be altered to recommend recycling of physically sound drums and smelting of all others rather than disposal by landfill.

*Richard A. Callahan*

RICHARD A. CALLAHAN, PhD, Capt, USAF, BSC  
Chief, Biological Environmental Studies Br

2 Atch  
1. Ltr, USAF Env Health Lab/CC w/a  
2. Ltr, " " 13 Sep 71  
Cy to: USAF Env Health Lab/CC  
McClellan AFB CA 95652

DEPARTMENT OF THE ARMY  
USAF ENVIRONMENTAL HEALTH CENTER (EHC) 45433  
KELLY AIR FORCE BASE TEXAS 78147

CC

10 November 1971

Trip Report - Dow Chemical Company (Capt R A Callahan & Mr Leopoldo Rodriguez)

Commander, USAF Env Health Lab/CC, Kelly AFB TX 78241  
AFLC/SGPE, WPAFB OH 45433

1. Place: Dow Chemical Company, Midland, Michigan
2. Inclusive dates of travel: 26-28 October 1971
3. Persons making trip: Capt Richard A. Callahan & Mr Leopoldo Rodriguez
4. Primary mode of transportation: Commercial Air
5. Purpose of trip: To confer with expert Dow personnel concerning the toxicity, chemistry quantitative and qualitative analytical methods and industrial hygiene procedures used by Dow when analyzing tetrachlorodibenzodioxins.

6. Persons contacted:

Kenneth Honeycutt, Manager Eng Serv, Gov Affairs Dept  
Joseph S. Natzic, Proj Manager, Env Control Syst  
Arnie R. Lietz, Eng Serv, Gov Affairs Dept  
Robert J. Pierce, Waste Control Eng  
Terry Batchelder, Aquatic Biologist, Waste Control Dept  
Warren B. Crummett, PhD, Organic Section Leader, Analytical Lab  
Howard Gill, Section Leader, GLC Anal Lab  
Rudy H. Stehl, PhD, Anal Methods Development Lab  
Ray A. Plepys, PhD, Business Manager, Anal Serv, Midland Div Anal Lab  
Richard R. Papenfuss, PhD, Anal Development  
Allen T. Maasberg, Dir Res Devel Gov Affairs Dept  
Fred K. Quigley, Dir Gov Affairs  
Verald R. Rowe, Dir Chem Biol Research (Corp)  
Perry J. Gehring, PhD, Asst Dir Chem-Biol Res  
J. David Wilson, Waste Control Engr  
Charles E. Hamilton, Waste Control Engr  
Harold R. Hoyle, Manager Indust Hygiene Sec  
Arnold W. Schaffer, Manager Env Health Serv  
Etcyl H. Blair, PhD, Manager, Res & Dev Agric Organic Chem (Corp)  
Keith C. Barrons, PhD, Res Biologist, Ag Organic Chem

7. Findings and Observations:

a. All of the information gathered on this tour is considered proprietary by Dow Chemical; therefore, this report will be general in nature.

b. The following facilities were toured:

(1) Dow Chemical Waste Treatment facilities including:

- (a) Bioassay capability
- (b) Sewage treatment plant
- (c) Rotary Kiln incinerator
- (d) Liquid chemical (chemical tars) incinerator
- (e) Waste water spill monitoring system
- (f) 2-4-D stack emission monitoring system

(2) Midland Division Analytical Laboratory

(a) Discussed analytical techniques and chemical properties of dioxin.

(b) Toured laboratory in which dioxin chemistry is performed.

(3) Dow Chemical Company Biological and Toxicological Laboratory - discussed the toxicity, mode of action and laboratory handling of dioxins.

(4) Midland Division Industrial Hygiene Section - reviewed laboratory handling procedures and contamination monitoring of dioxin compounds.

(5) General discussion of the problems associated with the disposal of orange herbicide. Discussion group consisted of:

(a) Ercyl H. Blair, PhD, Corporate Manager of Research and Development Agricultural Organic Chemicals.

(b) Keith C. Barrons, PhD, Research Biologist, Agricultural Organic Chemicals.

(c) Mr. Kenneth E. Honeycutt, Manager, Engineering Services, Government Affairs Dept.

(d) Mr. Arnie R. Lietz, Engineering Services, Government Affairs Department.

(e) Capt Richard A. Callahan, PhD.

(f) Mr. Leopoldo Rodriguez, M.S.

(g) Discussion limited to the problems associated with dis-

posing of orange herbicide. Although mention of the Dow Proposal #97125 was made by Mr. Honeycutt, specific discussions of this, or mention of any other proposal was completely avoided. Dr. Blair indicated he was very concerned that disposal of orange herbicide would be made without a proper monitoring program. After discussing the political climate of this action and the monitoring program this laboratory is in the process of formulating, he appeared somewhat mollified; although concerned about the final execution of this program.

#### 8. Recommendations and Conclusions:

a. This laboratory is capable of working with dioxin materials.

b. The best analytical method for both quantitative and qualitative analysis of this material is Gas Chromatography Mass Spectrometry (GC/MS). Dow uses an LKB 9000 GC/MS System. This laboratory's GC/MS System may lose dioxin compounds in its separator and therefore be unusable for dioxin analyses.

(1) Electron capture gas chromatography is quantitatively sensitive (10 ppb) to tetrachlorodioxins but is subject to positive interference by some other chlorinated hydrocarbons requiring extensive clean-up of the samples. The Dow experience with these materials includes their identification in chemically complex tars-residual materials from large scale chemical manufacturing operations. Under these conditions H.C.-GLC dioxin recoveries are often falsely high-never low. However emissions from a high temperature incineration process of orange herbicide will contain very little if any such interfering substances. Laboratory investigations can readily determine if GLC alone is adequate for emission monitoring.

c. Recommend:

(1) This laboratory immediately initiate the following investigation:

(a) Calibrate the sensitivity of its GLC and GC/MS Systems to tetrachlorodibenzo-p-dioxin (TCBD).

(b) Test collection and extraction procedures of TCBD from spiked air and water samples.

(c) Analyze the incomplete combustion products of spiked orange samples to determine if emission products which interfere with GC analysis of TCBD are generated. No attempt should be made by this laboratory to identify what other products are generated by incomplete incineration. This important aspect of the problem is being accomplished by contract.

(2) The information that will be generated by the above experiments is absolutely essential in order for this laboratory to assist the

Fuels Directorate in preparing the impact statement and statement of work for the incineration of orange herbicide. Deputy Assistant Secretary Moseman verbally requested the laboratory provide such assistance at the SAAMA meeting of 5 October 1971.

(3) A chemist be assigned full time to accomplish the above recommendations.



RICHARD A. CALLAHAN, PhD  
Captain, USAF, BSC, Chief, Biological  
Environmental Studies Branch



LEOPOLDO L. RODRIGUEZ  
Chemist  
Pesticide Branch

1 Atch  
Agenda



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



REPLY TO  
ATTN OF: CC

8 March 1972

SUBJECT: Trip Report

TO: Comdr, USAF Env Health Lab/CC  
Kelly AFB TX

THRU  
AFLC/SGPE

1. Place: Transvaal Inc., Jacksonville, Arkansas.
2. Inclusive Dates of Travel: 3 March 1972.
3. Person Making Trip: Captain Richard A. Callahan.
4. Primary Mode of Transportation: Commercial Air.
5. Purpose of Trip: Evaluate the potential of a Transvaal proposal to dispose of Orange Herbicide and to discuss this potential with Deputy Supply and Maintenance, Mr. Moseman.
6. Persons Contacted: Department of Defense:

Mr. F. K. Moseman, Deputy Supply and Maintenance to Assist. Secretary I&L., USAF.  
Dr. C. E. Minerak, Chief, Plant Sciences Lab., Fort Dietrick.  
Mr. C. W. Carter, Fuels Branch, HQ, USAF.  
Colonel F. Chanatry, Assist. Deputy Supply Serv., HQ, USAF.  
Dr. R. Scott, Chem. Scientist, DOD, Explosives Board.  
Mr. B. Knasel.  
Lt Colonel A. J. Wilkens, SFQ  
Mr. W. Vandeventer, SFQ  
Captain R. A. Callahan, EHL/K

Transvaal Inc.

Mr. M. F. Wilkerson, President  
Mr. J. A. Herholdt, Vice-President, R&D  
Mr. J. S. Calloway, Secretary-Treasurer  
Dr. A. E. Sidwell, Director of Research  
Mr. T. Bennett, Plant Manager

## 7. Findings or Observations:

a. Transvaal proposes to separate the Butyl herbicides from their contaminants - including TCDD - and to reformulate the 2,4,5-T and 2,4-D esters for commercial use. The TCDD components would be destroyed either by chemical, biological, or incineration techniques. Although the proposal cites some nonsensical objections to the incineration of Orange it has substantial merit. Recycling Orange and Orange II reduces the disposal of degradation product since reclaimed materials will be reformulated for registered uses. Although Transvaal claimed that only 60 percent of the material is recoverable; questioning revealed this was on a per run basis. Actual distillation efficiencies for this very pure product may run in excess of 90-95 percent. At the very least then this plan greatly reduces the volume of material to be disposed of.

b. Additionally Dr. Sidwell of Transvaal stated that he had data that indicated he could destroy the TCDD residue, thought to be contained in the distillation dregs, conveniently by splitting the ether bonds of the molecule. Supporting evidence he claimed was inconclusive, no supporting data was presented at the meeting. Transvaal suggested the AF support a \$16,000 - \$50,000 proposal over a five week period to prove this hypothesis.

## 8. Recommendations or Conclusions:

1. This avenue of disposal is worth further investigation.

2. Splitting the ether bonds of TCDD will likely yield dichlorophenols or phenates. These are common organic chemicals, easily disposed of by several conventional methods.

3. Any research performed by Transvaal must establish:

(a) What distillation fraction the TCDD is in.

(b) If the TCDD can be degraded as hypothesized by Transvaal.

(c) What products result from this degradation.

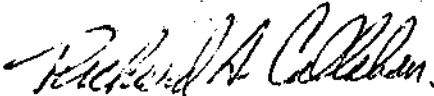
(d) Detailed disposal methods for all waste materials.

(e) Transvaal has levied the stipulation that, "No restrictions be placed on the disposal of products, except TCDD." (Pg. 13, Para. D. 1). The USAF must know that the products reclaimed from this operation will be used only for the purposes and under the conditions specified in the Memorandum from the Office of the Assistant Secretary of Defense, Health and Environment of 10 December 1971. (It is assumed that the phenoxy esters once distilled from the Orange formulation will be classified as non-tactical herbicides as defined by this Memorandum.)

(f) The detailed Transvaal disposal plan must contain a feasible monitoring capability that establishes what concentrations of 2,4-D, 2,4,5-T esters and TCDD are released to the environment during the re-distillation process. This system will include methods for blow-down water from the distillation condensation units and any other identifiable area of possible environmental contamination.

(g) The instrumentation of the Transvaal research laboratory is very limited. Prior to receiving AF funds Transvaal should be required to demonstrate to qualified technical personnel that they can in fact perform the required tasks.

(h) The recovery and resale of these components is technologically simple and has the potential of yielding large profits to Transvaal. Recommend that the appropriation for initial investigation be limited to \$10,000 or less. This recommendation assumes Transvaal does not demonstrate that more money is absolutely essential.



RICHARD A. CALLAHAN, PhD  
Captain, USAF, BSC  
Chief, Biological Env. Studies Branch

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



REPLY TO  
ATTN OF:

CC

21 March 1972

SUBJECT:

Trip Report - HQ USAF/SAPIL, Wash DC 20330

TO: Commander, USAF Env Health Lab/CC, Kelly AFB TX 78241  
AFLC/SGPE, Wright-Patterson AFB OH 45433

1. Place: HQ USAF/SAPIL, Wash DC 20330
2. Inclusive Dates of Travel: 9-10 Mar 1972
3. Person Making Trip: Capt Richard A. Callahan
4. Primary Mode of Transportation: Commercial Air
5. Purpose of Trip: To discuss with various HQ USAF/AFLC/SAAMA/AFRPL/ and EHL/personnel possible destruction methods for the herbicide Orange.

6. Persons Contacted:

9 Mar 72--

Chanatry, Fred I., Col, HQ USAF/AF/LGS  
Carter, C. W., HQ USAF/AF/LGSKE  
Price, C. E., Lt Col, HQ USAF/AF/LGSKE  
Goins, William D., HQ USAF/AF/PREED  
Bayer, J. E., Maj, HQ USAF/AF/PREV  
Sales, Jerry, AF Rocket Propulsion Lab, EAFB  
Johnson, C. A., Consultant to AFRPL  
Heaton, S. P., AFLC/DST W-PAFB  
Wilkins, A., Lt Col, SAAMA/SFQ  
Vandevanter, W. E., SAAMA/SFQT

10 Mar 72--

Chanatry, Fred I., Col, HQ USAF/AF/LGS  
Carter, C. W., HQ USAF/AF/LGSKE  
Bayer, J. E., Maj, HQ USAF/AF/PREV  
Heaton, S. P., AFLC/DST W-PAFB  
Wilkins, A., Lt Col, SAAMA/SFQ  
Vandevanter, W. E., SAAMA/SFQT

7. Findings or Observations: The meeting was in response to a memo from Gen Blank stating that Mr. Whitaker, was concerned that all possible avenues for disposing of this product had not been adequately investigated. Therefore, the purpose of the meeting was to fully investigate all these avenues. The following table summarizes the methods of disposal considered, their approximate completion dates, and costs, and an estimate of the adverse political impact anticipated by each. This latter parameter should be considered as cost factor in

the decision making process. The table reflects the personal opinion of the undersigned and does not necessarily reflect the consensus of opinion at the meeting.

<u>Method of Disposal</u>	<u>Approximate Completion of Disposal</u>	<u>Cost (millions of dollars)</u>	<u>Adverse Political Impact 1-5</u>
<b>1. Incineration:</b>			
a. Contractor Disposal	Dec 76	1.0	2
b. Purchase of Incinerator:			
(1) Incineration in CONUS	Sep 78	1.5	2
(2) Incineration on Johnson Island	Jan 76	2-3	1
<b>2. Use - Registration or Waiver of Registration of Orange Herbicide</b>	depending on need	0.5	2+
<b>3. Recycle:</b>			
a. Transvaal Proposal	May 75	1-2	1+
b. Recycling by other companies under investigation		-	-
<b>4. Microbial Degradation</b>			
a. Soil Degradation	Jun 73	0.5	3
b. Activated Sludge	1978?	Unk	3
<b>5. Deep Earth Disposal:</b>			
a. Deep Well Injection	May 73	1.07	5+
b. Cold Coal Hole Disposal (Using Former Underground Nuclear Test Site)	May 73?	0.57	2+

**6. Conclusions:** The best and cheapest method of disposal appears to be by use. This is a standard method of disposing of waste pesticides. The problem here would be either registering the material or obtaining a waiver from EPA. So far, such approaches to EPA officials have met with negative results. However, lateral approaches from high levels in the DOD to the equivalent EPA level may be very successful. This avenue should be thoroughly investigated. The environmental risks of long term storage should be emphasized.

a. The second cheapest and fastest method appears to be soil disposal. This method has the disadvantage of being coupled with substantial adverse political impact. However this method merits thorough investigation.

Site selection is a very critical factor if this method is to be pursued. Immediately after application of the herbicide a period (or periods, if multiple applications are made) of very high risk will ensue due to the possibility of translocation within the soil, into the air or surface waters. Control of the herbicide is lost immediately after application. A significant problem with this technique is the possibility of ground water contamination. Thorough knowledge of the soil profile, the geology, the ground water levels and rainfall patterns of the area will be necessary before an intelligent impact statement of this method can be written.

b. The third fastest method of disposal - contractor incineration - is the best understood method. Most of the adverse political impact involved in this method of disposal has already been absorbed by the USAF. The method is fairly rapid and the material is completely destroyed upon disposal. This is therefore a single risk technique. Since the material will be thoroughly destroyed during this disposal process; no residue remains to cause future problems. This is an advantage over the previous two methods, where a recycled product, possibly undegraded herbicide or a contaminant, may cause repercussions long after disposal is complete. Another unique advantage of this method is that the herbicide is always under human control. The destruction process can be monitored and altered at any time.

c. Recycling the product for reformulation and reuse has some appeal. Its disadvantages are time, cost and at this time - very little feasibility data (See Atch).

d. The last method of disposal - deep earth disposal - has the advantages of being rapid and competitively priced. However, it has glaring disadvantages in the anticipated strong adverse political impact and the fact that control over the material is lost upon disposal, yet the material will persist for years. Cold hole disposal, however, may have considerably less political impact, and merits further investigation.

9. AFRPL has been requested by USAF to investigate the incineration products of Orange herbicide. Dr. Sales indicated his people will contact this Laboratory for information.

#### 10. Recommendations:

a. Using Orange Herbicide on federal property under the proper conditions is the superior method of disposal and should be pursued as such.

b. Incineration by contractor offers a good mix of advantages and has no glaring disadvantage.

c. The chief competitor of incineration as a destruction method is soil biodegradation. This method should be thoroughly pursued.

d. The other methods should be investigated as contingencies.

e. This Laboratory supply AFRL with data when requested.

SIGNED  
RICHARD A. CALLAHAN, Ph.D.  
Capt, USAF, BSC  
Chief, Biological Env Studies Br

1 Atch A/S  
Copy to:  
AFIC/SG  
USAF/SGAAP  
USAF, PREV  
USAF/LGSKE  
USAF/EHL/CC  
McClellan AFB CA 95652

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



REPLY TO  
ATTN OF: CC

3 April 1972

SUBJECT: Trip Report - Mississippi State University

TO Commander, USAF Env Health Lab/CC, Kelly AFB TX 78241  
AFIC/SGPE, Wright-Patterson AFB OH 45433

1. Place: Mississippi State University
2. Inclusive Dates of Travel: 22 - 24 March 1972
3. Person Making Trip: Captain Richard A. Callahan
4. Primary Mode of Transportation: Commercial Air
5. Purpose of Trip: To collaborate with Mississippi State scientists concerning the pyrolysis of Orange Herbicide.
6. Persons Contacted:

W. Vandeventer, SAAMA/SF, Kelly AFB TX  
Walter K. Porter, Jr., Assoc. Director, Miss. Ag. and For.  
Experiment Station, State College, Mississippi  
William R. Fox, Head, Agric. and Biological Engineering  
C. Dale Hoover, Head, Agronomy Department, Mississippi State  
University Experiment Station, State College, Mississippi  
Ben F. Barrentine, Head, Biochemistry Dept., Mississippi State  
University, State College, Mississippi  
B. J. Stojanovic, Soils Microbiologist, Agronomy Dept.  
M. V. Kennedy, Biochemist, Biochemistry Dept.  
Fred L. Shuman, Jr., Assoc. Agriculture Engineer, Agricultural and  
Biological Engineering Department

7. Findings or Observations:

A. Doctors Stojanovic, Shuman and Kennedy formed a multidisciplinary research team investigating the biological, chemical and engineering aspects of pesticide disposal in 1967. They constitute the oldest and best established team in this area in the national academic community. Doctor Stojanovic and the undersigned corresponded in 1969-1970 concerning the incineration of herbicides. Therefore meeting with this team was a pleasurable and stimulating experience. Their research publications are well known and of excellent professional quality.

B. Mississippi State is using a three pronged experimental approach to provide information concerning the combustion of Orange Herbicide. These approaches are:



(1) The combustion of boats of Orange Herbicide in a static tube incinerator. a. A "boat" containing gram quantities of Orange are sealed in a heated combustion tube. Oxygen or air is fed into the tube and emission products collected in organic solvent scrubbers. b. This device provides some insight into possible degradation products of Orange up to a given temperature. However; since the herbicide is introduced in slug amounts; retention time, average emission gas temperatures and oxygen fuel ratios fluctuate wildly while the sample is combusted. Therefore combustion products from this device are not directly comparable to those expected from an incinerator with a 2-4 second retention time and a constant, steady feed of herbicide and air. c. Gaseous emissions and residue from this process are being analysed by gas liquid chromatography (GLC) for the NB esters of 2, 4-D; 2, 4, 5-T and TCDD (2, 3, 7, 8, tetrachlorodibenzoparadioxin). Doctors Kennedy and Stojanovic agreed that positive qualitative identification of other GLC peaks being observed could not be made by GLC alone or by X-ray Crystallography. d. The Mississippi State team does not have ready access to the instruments necessary to qualitatively identify these intermediate pyrolysis products of Orange and TCDD.

(2) Combustion of Orange Herbicide by Differential Thermal Analysis. (DTA)

a. Samples of Orange Herbicide are exposed to a programmed rise in temperature from ambient up to 1200°C. Chemical rearrangement of the molecules either absorb heat (endothermic reaction) or release heat (exothermic reaction). These changes in the temperature of the sample are recorded relative to the background temperature of a blank.

b. DTA provides information concerning the critical temperatures at which compounds undergo molecular rearrangements. Devices are available (although the MSU team does not currently have them) that measure the change in weight associated with a given rearrangement.

c. This technique has all the disadvantages of the former. Combustion products generated under the conditions of this instrument may be very different than those of a flow through incinerator.

d. The DTA data, reported in the MSU team's 1st and 2nd preliminary reports, indicated that under the pyrolysis conditions of this experiment the NB esters of 2, 4-D and 2, 4, 5-T degraded completely at 530°C, but that TCDD was not completely degraded until over 900°C. This is valuable data.

(3) Combustion of Orange Herbicide in an intermediate size-flow through incinerator.

a. Construction of a three stage incinerator with a capacity to burn 11-12 gallons/minute liquid wastes is nearing completion in the

Mississippi Delta area. This \$28,000 research tool will be used to provide data concerning the combustion efficiency of Orange Herbicides.

b. Data from this facility will be very meaningful concerning the emissions of the NB esters of 2, 4-D, 2, 4, 5-T and TCDD expected from a large scale incinerator.

c. This facility is, as yet untested. The comparative efficiency of this unit relative to the commercial facilities under investigation will be necessary for meaningful analysis of Orange incineration data.

d. The scrubbing system of this facility is as yet incomplete.

e. This facility will not contribute knowledge of the intermediate pyrolysis products of Orange discharged during incineration since MSU does not have the capability of identifying these products.

C. The research contract at MSU is not addressing the question of intermediate pyrolysis products generated with the incineration of Orange herbicide. Discussions with the MSU scientists indicated that they had not been asked to furnish such data. They were briefed on the USAF's political vulnerability in this action and on the tentative refusal of Illinois and Texas to have the incineration performed in those states. They agreed that knowledge of the intermediate pyrolysis products of Orange and TCDD could be a central issue when the AF reapproaches these states and the EPA for permission to contract incinerate Orange. They agreed that a combination of GLC and Mass Spectroscopy (GLC, MS) would supply such data.

(1) Dr. Stojanovic stated that the MSU team would explore the possibility of obtaining GLC, MS analyses of Orange combustion products commercially.

(2) Dr. Stojanovic offered to expand the scope of the DTA analysis in tandem with the GLC, MS analysis at a cost of \$10,000 - \$12,000 (DTA expansion alone).

D. Mr. Vandeventer asked the MSU team if they would be available to brief officials from Hq USAF, and the state of Texas at Kelly on the pyrolysis of Orange Herbicide in early June. Mr. Vandeventer stressed that such a meeting was tentative. The MSU team agreed assuming appropriate travel funds were made available.

E. The undersigned presented the MSU team with a copy of the monitoring program prepared by this Laboratory (Atch 1) and requested their written comments. The fact that many of the parameters mentioned in the report, such as the incineration temperature and the air/fuel ratio etc, are tentative pending the conclusion of their research was stressed.

F. The MSU team showed interest in the flow through incinerator designed and built in this Laboratory and used to generate data used in writing the monitoring program (Atch 1). This incinerator overcomes many of the experimental problems mentioned in paragraph B (1) and (2) above - namely it closely approximates the air fuel mixtures, dwell times, temperatures and the aerosol fuel injection method of the large commercial incinerators. The fact that - small incinerators inherently have larger surface: volume ratios than large incinerators and that this incinerator was a conductively heated versus the flame fired commercial units was mentioned. The MSU team stated they plan on constructing a similar laboratory unit, using the same fuel injection running system. They were informed that such a pump could be loaned to them from this Laboratory. Diagrams of the unit built by this Laboratory were left with the MSU team (Atch 1).

#### 8. Conclusions:

A. The research conducted at MSU will provide much of the information concerning the combustion of Orange herbicide required by the EPA and other interested government and special interest groups.

B. This research is being performed in a sound and correct scientific manner by this competent research team.

C. This research effort will not identify the partial degradation products of Orange and Orange II herbicides. The USAF should anticipate being required to demonstrate that no toxic intermediate degradation products are released during incineration of Orange. This Laboratory understood this research would generate this data - ARS letter of 26 Nov (Atch 2) para 1 "... to assure that no harmful effluent gases are emitted to the atmosphere" and para 2, 1 "Determine the temperature required for complete thermal degradation, the degradation products, and the volatile gases of Orange herbicide containing dioxin." The MSU team understood this requirement to concern only the ultimate combustion products.

D. A decision is necessary concerning the detailed strategy to be used in reapproaching the EPA and the states of Texas and Illinois concerning the incineration of Orange herbicides.

(1) The AF is currently in a position to:

a. Monitor the discharges of the NB esters of 2, 4-D and 2, 4, 5-T and TCDD in the effluent gases and blow down water from commercial incinerators at the ppb level. (EHL/K monitoring program atch 2)

b. Provide data indicating the NB esters of 2, 4-D and 2, 4, 5-T and TCDD thermally decompose at 550°C and 920°C under the conditions of the DTA and static incinerator mentioned above (MSU data).

c. Provide limited data indicating the total discharges of the esters of 2, 4-D plus 2, 4, 5-T and TCDD were  $2 \times 10^{-6}$  and 3% respectively of the inputs to a scale flow through burner with dwell times of 2 to 4 seconds at a temperature of about 900°C. (EHL/K data)

(2) MSU will increase the data in 8, D (1) b and c above and provide additional data concerning the emissions of the esters of 2, 4-D and 2, 4, 5-T and TCDD from an intermediate sized flow through incinerator.

(3) The only weakness in this program is the unknown quantity and chemical nature of incompletely combusted herbicide and TCDD. These oxidation products are expected to be innocuous at the concentrations present in the gas and water discharges. Identification of these products would remove all reasonable remaining questions concerning the safety of contract incineration.

a. This Laboratory can supply this data by June 1, 1972 provided the Finnigan Model 3000/150 system, presently in the FY 73 procurement appropriation budget is immediately ordered.

#### 9. Recommendations:

A. Recommend the meeting between Hq USAF personnel, SAAMA/SF and the State of Texas personnel mentioned by Mr. Vandevanter should include representatives of the State of Illinois EPA, respective regional representatives of the Federal EPA and personnel from the Environment Health Laboratory/Kelly.

B. Recommend immediate approval and funding of the Finnigan Model 3000/150 Gas Chromatograph-Mass Spectroscopy system currently in this Laboratory's FY 73 procurement appropriation budget.

C. Recommend this Laboratory be requested to provide the information described in para 8 D (3) above by June 1, 1972.

D. The MSU team be requested to participate as consultants to the AF in checking the performance of the contractors incineration operation and monitoring system.



RICHARD A. CALLAHAN, Ph.D.  
Capt, USAF, BSC  
Chief, Biological Env Studies Branch

2 Atchs  
1. Cy Monitoring Program  
2. ARS ltr dtd 26 Nov

**ADDENDUM**

**Progress Report No. 3**

**Thermal Decomposition of Orange Herbicides**

**Under**

**The Amendment No. 2 to the USDA**

**Cooperative Agreement No. 12-14-100-10, 673(34)**

**Period Covered: March 1 to March 31, 1971**

**Investigators: B. J. Stojanovic, Dept of Agronomy-Soils  
M. V. Kennedy, Dept of Biochemistry**

**FOR OFFICIAL USE ONLY**

**PAGES WITHDRAWN**

CC

31 May 1972

**Trip Report - Marquardt Company, Van Nuys, California**

Commander, USAF Env Health Lab/CC, Kelly AFB TX 78241  
AFLC/SGPE, Wright-Patterson AFB OH 45433

**IN TURN**

1. **Place:** Marquardt Company, Van Nuys, California.
2. **Inclusive Dates of Travel:** 23 - 24 May 1972.
3. **Person Making Trip:** Captain Richard A. Callahan.
4. **Primary Mode of Transportation:** Commercial Air.
5. **Purpose of Trip:** The Air Force Rocket Propulsion Laboratory is funding an investigation concerning the pyrolysis of Orange herbicide in a SUR (Sudden Expansion) burner. The purpose of the study is to; study the pyrolysis products of Orange herbicide; and to study the incineration efficiency of the SUR incinerator. The meeting of 24 May reviewed the statement of work (Atch 1) concerning this contract, especially the sampling procedures and schedules. The contracting companies are:

Marquardt Company, Van Nuys, California - Incineration  
West Coast Technical Service, Inc., San Gabriel, CA - Chemical Analysis

**6. Persons Contacted:**

USAF, Rocket Propulsion Laboratory  
Dr. Richard Weiss  
Mr. John Nakamura  
Mr. Berge B. Goshgarian  
Capt. Joe Martone

Marquardt Company  
Mr. R. P. Babbitt, P.E.  
Mr. J. L. Clure  
Dr. Larry Marantz

West Coast Technical Services  
Dr. H. Dwight Fisher

## 7. Findings and Observations:

a. Several changes were made in the sampling protocol of the statement of work. These included adding analysis for the phenoxy herbicide acids, and changes in the procedure to validate the sampling techniques.

b. The SUE burner is claimed by the manufacturer to be more efficient than conventional burners. This high efficiency is thought to be caused by burning the waste in "radical rich" eddies created by the expansion "step" designed into the burner. Dwell time in the burner is only about 0.1 seconds versus 2-3 seconds in other burners. Temperatures can reach 3000°F.

c. This study should generally determine if it is feasible to burn Orange in SUE burners.

d. The study will have limited applicability concerning the large "conventional" incinerators currently under consideration.

(1) As mentioned above the combustion process in the SUE is different from conventional burners.

(2) Gas samples will be taken in bottles and analysed by a subcontractor (West Coast Technical Services) several hours later. This process has several disadvantages.

(a) Storage of gas and condensed chemicals in an acidic atmosphere will result in further degradation of many pyrolysates that may not have occurred during the scrubbing process or after discharge to the environment.

(b) Quantitatively removing chlorinated organic compounds from metal surfaces is an extremely difficult task. This will be especially true with TCOD. This problem will be further exacerbated by the inaccessibility of the inside of a gas sampling bottle.

(c) The Perkin Elmer RMU 6D Mass Spectrometer used by the contractor will not be able to analyse in the ng concentrations necessary to qualitate the majority of pyrolysates.

e. SUE burners may prove to be ideal for installation on Johnson Island to incinerate Orange. SUE burners are much smaller than conventional burners of similar capacity; and therefore cheap to construct and relatively easy to move.

### SIGNED

RICHARD A. CALLAHAN, Ph.D.  
Capt, USAF, BSC  
Chief, Biological Env Studies Branch

Copy to:  
USAF Env Health Lab/GC  
McClellan AFB CA 95652

AFRPL/Dr. Richard Weiss  
Edwards CA 93523

APPENDIX F

AUTHORIZATION AND DEFINITION OF SUPPORT

	<u>Page</u>
F-1 AFLC/SG Letter Defining Limits at EHL/K Support to SAAMA/SF-----	95
F-2 Environmental Health Laboratory/Kelly Letter Defining Support to SAAMA/SF-----	96



DEPARTMENT OF THE AIR FORCE  
HEADQUARTERS AIR FORCE LOGISTICS COMMAND  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433



26 Oct 1971

REPLY TO  
ATTN OF SG

SUBJECT: Request for USAF Environmental Laboratory Support  
(Your ltr, 13 Oct 71)

TO: SAAMA/SF

1. The support requested in subject letter is not considered to be appropriate to the USAF Environmental Health Laboratory, Kelly AFB. Among the reasons for this decision are:

a. The mission of the environmental health laboratories is to support Air Force operation and missions. The resources of the laboratory are not sufficient to support what appears to be a civilian contractor operation and to maintain timely support of Air Force operations.

b. The environmental impact of commercial incinerations should be an important consideration in the award of any contract. It would appear that any Statement of Work and Procurement Contract should include requirements for appropriate surveillance of the incinerator operation.

2. The services of the Environmental Health Laboratories at Kelly and McClellan AF Bases can be made available for assistance in preparation and/or evaluation of an environmental impact statement if required and of the technical suitability and accuracy of a Statement of Work and Procurement Contract. The laboratories could provide some assistance in the development or evaluation of sampling techniques and analysis procedures.

FOR THE COMMANDER

S. E. LIFTON  
Colonel, USAF, MC  
Surgeon

7/10/29

ACTION TO:	✓ 10
CODE NO:	5-1
DISPOSITION:	
APPROVED BY:	St/
AUTHORITY:	



CC

15 December 1971

Herbicide Incineration at a Commercial Facility (EHL/K Spec Proj 71-50)

SAAMA/SF

1. The following investigations are being pursued at this laboratory in order to provide methods for sampling and analyzing emissions and effluent discharges resulting from the incineration of Orange herbicide. These investigations will be limited to those authorized by AFLC/SG ltr of 26 Oct 71 (Atch 1).

2. This investigation has three objectives:

a. Determine the practical limits of detection for 2,3,7,8 tetrachloro-dibenzo-P-dioxin (TCDD) and the butyl esters of 2,4-D and 2,4,5T in stack emissions and wastewater effluents.

b. Determine if incomplete pyrolysis of Orange herbicide generates substances which interfere with the gas liquid chromatography (GLC) of TCDD thereby invalidating this method of analysis for purposes of monitoring stack and water discharges.

c. Determine practical, efficient procedures for extracting TCDD and active herbicide residues from stack gases and water samples. These methods will provide samples suitable for GLC analysis.

3. The following progress has been made to accomplish these goals:

a. The industrial hygiene problems associated with TCDD have been reviewed and necessary steps taken to insure its safe handling in this laboratory.

b. Appropriate GLC columns have been made and conditioned. The instrumental limits of detection of TCDD in this laboratory are less than 20 picograms ( $20 \times 10^{-12}$  grams). Actual analysis of stack or water samples will, of course, be much less sensitive than 20 picograms. These results indicate that, as anticipated, very small amounts of TCDD can be detected in discharges assuming no interfering substances are present. The reagent grade TCDD supplied appears pure in GLC analysis.

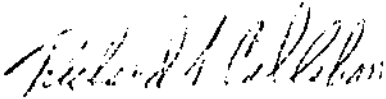
c. A continuous, controlled feed, all glass burner has been designed and fabricated. The burner is currently being tested burning methanol. Techniques for empirically measuring the average gas retention time under various controlled operating conditions are being developed.

d. All glass scrubbing and sampling systems have been fabricated and are ready for testing.

e. Feed rate parameters such as the fuel to air ratios and theoretical emission gas volumes have been developed by Maj Suggs at the McClellan EHL. Maj Suggs is currently developing the theoretical ground concentrations of emission products under various emission concentrations and assuming the worst possible weather conditions from stacks sixty and 100 feet high.

4. The attached Milestone and Progress Chart (Atch 2) quantitates these actions to date in relation to the final mission goals.

FOR THE COMMANDER



RICHARD A. CALLAHAN, PhD,  
Captain, USAF, BSC  
Chief, Biological Env Studies Br

2 Atch

1. Ltr AFLC/SG 26Oct71
2. Milestone Chart

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