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Environmental Monitoring and Support Laboratory's
Responsibilities for Sampling and Analysis for Dioxin
in Herbicide Orange

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October 29, 1975

I. INTRODUCTION

In late September, 1975, the Environmental Monitoring and Support Laboratory (EMSL) was requested by ORM of EPA to assist in the surveillance for Dioxin (chemical name: 2,3,7,8 Tetrachlorodibenzo-p-dioxin; also abbreviated as TCDD) from the Air Force pilot plant disposal program for Herbicide Orange. This pilot plant program is scheduled for a 6-week period beginning with the week of 11/3/75.

The principal functions of EMSL are:

1. to procure, transport to the site, and set-up the sampling equipment,
2. to provide the necessary technical direction to an EPA-selected contractor who will perform "fenceline" ambient monitoring and subsequent analysis, and
3. to design and implement a Quality Assurance (QA) program for both the sampling and analysis procedures.

The purpose of this document (protocol) is to delineate the involvement and responsibilities of EMSL in this project.

II. BACKGROUND

Through a request from the U. S. Air Force, the EPA was asked to assist in monitoring a pilot plant disposal/reclamation project for removal of TCDD from the herbicide designated as Orange. The pilot plant was to be assembled on a 40x50 foot pad on the grounds of the Naval Construction Battalion Center at Gulfport, Mississippi. This site was selected by the USAF because it is presently a storage location for a large quantity of the Orange. The actual operation of the pilot plant is to begin around 11/3/75 for a projected 6-week period to process 30 barrels (1650 gallons) of Orange. The composition of the herbicide (diluted for the pilot plant operation) is 90% acetone and 10% Orange with a TCDD concentration of 2 parts per million (by weight) in Orange.

Because of the extreme toxicity of the TCDD in the herbicide, the USAF had prepared a report entitled "Disposition of Orange Herbicide by Incineration", dated 11/74. This document (775 pages) describes in detail the project history and projected environmental impact and contains an extensive appendix including sampling (source and ambient) methodology. The pilot plant was built and will be operated by Agency Chemical, Inc. (Houston, Texas). The principle emission point of the pilot plant is a 2-inch exhaust stack on the outlet of the caustic scrubber. The height of the stack above ground level is not known exactly at this writing but is at least 10 feet and probably no more than 20 feet. The exhaust gas flowrate is 2 CFM. There are also at least 7 process vents with charcoal scrubbers at various points in the

pilot plant, which could add to the general area emissions. The storage and transfer of the herbicide could possibly be an additional source.

Ambient (actually "in-plant") and source monitoring of the operation will be conducted by a team from the USAF Environmental Health Laboratory at Kelly AFB in San Antonio, Texas. Source samples will be collected from the 2-inch stack. The "in-plant" samples will be collected at the center of the pad and at a 25-foot distance on all four sides. The sampling train utilized is identical for source and ambient except that an additional benzene bubbler and silica gel scrubber in series is used in the source train. Since the EPA's responsibility is "fenceline" monitoring, EMSL will define the "fenceline" location and instruct the contract accordingly.

III. SAMPLING

A. Site Selection

In order to select the sampling location, the available meteorology information for Gulfport was obtained by Mr. Fankhauser from the USAF. Utilizing the available source emission information and meteorology and consulting with Mr. D. B. Turner (EPA/ERSL) it was determined that sampling at 50 meters (164 ft) would be the optimum distance. Since the Naval Center boundary is too far away to be considered a realistic "fenceline", this 50-meter distance was chosen to represent the highest concentration under expected meteorological conditions. The USAF sampling locations are considered "in-plant" as compared to the EPA "fenceline" locations. The number of fixed sampling sites at this distance from the center of pad was selected as 8 (numbered 1 thru 8), spaced radially at 45° intervals. (See Figure 1) This provides a spacing of 125.5 feet between sampling sites around the pad. Additionally, one mobile site (Number 9), probably on a pick-up truck, would be placed downwind at the 50 meter distance based on the prevailing wind direction at the start of a test. As of this writing no on-site visits have been made by EPA personnel because of time and security constraints, and only verbal descriptions of the site area have been obtained. In practice the actual site selections may be altered slightly because of physical obstructions.

B. Apparatus

Based on the DRAFT sampling methodology provided by the USAF and subsequent verbal information from Major Gokelman and Captain Normington, the components of the sampling train were obtained (requisitioned) by EMSL. Because of the importance of individual samples, it was decided to operate parallel trains (designated A and B) at each sampling location. The EPA analysis contractor will analyze samples from Train A and archive samples from Train B for later analyses. This gave a total of 18 trains (16 fixed, 2 mobile). A schematic diagram of the train is

given in Figure 2. The USAF plans to operate only 2 benzene bubblers in series for ambient sampling, however, some carryover from the first to second impinger has been measured in earlier USAF tests. It was decided by EMSL to add the third bubbler as a safety precaution. Since the bubblers are normally poured together for subsequent analyses, numbers 1 and 2 will be poured together and number 3 will not be analyzed unless measurable amounts are found in 1 and 2. EMSL, utilizing on-hand equipment and emergency purchase orders, should have all sampling equipment ready to ship by Thursday, P.M., 10/30/75. It is intended that EMSL will provide an EPA van and driver to leave on the following morning to drive the equipment to Gulfport for use by the Contractor.

C. Operation

On-site operation of the samplers is to be performed by the EPA Contractor (Gulf South Research Institute, New Orleans, La.). An EMSL representative will be available in Gulfport to set up the equipment and train the Contractor in operation and data collection. The EMSL representative will not be required to go on-site during pilot plant operation. As a safety precaution all EMSL personnel going to Gulfport will have the extensive physicals described in Figure 3 before and after the sampling tests because of possible contact with collected samples or contaminated equipment. Detailed written operation instruction and data collection forms are being prepared and will be provided to the Contractor. In general, the sampling trains will operate simultaneously for the duration of the daily pilot plant operation. The pilot plant is expected to operate from 5-7 hours per day, for 3 days per week. All pilot plant operations are intended for fair weather daytime periods only. One integrated sampler run (including duplicate in parallel) will be made per day per site. The parallel train samples will be archived by the Contractor in amber bottles for subsequent split sample analyses if detectable amounts are measured in the Contractor samples. The analytical laboratory for the split sample analyses has not been selected.

All sampling will include quality control checks - leak and flow tests - as prescribed by the sampling procedure to be prepared by EMSL and supplied to the Contractor.

D. Analyses

The analyses will be performed by the EPA Contractor. EMSL's responsibility will be only to provide a QA program for the analytical procedures, primarily to determine the precision (repeatability) of the analytical results. The description of this QA plan is also attached to this document.

IV. QUALITY ASSURANCE

1. Sampling

a. Sampling train - Prior to sampling the entire sampling train must be checked for leaks. This may be performed by closing off

all flows thru the inlet of the bubbler and observing the occurrence of bubbles in the train. If bubbles occur, necessary action should be taken to eliminate the leak.

b. Flow meter - All field flowmeter devices must be checked to assure proper flow by the Contractor. These devices must be checked at least every two weeks by a secondary calibrated standard mass flow meter. This calibrated mass flow meter will be furnished by EPA.

c. Data sheets - The contractor will complete all information on the EPA supplied data sheets (see Figure 4). One data sheet will be completed for each train for each run.

d. Sampling flow rate - During the sampling period the Contractor must assure proper flow rates (1.0 liter/min.). At least three flow checks must be taken and recorded by the Contractor during each sampling period.

2. Analysis

a. Static Reagent Blanks - A representative sample of the pesticide grade benzene must be tested by the Contractor for background on a scheduled basis and the results must be sent to EPA.

b. Dynamic Reagent Blanks - Prior to each sampling period a dynamic (system blank) reagent blank must be performed by the Contractor. The results must be sent to EPA.

c. Blind Samples - EPA prepared blind samples will be furnished by EPA to the Contractor on a random schedule. These samples must be considered as field samples and results reported to EPA. Total number of reference samples will not exceed 40.

d. Field Samples - All field samples must be analyzed by the Contractor. The Contractor is responsible for all analytical calibration curves and reporting field results that are statistically valid. A bound notebook for sample data logging must be maintained. Copies of all sampling data sheets must be sent to EPA.

V. EQUIPMENT AND MANPOWER

1. Equipment

a. Government owned equipment (supplied to Contractor)

- (1) 60 - fritted Greenburg-Smith bubblers
- (2) 40 - Greenburg-Smith traps
- (3) 18 - shelters and support equipment
- (4) 2 - meteorology stations

- (5) 9 - portable generators
- (6) 50 - amber glass storage bottles
- (7) 425 lbs. - 18-30 mesh charcoal
- (8) 40 - quality control samples
- (9) glass wool
- (10) miscellaneous glassware, clamps, and fittings

b. Equipment furnished by Contractor

- (1) 40 gals of pesticide grade benzene
- (2) Shipping containers for transferring of samples for

analysis.

2. Manpower (estimated)

a. Government (EMSL/RTP)

- | | |
|---------------------------------------|----------------------|
| (1) Planning and meeting attendance | 0.5 manmonths |
| (2) Preparation of equipment | 4.5 manmonths |
| (3) On-site training and consultation | 1.5 manmonths |
| (4) Project officer | 2.5 manmonths |
| (5) Refurbishing of equipment | <u>1.5 manmonths</u> |

Total	10.5 manmonths
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b. Contractor

- (1) Operation of samplers and met. equipment
- (2) Sample handling and transporting
- (3) Analysis of samples
- (4) Analysis of QC samples
- (5) Final report - all sampling data

Figure 1. EPA Sampling Locations
TCDD Study

Mobile Site
(position
variable)

9

8

7

6

1

50m

2

3

4

5

EPA Sampling sites

USAF Sampling sites

Scale: 50 feet

10/29/25

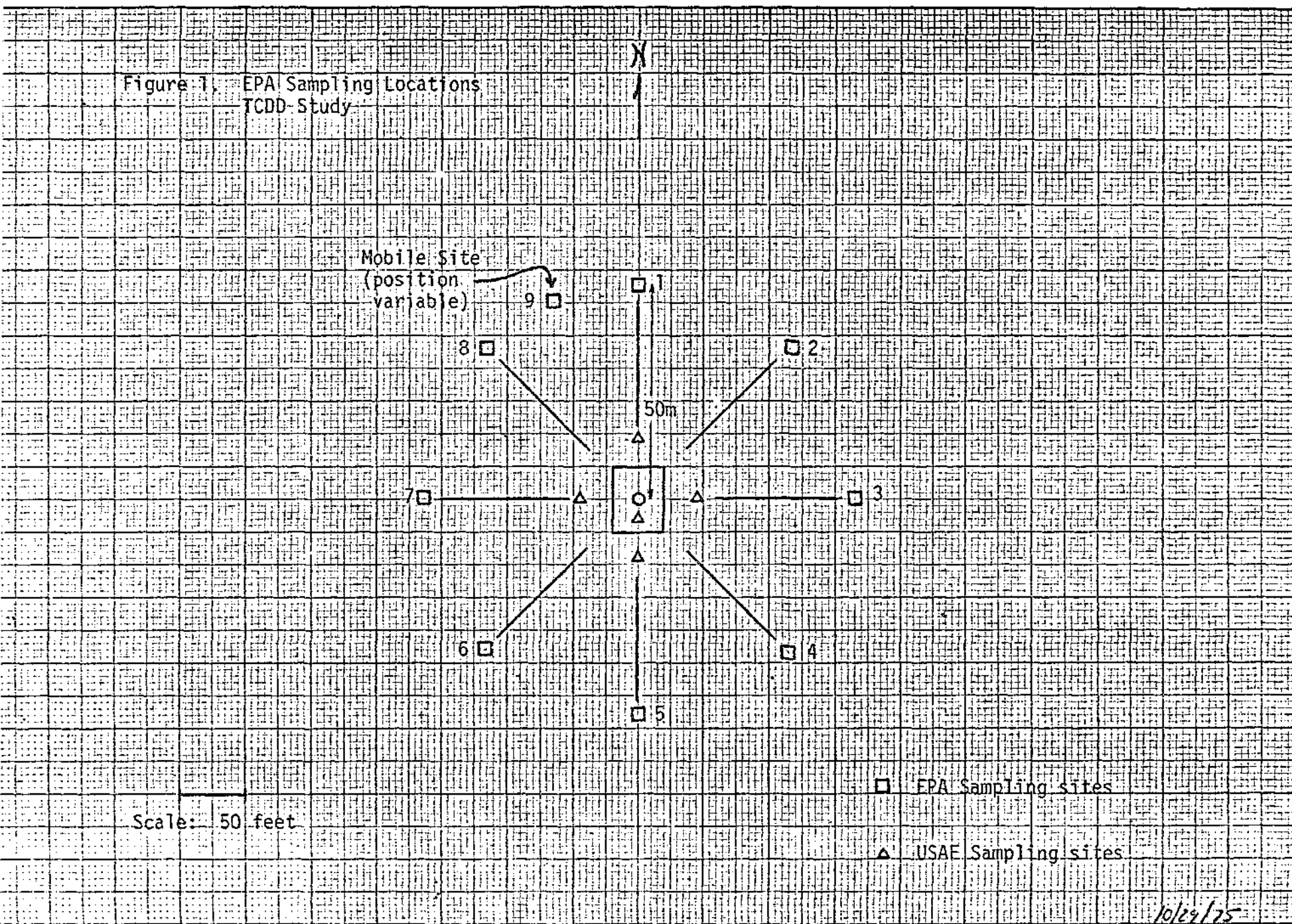
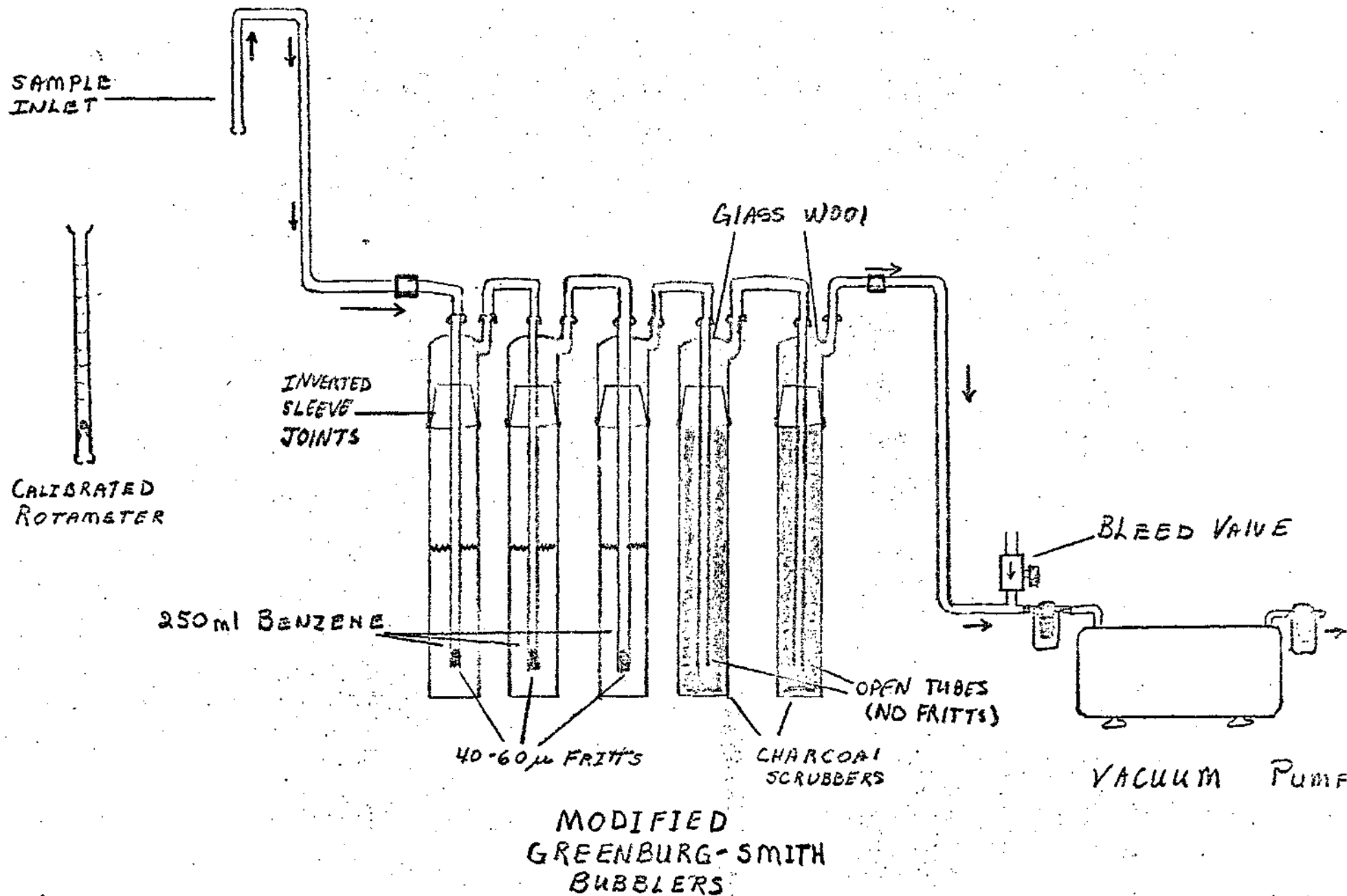


Figure 2. EPA Sampling Train for TCDD



10/29/75

10/1/65 } RTP { 549-2248
10/1/66 } { 549-2151

Figure 3. TCDD Medical Examination:

SUGGESTED U.S.A.F. MEDICAL EXAMINATION

1. Complete hemogram and hematocrit & platelet count
2. Prothrombin time
3. Serum lipids
4. Serum glutamic oxaloacetic transaminase (S-GOT)
5. Serum glutamic pyruvate transaminase (S-GPT)
6. Serum bilirubin
7. Blood glucose
8. Complete urinalysis
9. Chest X-ray

Physician to carefully examine skin and liver.

Dioxin manifests symptoms for moderate exposure in chlor-acne after several weeks or months. More heavy exposure results in acute liver damage and ultimate death.

For this exercise, the permissible ambient air concentrations are less than the analytical capability for detection. Stack emissions are not permitted in excess of 0.2 ng/M³. The detection limits using the best sampling techniques known are 0.1 to 0.2 ng/M³.

2,3,7,8-Tetrachlorodibenzo-p-dioxin is the most toxic chemical known to man. The OSHA and NIOSH have no official TLV. The known toxic concentrations for small rodents is 1-10 ng/animal. Human exposures must be below this limit but at this time there is no information available to provide guidance.

J. M. Schaeffer
416-2358

Herbicide Orange Pilot Plant Study

Data Sheet

DATE:

OPERATOR:

Start Time: _____ hr.

SITE NUMBER:

TRAIN NUMBER:

NOTE: This data sheet is for one Run/Train/Site only.

Volume of Benzene	Bubblers		
	1	2	3
Before Sampling			
After Sampling			

Elapsed Time	
End	
Start	
Elapsed	Minutes

Bubblers Flow rate, l/min.*	
Initial	
After 20 min.	
After 1 hr.	
After 3 hrs.	
Final	

Bubbler	Storage Bottle I. D. Number
1	
2	
3	

*Should be 1.0 l/min.

Figure 4. TCDD Sampling Data Sheet