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PILOT PLANT TESTING OF HOT GAS BUILDING DECONTAMINATION PROCESS

Task 1; Contract No. DAAA 15-86-D-0001

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Prepared for

U.S. Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground, Maryland 21010

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<p>In this project, Task 1 of the third phase of the Novel Process program, the hot gas decontamination process was used in a pilot scale study to determine its effectiveness in removing a controlled amount of chemical agent mustard (HD) from a small, multi-material test structure. The test structure and burner system were located in an environmentally controlled test chamber and were controlled remotely. Coupons of painted and unpainted concrete and steel were mounted on the inside of the test structure and two areas on the floor were spiked with low quantities (4mg/square inch) of HD to simulate conditions in a contaminated building. Pretest blanks and spike samples were collected and analyzed in accordance with methodology verified in the laboratory for extracting HD from pulverized concrete, from soil, and from painted and unpainted concrete and steel surfaces.</p>						
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The structure was closed for two days before burner startup during which time data on pressure, air flow and the HD concentration in the air of the test structure were collected. When the burner was started, the real time monitoring data of the air indicated that volatilization of HD and/or HD breakdown products occurred. This was confirmed by bubbler data. The volatilization reached a peak about 40 minutes after the burner was fired, then diminished to background level approximately 8 hours later.

The air temperature inside the test structure was raised in increments each hour to 750 F, until all points on the building had reached 300 F for one hour. It took 35 hours from the start of the test to reach this condition. Cooldown to less than 105 F took 38 hours, for a total testing time of 73 hours.

After cooldown, the test structure was opened and photographed, and all samples were collected and analyzed. No sample subjected to the hot gas process showed HD above the theoretical detection limits which indicates that the hot gas process is effective for removing HD from these materials.

The results of this study indicate that the hot gas decontamination concept is a promising technology for the decontamination of buildings and large pieces of equipment in the field. However, it is recommended that improved methods be developed to sample and analyze agent from the surface of painted and unpainted concrete. It is also recommended that air sampling techniques be used that are not affected by water emanating from the concrete during the process.

ACKNOWLEDGEMENTS

We would like to acknowledge the technical and administrative helpfulness of Mr. Andrew Roach, USATHAMA project officer throughout most of this task, Mr. Greg Mohrman, who succeeded him during the final stages, and Mr. Bob Bartell, Chief of Process Development Branch of USATHAMA. We also would like to thank Mr. David Gladden, Project Officer at Dugway Proving Ground, and his crew, for providing the necessary support to run the pilot test and analyze the samples on site. Furst Construction of Salt Lake City, Utah constructed the test structure, and Blair-Alexander Engineering, Inc. of Denver, Colorado, constructed and serviced the burner equipment.

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EXECUTIVE SUMMARY

The Army is the custodian of many buildings that have been contaminated during the manufacture, storage, and demilitarization of chemical warfare agents and munitions. Before the buildings can be reused, declared excess or torn down safely, they must be processed to remove residual contamination.

The Army has embarked on a multi-phase program entitled "Development of Novel Decontamination Techniques for Chemical Agent (HD, GB and VX) Contaminated Facilities". The goal of this program is to develop new technologies to decontaminate buildings and large equipment contaminated with chemical agents. In Phase I, "Identification and Evaluation of Concepts", 65 concepts were evaluated to address the problem. In Phase II, "Laboratory Evaluation of Novel Agent Decontamination Concepts", seven concepts from the Phase I study were tested for their ability to remove controlled amounts of chemical agents from samples of building materials to levels below detection limits. Based on effectiveness, reliability, waste product characteristics, possible hazards and approximate cost, three decontamination concepts (hot gas, steam, and 1-octylpyridium 4-aldoxime bromide, or OPAB) were selected for further engineering evaluation. Both the steam and hot gas processes penetrated porous materials such as concrete and appeared promising for building decontamination. The hot gas decontamination concept was found to be the most cost effective, and was recommended for further evaluation in a pilot test in the third phase of the program.

"Advanced Development and Field Testing of Novel Processes Technologies to Decontaminate Chemical Agent Contaminated Facilities" is Phase III of the Novel Processes program. Task 1 in Phase III is the first large-scale test of the hot gas process for effectiveness in decontaminating agent-contaminated building materials. The portion of the Task 1 test program conducted at Dugway Proving Ground (DPG) is the subject of this report which comprises two volumes. The results of the analytical and sampling methods verification studies the design of the test structure and the pilot test are reported in this volume. Volume 2 is an appendix that contains all of the data generated during the pilot test. The results of other activities completed as part of Task 1, including an analysis of regulations that may be applicable to the

field operation of the hot gas process, a preliminary economic analysis of the process supplied to larger structures, and the results of a field sampling survey at Rocky Mountain Arsenal (RMA) are detailed in separate letter reports.

In Task 1, the hot gas decontamination process was used in a pilot scale study to determine its effectiveness in removing a controlled amount of chemical agent mustard (HD) from a small test structure with walls made from poured concrete, solid and hollow concrete blocks, and from mild steel. Portions of each wall were painted and unpainted. The test structure and burner system were located in an environmentally controlled test chamber and were controlled remotely. Coupons of painted and unpainted concrete and steel were mounted on the inside of the test structure and two areas on the floor were spiked with low quantities (4mg/square inch) of HD to simulate conditions in a building decontaminated to meet 3x criteria (defined in DARCOMR 385-31 and DARCOMR 385-102). Pre-test blanks and spike samples were collected and analyzed in accordance with methodology developed in the laboratory for extracting HD from pulverized concrete, from soil, and from painted and unpainted concrete and steel surfaces.

The structure was closed for two days before burner startup during which time data on pressure, air flow and the HD concentration in the air in the test structure were collected. When the burner was started, the real time air monitoring data indicated that volatilization of HD and/or HD breakdown products occurred. This was confirmed by bubbler data. The volatilization reached a peak about 40 minutes after the burner was fired, then diminished to background level approximately 8 hours later.

The air temperature inside the test structure was raised in increments to 750°F until all points monitored on the building had reached 300°F for one hour attaining this condition required 35 hours. Cooldown to less than 105°F required 38 hours for a total test duration of 73 hours.

After cooldown, the test structure was opened and photographed, and all samples were collected and analyzed. Results of the analyses indicated that no HD above the detection limits were found in the samples of building

no HD above the detection limits were found in the samples of building materials subjected to the hot gas process.

The results of this study indicate that the hot gas decontamination concept is a promising technology for the decontamination of buildings and large pieces of equipment in the field. However, it is recommended that improved methods be developed to sample and analyze agent from the surface of painted and unpainted concrete. It is also recommended that air sampling techniques be used that are not affected by water emanating from the concrete during the process.

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TASK 1: CONTRACT NO. DAAA 15-86-D-001
DEVELOPMENT OF NOVEL DECONTAMINATION
TECHNIQUES FOR CHEMICAL AGENT (GB, VX, HD)
CONTAMINATED STRUCTURES, PHASE III

REPORT AMXTH-TE-CR-87130
PILOT PLANT TESTING OF HOT GAS BUILDING
DECONTAMINATION PROCESS

for

UNITED STATES ARMY TOXIC AND HAZARDOUS
MATERIALS AGENCY

Prepared By

BATTELLE COLUMBUS LABORATORIES

23 October 1987.

1.0 INTRODUCTION

The U.S. Army owns many facilities, which may include buildings or large items of equipment, that have been used in the manufacture, processing, loading, storage and destruction of chemical warfare agents. These operations involve permanent facilities and a variety of process and handling equipment. As part of their responsibilities in DoD property disposal, USATHAMA must identify, contain and eliminate toxic and hazardous materials at facilities that have been declared excess or are candidates for excessing. With this mandate, USATHAMA must provide not only the technical basis to implement decontamination but also the standards to ensure that decontamination has been effective.

The only currently approved method of decontaminating materials so that they can be released from government control is incineration at a temperature of 1000°F for 15 minutes. Materials exposed to such conditions are described as having attained "5X" status and are defined as suitable for unrestricted use (DARCOMR 385-102, 1982). However, the expense required to accomplish such

decontamination is excessive. The successful development of an alternative decontamination technique which would not require the dismantling of a facility and which would result in a 5X decontamination status rating (or its equivalent) without incineration represents a potentially large cost savings to the Government. Many facilities that have been contaminated with chemical agents are structurally sound and it would be most desirable to decontaminate such facilities by a process that would not damage structural integrity. Free from chemical agent contamination, the facility could be reused or replaced with substantial cost savings.

USATHAMA has instituted the multi-phase Novel Process Technologies program to investigate ways to decontaminate buildings and other structures in the field. Phase I and II were concept development and laboratory evaluation studies of methods for decontaminating building materials (see section 2.0). The hot gas decontamination concept was chosen as the most effective remover of chemical agent from a variety of materials as well as the most cost effective out of 65 concepts evaluated.

As part of Phase III, in which the novel process technology is to be adapted to the field, Battelle Columbus Laboratories was tasked by USATHAMA to perform an experimental large-scale pilot test using the hot gas process to remove controlled amounts of agent mustard (HD) from a small test structure with walls made of poured concrete, solid and hollow concrete block, and from mild steel. Surfaces were both painted and unpainted. A propane burner was attached to a port in the test structure, and heated air was blown into the test structure and exhausted through a charcoal filter. The test structure and burner system were located in an environmentally controlled test chamber and were controlled remotely. Coupons of painted and unpainted concrete and steel, mounted on the inside of the test structure, and two areas on the concrete floor were spiked with approximately 4mg/square inch of HD. Pre-test blanks and spike samples were collected and analyzed in accordance with methodology developed in the laboratory for extracting HD from pulverized concrete, from soil, and from painted and unpainted concrete and steel surfaces.

The results of the analytical and sampling methods verification studies and the pilot test itself are reported in this volume. Volume two is an appendix that contains all of the data generated during the pilot test at Dugway Proving Ground (DPG).

2.0 BACKGROUND

Phase I, "Identification and Evaluation of Concepts", was performed by Battelle Columbus Division (BCD)*. Sixty-five concepts were generated and described in sufficient detail to permit their evaluation against the criteria of mass transfer, destruction efficiency, safety, damage to structures, penetration depth, applicability to complex structures, operating costs, capital costs, and waste treatment costs (Table 2-1). Seven of the most promising concepts, which included Hot gases, Steam, N-Octyl-pyridinium 4-aldoxime bromide (OPAB), Monoethanolamine (MEA), FREON 113 Vapor Circulation, Ammonia Gas or Ammonia Steam, and Flashblast were recommended for further study in Phase II, "Laboratory Evaluation of Novel Agent Decontamination Concepts".

The laboratory study, also performed by BCD, was designed to evaluate experimentally the concepts selected from Phase I and to recommend one to three of the most promising concepts for field evaluation in Phase III**. The ideal concept sought was a single decontamination process that would effectively decontaminate building materials such as concrete and steel and remain cost effective. Laboratory evaluation included testing the effectiveness of the seven concepts in decontaminating progressively more complex substrates: first, from glassware, then from unpainted stainless steel coupons, and finally from painted and unpainted mild steel, painted stainless steel and concrete coupons.

*H. P. Benecke, et al., Development of novel decontamination and inerting techniques for explosives contaminated facilities, Phase I: Identification and evaluation of novel decontamination concepts, Battelle Columbus Division to U.S. Army Toxic and Hazardous Materials Agency, Report DRXTH-TE-CR-83211, July, 1983.

**E. R. Zamejc, et al., Development of novel decontamination and inerting techniques for chemical agents (GB, VX, HD) contaminated facilities, Phase II: Laboratory evaluation of novel agent decontamination concepts, Battelle Columbus Division to U.S. Army Toxic and Hazardous Materials Agency, Report AMXTH-TE-TR-85012, 21 June, 1985.

TABLE 2-1. AGENT DECONTAMINATION CONCEPTS EVALUATED DURING PHASE I
(from the Phase II Final Report)

<u>CHEMICAL</u>	<u>PHYSICAL/EXTRACTION</u>
OCTYL PYRIDINIUM 4-ALDOXIME BROMIDE (OPAB)	SURFACTANTS
DS2 (A)	STRIPPABLE COATING
CD-1 (B)	VAPOR CIRCULATION
SUPERTROPICAL BLEACH (STB)	SOLVENT CIRCULATION
ALL PURPOSE DECONTAMINANT (APD) (C)	SUPERCRITICAL FLUIDS
MONOETHANOLAMINE	ULTRASONIC EXTRACTION
GAMMA RADIATION	
NITRIC ACID	
AMMONIUM HYDROXIDE	<u>PHYSICAL/ABRASIVE</u>
HYPOCHLORITES	HYDROBLASTING
DANC	ACID ETCH
GASEOUS AMINES	SANDBLASTING
CHLORINE	DEMOLITION
STEAM	VACU-BLASTING
AMMONIA/STEAM	CRYOGENICS
PERCHLORYL FLUORIDE	SCARIFICATION
GERMAN EMULSION	ELECTROPOLISHING
HYDROXAMIC ACIDS	DRILL AND SPALL
SODIUM HYDROXIDE SOLUTION	
DIMETHYLSULFOXIDE	<u>THERMAL</u>
MACROCYCLIC ETHERS	FLASHBLASTING
PROPIONYL FLUORIDE	HOT PLASMA
PHENOLS/CATECHOLS	MICROWAVE HEATING
CARBONATE/BICARBONATE SOLUTIONS	FLAMING
CHLORITE SOLUTIONS	HOT GASES
CHLORINE DIOXIDE	SOLVENT SOAK/BURN
NITROGEN TETROXIDE	INFRARED HEATING
BORON TRIFLUORIDE	CARBON DIOXIDE LASER
OZONE	ELECTRICAL RESISTANCE CONTACT HEATING
SULFUR DICHLORIDE	
UV/OZONE	
ULTRASONIC DECOMPOSITION	
COPPER LIGANDS	
VANADIUM CATALYZED HYDROLYSIS	
ANTHRANILIC ACID-SILVER COMPLEXATION	
MAGNESIUM HYDROXIDE IMPREGNATED ALUMINA	
COMPLEXATION WITH MOLYBDENUM LIGAND	
PERBORATES	
MICROBIAL DEGRADATION	
PERMANGANATE SOLUTIONS	
ENZYME PROTEINS	
SODIUM SULFIDE	

- A) Mixture of 70 weight percent diethylenetriamine, 28 weight percent methyl cellosolve and 2 weight percent sodium hydroxide.
- B) Mixture of 55 volume percent monoethanolamine, 45 volume percent propylene glycol and 2.5 weight percent lithium hydroxide.
- C) Mixture of 54 weight percent monoethanolamine, 44 weight percent isopropanolamine and 2.5 weight percent lithium hydroxide.

The hot gas concept, in which a temperature of 300°F was maintained for 60 minutes, was found most effective in decontaminating HD, GB, and VX contaminated painted and unpainted steels and porous materials. In the hot gas process, the interior of a building is heated by hot exhaust gases from a combustion device located outside the building. As the temperature is increased, the chemical agents and their decomposition products are volatilized from the structure and the exhaust gases are filtered or passed through an afterburner.

The steam decontamination concept and the OPAB decontamination concept were also effective, but the engineering analysis indicated OPAB would be less effective than either steam or hot gas. An economic analysis of the recommended concepts indicated that the hot gas concept would be more economical to use than the steam concept. Based on these results, Battelle and USATHAMA selected the hot gas decontamination concept for field evaluation in this first task of Phase III, "Field Testing".

3.0 OBJECTIVES

The main objective of the hot gas decontamination pilot program was to further test the feasibility of the hot gas process before attempting full-scale implementation. The objectives of Task 1, Phase III are as follows:

1. Demonstrate the feasibility of the hot gas process in pilot-scale decontamination tests of an actual contaminated structure, room, or sump.*
2. Verify and/or develop analytical and sampling techniques for agents on or in field building materials, soils, water, etc.
3. Determine visually the effects of the hot gas process on actual structures and representative materials.
4. Identify contamination levels and profiles in field structures (results to be reported in a separate letter report, December, 1987).
5. Obtain data for use in satisfying regulatory/safety agency requirements for application and validation of a decontamination process (results to be reported in a separate letter report, December, 1987).
6. Confirm/refine the assumptions made in previous engineering and economic evaluations such that a detailed design of a full-scale process can be made (results to be reported in a separate letter report, January, 1988).

*USATHAMA originally envisioned a pilot test on a structure in the field at an installation. However, after a site selection review by USATHAMA was conducted, it was decided that a pilot test conducted on a specially constructed test structure spiked with chemical agent in a controlled environment would be more appropriate for the first large scale pilot testing of this process involving chemical agents.

4.0 TECHNICAL APPROACH

Task 1 included the development of sampling and analytical methodologies for field contaminated facilities, limited sampling of existing field facilities, and the design and operation of a pilot-scale hot gas decontamination system.

Mustard (HD) was the chemical agent of interest in the Task 1 program. This agent was selected because of indications in previous laboratory work (Phase II) that HD was the most readily recoverable from concrete of the common chemical agents. This made HD most suited to the objectives of the task. The methods development, field survey and pilot test were all designed with HD as their focus.

4.1 Subtask Review

The work for this task was organized into the following nine subtasks, each comprised of several activities:

- Subtask 1 -- Task Management
- Subtask 2 -- Phase II Design Review
- Subtask 3 -- Analytical/Sampling Methodology
- Subtask 4 -- Field Survey
- Subtask 5 -- Pilot System Design and Fabrication
- Subtask 6 -- Test Plan
- Subtask 7 -- Procedures and Documentation
- Subtask 8 -- Pilot Test
- Subtask 9 -- Draft Report

In Subtask 2, Design Review, knowledge gaps in the Phase II report were identified, pilot test parameters were identified (see section 4.2) and preliminary outlines of the test structure and pilot test design were made. The Subtask 2 report was submitted to USATHANA in May, 1986, and is included in Appendix A.

In Subtask 3, Analytical/Sampling Methodology, field sampling methods were developed for retrieving HD from steel and concrete surfaces and from concrete at depth. Analytical methods were verified for extracting HD from pulverized concrete, soils, and swab samples collected from painted and unpainted steel and concrete surfaces. Section 8.4.1 contains a brief summary of the results of those studies as applied to the pilot test operation. A more detailed account of the methods verification and sampling method development is given in the Subtask 3 report which was submitted to USATHAMA in August, 1986 and is included in Appendix B.

The objective of Subtask 4, Field Survey, was to determine levels of contamination of HD in actual buildings that could be candidates for the hot gas decontamination process. A field sampling of a hot gas stream from a potentially contaminated concrete pit in Building 537 of the Rocky Mountain Arsenal, Colorado (RMA), was conducted in May, 1987, as part of this subtask. Results of this operation are summarized in a separate letter report (Scheduled November, 1987).

Subtask 5, Pilot System Design and Fabrication, included initial design of the system, drawings of the system setup (submitted to USATHAMA in August, 1986), fabrication of the test structure, and instrumentation setup and checkout (see sections 5 and 6 of this report).

Subtask 6, Test Plan, was the operational guide for the pilot test. It is provided in this report as Appendix C.

Subtask 8 was the pilot test operation itself, which is detailed in section 7 of this report. Results of the pilot test are described and discussed in section 8.

The objective of Subtask 7, Procedures and Documentation, was to gather and analyze federal, state, and Army regulations that would impact the application of the hot gas decontamination process to the field. Results of

this analysis of safety and environmental regulations are reported in a separate letter report (Scheduled December, 1987).

Subtask 9, Draft Report, included a summary of all preparation and results of the pilot test. A preliminary economic analysis of the application of the hot gas process to field structures was also performed as part of this subtask. This analysis is reported separately in a letter report (Scheduled January, 1988).

4.2 Pilot Test Parameter Selection

The original task order called for the pilot test to be performed on an existing field structure at an installation, but after a site review by USATHAMA, it was determined that an intermediate step--changing from a field test on an existing structure to a scaled-up laboratory test--would provide more control of the environment and test parameters. Thus, the approach was changed. The pilot test was subsequently performed on a small fabricated test structure comprised of a variety of building materials spiked with HD and decontaminated in an environmental test chamber. Pretest spiked samples and post test samples of the building materials were analyzed and compared, and various air monitoring data were gathered during the test by bubblers and Automatic Continuous Air Monitoring Systems (ACAMS). A test chamber in Building 3008 at OPG was selected for the site of the pilot test.

Because the pilot test was to be designed to simulate decontamination of structures which may be encountered in the field, it was decided that the concentration of the HD spikes should approximate those found in buildings in a "3x" condition. This condition is defined by the regulations in terms of vapor concentrations in air, not as concentrations in materials (see DARCONR 385-31 for HD, and DARCONR 385-102 for GS and VX). Thus, "3x" decontamination has been achieved when a chemical decontaminant has been applied to a surface and less than 0.003 mg/m^3 of HD is present in an 8-hr bubbler analysis of the offgassed air of the structure (DARCONR 385-31). The concentration of HD in bubbler solution that correlates to the 0.003 mg/m^3 definition is

approximately 0.1 ug/ml, which is essentially the detection limit of the analytical method used to analyze the bubbler samples.

In an effort to translate the regulatory criteria to concentration of HD in building material, the theoretical method detection limits developed in Subtask 3 were used as minimum criteria for spiking values (see section 8.5). Army safety and surety regulations and policies dictated the upper limit of the concentration levels of the spiking. A concentration of approximately 4 mg/sq.in. was used to spike the test structure coupons and the floor spills.

The experimental design parameters of the pilot test were selected after review of the Phase II report was conducted and a heat transfer analysis was made (Appendix A). The following design parameters were selected:

1. The gas temperature inside the test structure must not exceed 750 F based on the Phase II conclusion that concrete is not seriously damaged when heated to temperatures below 750 F.
2. The temperature of structural materials must be maintained at 300 F for at least 60 minutes to assure the decontamination of HD (from results of studies in Phase II).
3. The test structure must be mounted on a base of soil which would be sampled after the test. This requirement was established to determine if HD in concrete, when submitted to a unidirectional heat source, maintains its chemical integrity and migrates away from that source into the surrounding soil.

The following sections detail the pilot test design, operation, and results.

5.0 PILOT TEST EQUIPMENT AND FACILITIES

5.1 Test Structure Design

The pilot test was conducted in Room 212V, an environmental test chamber in Building 3008 at Dugway Proving Grounds (DPG), July 17-23, 1987. The test was a joint effort between Battelle, which designed and provided technical oversight for the test, and Dugway Proving Grounds, which actually performed the test operations and provided laboratory services. Duties of each organization are detailed in the Test Plan (see Appendix C).

This section describes the test structure design, coupon design, equipment layout, the burner design and control, the data acquisition system and the analytical services.

The test structure was a 10 x 8 x 8 foot building with a steel door opening onto an 8 x 8 foot platform as sketched in Figure 5-1. Each wall of the building was made of a different material: one of 12-inch thick concrete, one of standard 16 x 8 inch by 8-inch thick solid concrete block, one of 16 x 8 x 8 inch hollow concrete block, and one of 0.25 inch steel plate. One-half of the building floor was 12-inch thick concrete and the other half was 6-inch thick concrete. The platform floor and building ceiling were 0.25 inch steel plate. The entire structure was mounted on a one foot high, 12 x 8 foot base made of I-beams, which was filled with local soil from DPG. (Local soil, which has some clay in it (see Appendix E), was used instead of the sand called for in the design review (Appendix A) because its heterogeneous composition was judged to more accurately approximate actual field conditions). The base was supported 13 inches from the floor of the test chamber by four casters. Therefore, the top of the soil base and the test structure floor were 25 inches above the test chamber floor. The steel door frame and penetrations for heating, cooling, and room exhaust ducts were located in the steel wall of the test structure. The entire structure was insulated with high temperature fiberglass insulation (2 inch thick Owens Corning Insul-Quick).

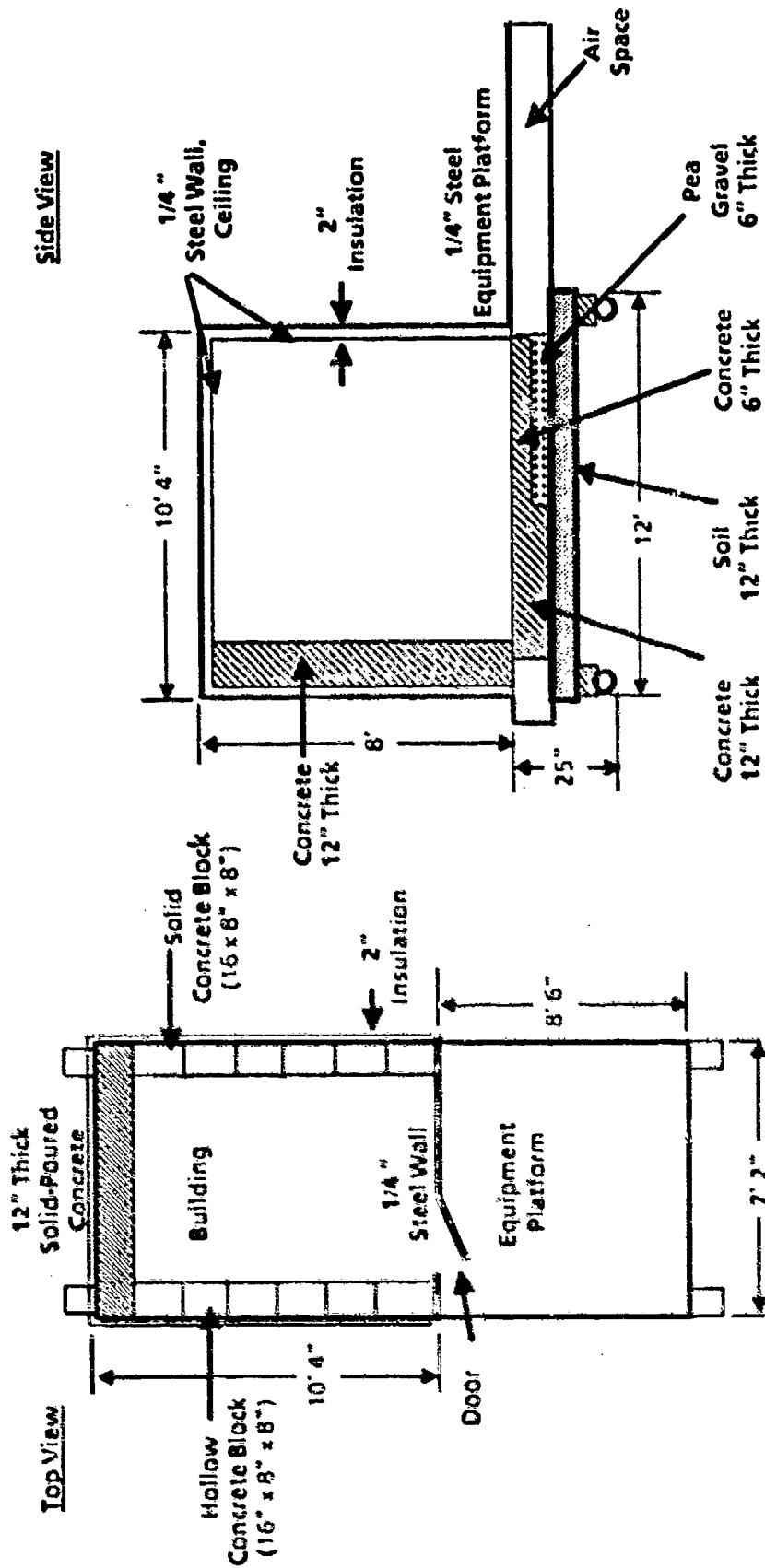


FIGURE S-1. SKETCH OF TEST STRUCTURE

5.2 Coupon Design

Both concrete and carbon steel coupons were placed in the test structure. The steel coupons were installed on the steel wall; concrete coupons were installed on the test structure's floor and concrete walls. The concrete coupons measured 5x5 inches, and were fabricated in two thicknesses: 0.25 and 0.5 inches. Both low porosity and high porosity concrete coupons were prepared in order to simulate more closely the variety of concrete which can be found in the field. The cement to sand ratio for the low porosity concrete was 0.4:1:1.6 and for the higher porosity concrete was 0.6:1:3. The steel coupons also measured 5x5 inches, and were made of 0.25 inch carbon steel.

The mounting clips used for holding the coupons against the test structure walls consisted of the following components:

1. Tool Holder, No. 68 Finger Grip (cut in half),
2. Machine Screw Anchor, Tampin, 10-24 (rated at 150 lbs safe load),
3. Steel Bushing, 5/16 inch OD x 3/16 inch ID x 17/32 inch long, and
4. Steel Machine Screw, RH 10-24 x 1 inch.

Figure 5-2 is a sketch of the assembled clip.

The equipment floor plan is shown in Figures 5-3 and 5-4.

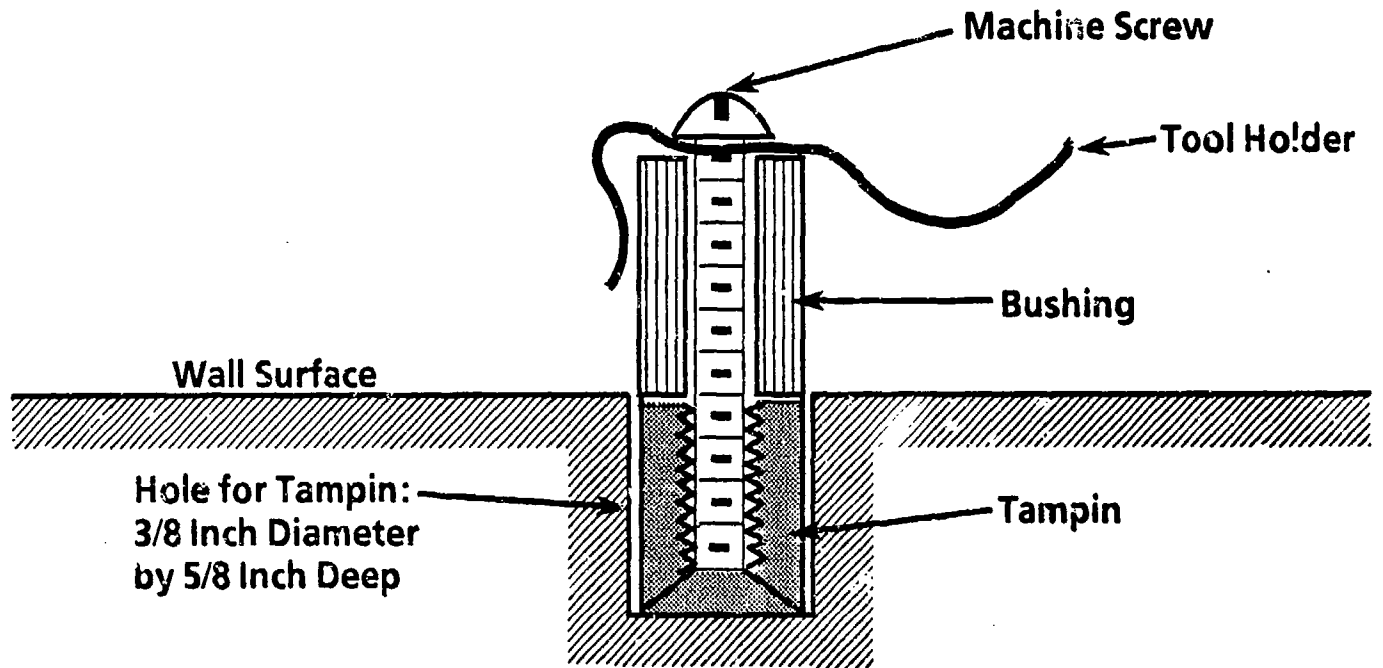


FIGURE 5-2. SKETCH OF COUPON MOUNTING CLIP

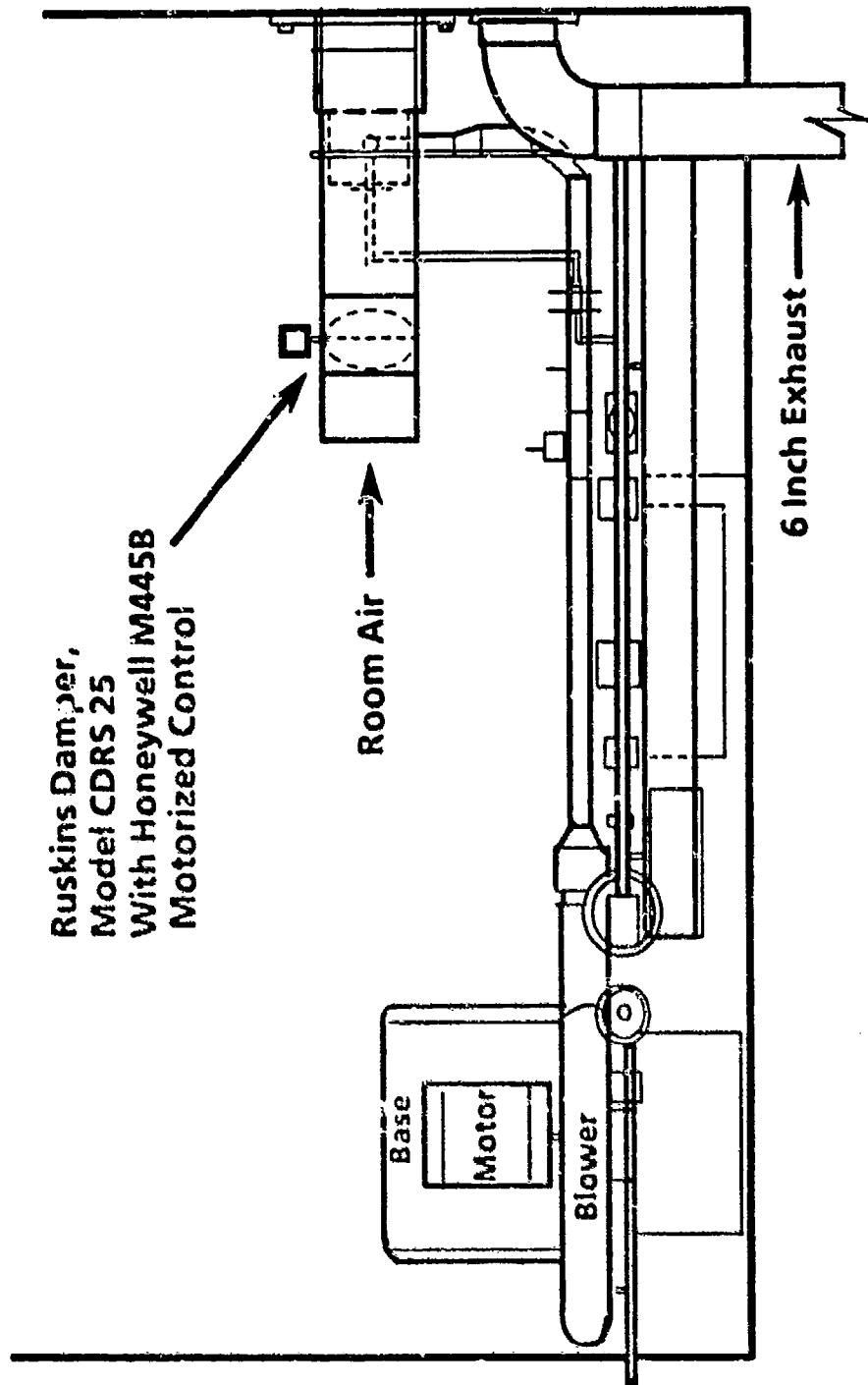
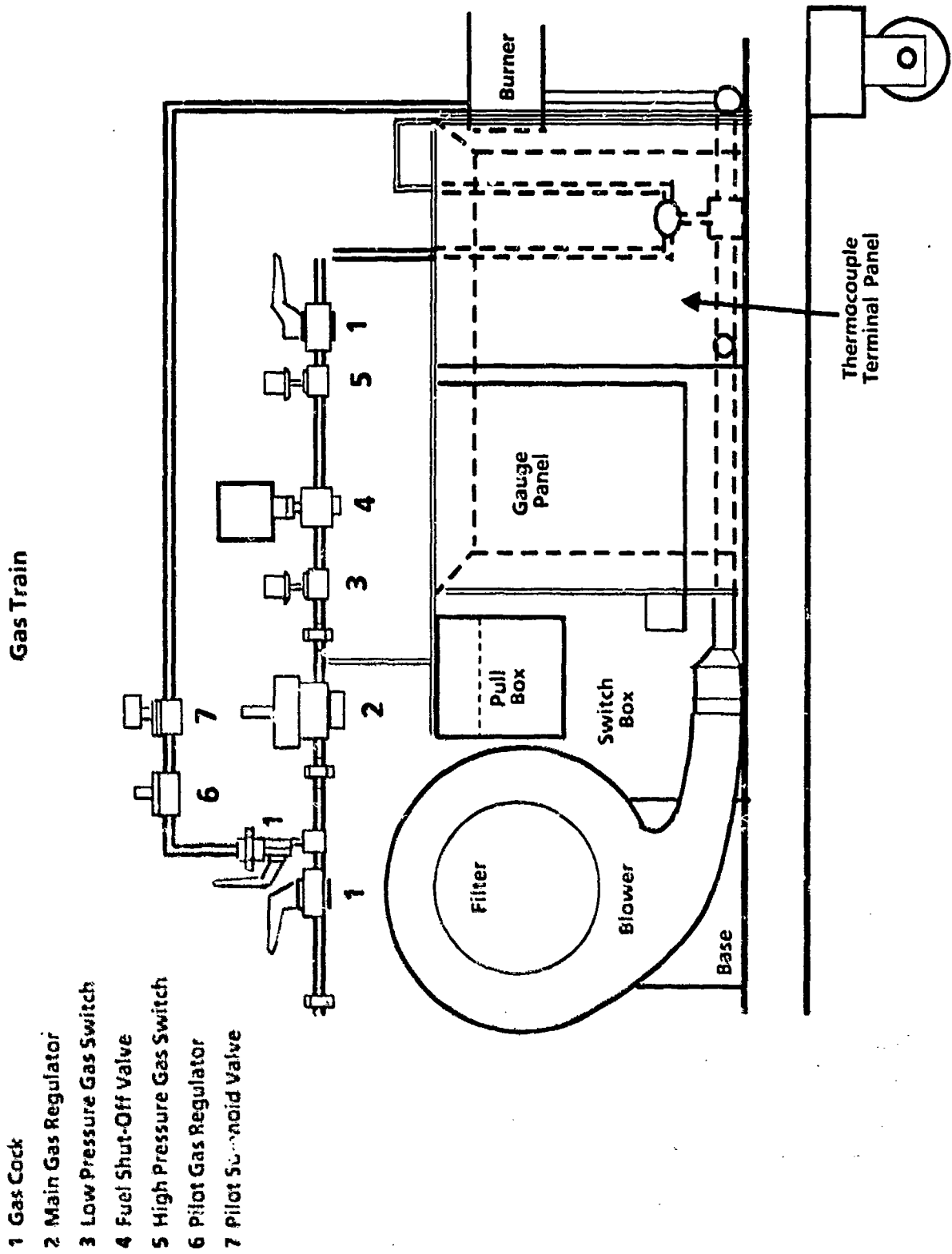


FIGURE 5-3. EQUIPMENT FLOORPLAN - TOP VIEW



- 1 Gas Cock
- 2 Main Gas Regulator
- 3 Low Pressure Gas Switch
- 4 Fuel Shut-Off Valve
- 5 High Pressure Gas Switch
- 6 Pilot Gas Regulator
- 7 Pilot Solenoid Valve

FIGURE 5-4. EQUIPMENT FLOORPLAN - SIDE VIEW

5.3 Equipment Layout and Thermocouple Placement

Figure 5-5 indicates thermocouple placement in the test structure. Standard type K Chromel Alumel thermocouples were used. The flexible type was used to monitor material temperatures (in the solid walls and on the surfaces), and the rigid probe type was used to measure air temperatures in the test structure and in the middle of the hollow block wall.

The following temperatures were monitored in the test structure:

- 1) Inside test structure room (TC 41)
- 2) Inside test structure room, second location (TC 42)
- 3) Inside concrete wall (TC 1)
- 4) Middle concrete wall (TC 2)
- 5) Outside concrete wall (TC 3)
- 6) Inside solid block wall (TC 11)
- 7) Middle solid block wall (air space) (TC 12)
- 8) Outside solid block wall (TC 13)
- 9) Inside hollow block wall (TC 21)
- 10) Middle hollow block wall (TC 22)
- 11) Outside hollow block wall (TC 23)
- 12) Steel wall (TC 43)
- 13) Steel ceiling (TC 54)
- 14) Steel ceiling, second location (TC 55)
- 15) Inside 6 inch floor (TC 8)
- 16) Outside 6 inch floor (TC 9)
- 17) Inside 12 inch floor (TC 27)
- 18) Middle 12 inch floor (TC 28)
- 19) Outside 12 inch floor (TC 29)

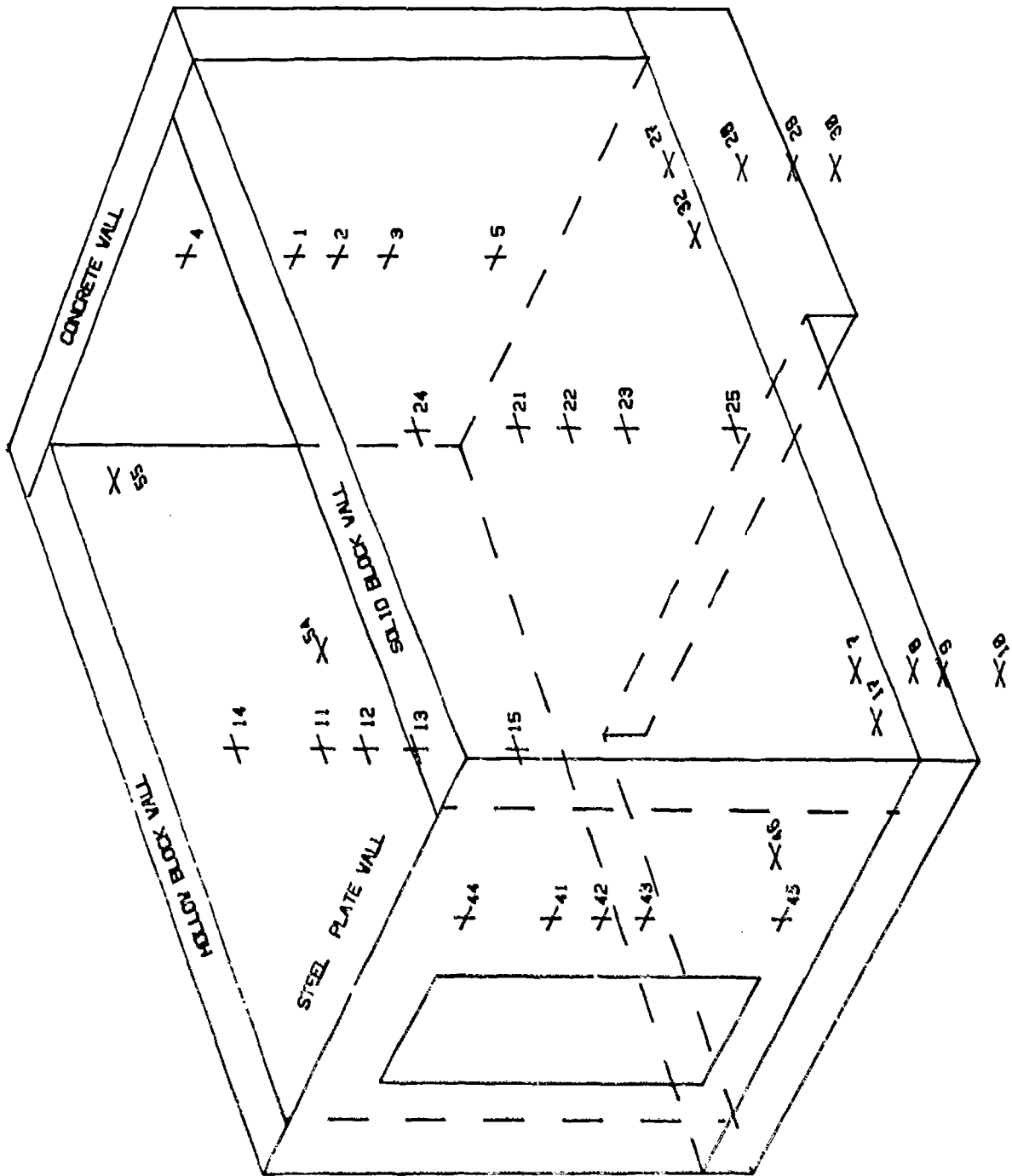


FIGURE 5-5. THERMOCOUPLE PLACEMENT IN THE TEST STRUCTURE

- 20) Bottom soil, 6 inch floor (TC 10)
- 21) Bottom soil, 12 inch floor (TC 30)
- 22) Duct from test room before dilution (TC 47)
- 23) Duct from test room after dilution (TC 48)
- 24) Air to burner (TC 50)
- 25) Chamber air near test structure (TC 51).

5.4 Burner Design and Control

5.4.1 Burner Design

A 1.5 inch KINEMAX medium velocity burner, manufactured by Maxon Corporation, was used to heat the test structure. The maximum capacity for this particular burner is 550,000 Btu/hr. For the purpose of this demonstration it was designed to operate on propane fuel and was supplied with a combustion blower rated at 100 standard cubic feet per minute (scfm). A constant air flow to the burner was used throughout the test.

5.4.2 Burner Control

Thermocouple No. 41 which monitored the air two feet inside the test structure actuated a control at the proportioning control panel to modulate the fuel flow through the combustion train to the burner. The control panel was located in a control room outside the test chamber. Therefore the temperature setpoints (test structure heat-up) could be controlled without entering the test chamber.

Standard industrial safety features built into the burner system included:

1. Automatic shutdown of burner if flame out was sensed by a UV flame detector;
2. Automatic shutdown of burner if high or low gas pressure or high or low burner temperature were sensed; and

3. Automatic shutdown of burner if loss of air flow through the combustion blower was detected.

The dilution air entering the hot gas exhaust duct was controlled by a barometric damper which was adjusted (0.5 in. H₂O) to maintain a negative pressure (-0.2 in. H₂O) in the test structure. Two 1200 actual cubic feet per minute (acfm) blowers were located downstream of the barometric damper and carbon filter in the exhaust duct. These blowers were required during the test to assure adequate tempering of the exhaust gas before it passed through the filter.

A remote cooling air damper, located at the structure's cooling air inlet, was closed during heat-up of the building. At the end of the test, when the burner was turned off, this damper was opened to allow cooling air to enter.

A line diagram of flows and controls for the burner system is shown in Figure 5-6.

5.4.3 Emergency Shutdown of System

In addition to the automatic shutdown safety features of the system, there was also a master control switch on the control panel that could be used in case an unanticipated problem arose in which would not automatically initiate shutdown of the system.

5.5 Data Acquisition System

The data acquisition system, GP-DAS, was provided by DPG. It was housed in a mobile unit (trailer) outside of Building 3008. The system monitored air flow data, thermocouple data, and the pressure inside the test structure continuously.

5.6 Analytical Laboratory

All sample analyses were conducted in the DPG laboratories by DPG personnel. The bubbler samples were analyzed in the laboratory of the Assay Branch, and the material samples and all methods verification analyses were performed by the laboratory in the Technology Branch. Laboratory quality assurance overview was provided by Battelle personnel.

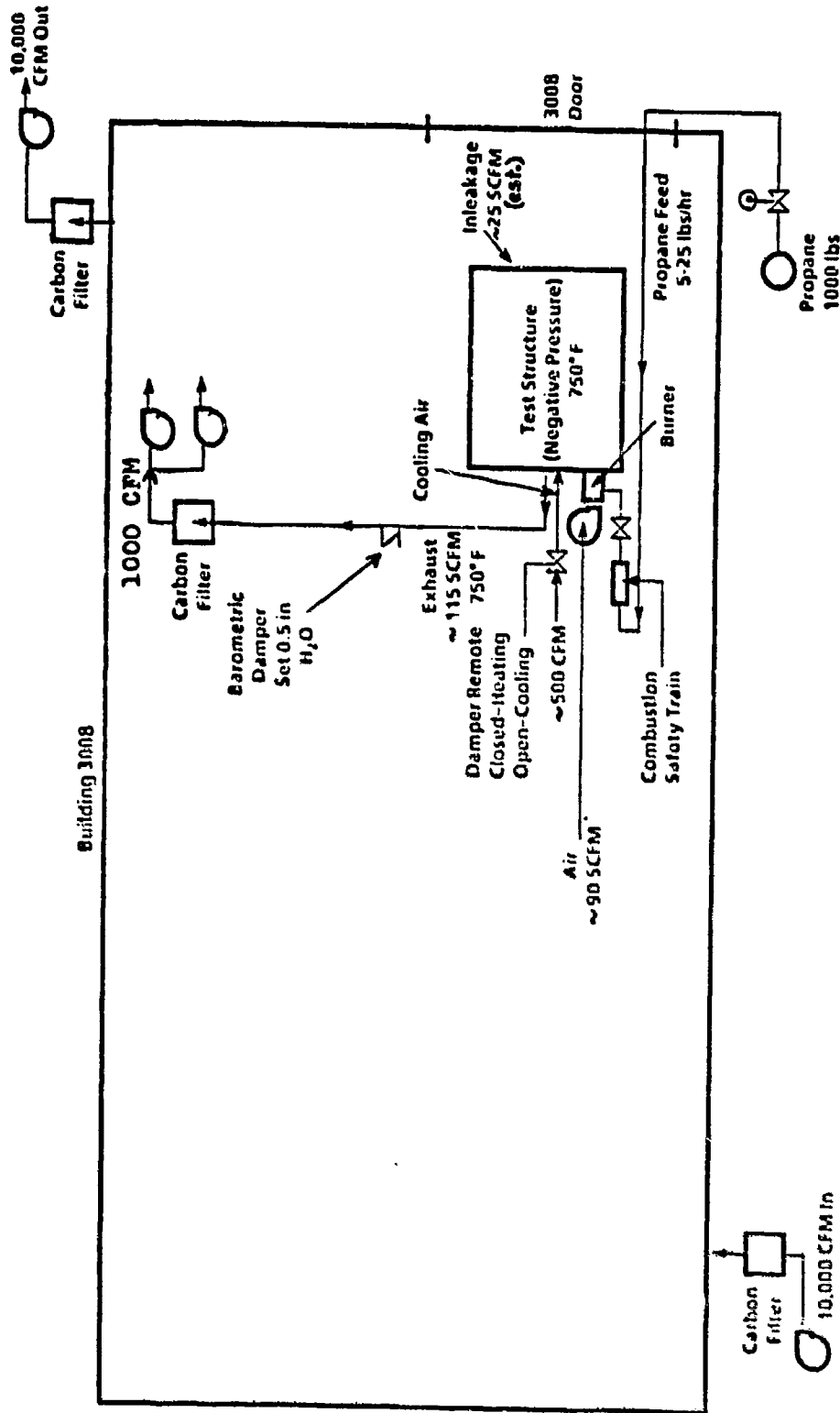


FIGURE 5-6. LINE DIAGRAM OF FLOWS AND CONTROLS

6.0 PREPARATION FOR PILOT TESTING

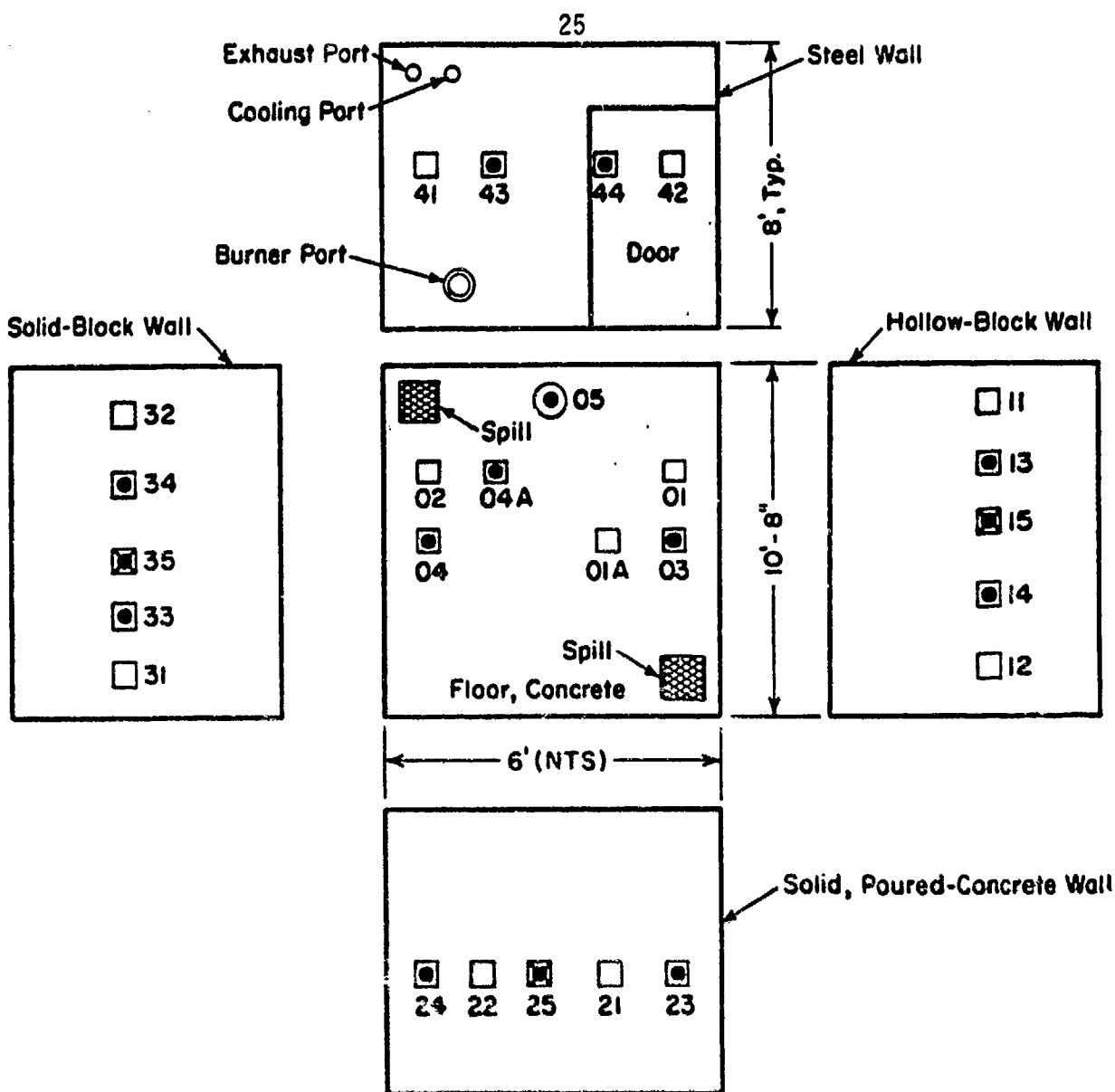
6.1 Installation of Coupon Holders and Design of Coupon Placement

After the test structure was delivered to DPG, it had to be installed in the test chamber and the instrumentation tested. This section describes the pre-test preparations which included coupon holder installation, air flow measurement, and burner modification and checkout.

The locations for the coupons were marked on the inside walls of the test structure according to the placement specified in Figure 6-1. A template and hammer drill were then used at each of these marked locations to drill out the holes for the mounting clips. Lead tampins were inserted in each of the holes, and the mounting clips described in Section 5.2 were screwed in place.

The test design called for all coupons to be spiked and mounted on the walls with their spiked faces facing into the test structure, except the 3 coupons marked in Figure 6-1. These coupons were to be spiked and mounted with the spiked faces toward the wall to approximate agent in concrete at depth. In addition, a 5 inch diameter circular plug of concrete, the "sunken coupon" of Figure 6-1, was spiked on the bottom and inserted into a 4 inch deep hole cut out of the unpainted concrete floor. This coupon was designed to simulate HD contamination at depth. Analysis of coupon no. 15, 25, 35 and this plug would help determine the efficiency of the hot gas process in destroying HD at depth in concrete.

To simulate a spill of HD onto concrete, two 20 x 20 inch areas on the painted and unpainted sections of the concrete floor were ridged in concrete caulking. A controlled spike was delivered to each of these two confined areas and allowed to soak into the concrete for about three days prior to the burner startup. Samples from the spiked areas were collected by drilling and extracting the pulverized concrete (see Appendix B). Drill samples from the



LEGENDS	
<u>Coupon Code</u>	Painted: concrete and steel areas
00 Concrete floor	Unpainted: concrete and steel areas
10 Hollow-block wall	Spiked coupon
20 Concrete wall	Spiked coupon with face to wall
30 Solid-block wall	Spiked coupon (4" deep in concrete floor)
40 Steel wall	Unspiked coupon

FIGURE 6-1. COUPON PLACEMENT IN TEST STRUCTURE

concrete wall beneath coupon no. 15, 25 and 35 would be collected as well to determine if detectable HD had migrated into the concrete wall from the spiked coupon faces.

6.2 Air Flow Measurement

Prior to the test, the system was operated with ambient air, primarily to verify that:

1. negative pressure (-0.1 to -0.2 in water column) could be maintained inside the test structure, and
2. air flow in the exhaust duct, after dilution and before the 1200 acfm carbon filter, was at least 1000 acfm.

Negative pressure was required in the test structure to prevent HD contaminated gases from leaking into the test chamber; low pressure was needed to minimize in-leakage. The exhaust system was cooled with dilution air to protect the carbon filters which can sustain a maximum temperature of about 400°F. A minimum flow of 1000 acfm was maintained.

Other system flows which were required as general data input were the combustion air flow and exhaust flow prior to dilution through the barometric damper.

6.3 Instrument Checkout

The thermocouples in the test structure, exhaust duct, combustion air, and test chamber were calibrated using millivolt (mv) signal generators. All pressure transducers were zeroed and calibrated over the full response range. Using a milliamp (ma) signal from each of the transmitters, all the indicator/controller readouts in the control trailer control panel were then standardized to give the correct readouts.

Each Magnehelic pressure gauge was zeroed and checked against the corresponding pressure transducer readout in the GP-DAS trailer.

6.4 Burner Modification and Startup

The system equipment furnished by the building contractor was not adequate to provide automated control of fuel flow to the burner. Blair-Alexander Engineering (supplier of the burner) was contacted to provide initial adjustment and light-off of the system, as well as to modify the system to make burner control more fully automated. Blair-Alexander Engineering provided the following equipment for this modification:

1. high temperature cut-off,
2. temperature controller,
3. two thermocouples, and
4. motor for the fuel control valve.

Following the installation and wiring of these components, the fuel valve was sized and the 0-100% span was set on the valve control motor. During the initial startup of the burner, it was determined that a constant combustion air flow of 60% of maximum gave the best light-off and flame throughout the burner operating range.

Startup and operational procedures for the actual test are detailed in the Burner SOP (Appendix D).



Figure 6-2. Test Structure, Equipment Platform, and Burner Assembly in the Environmental Chamber

7.0 TESTING

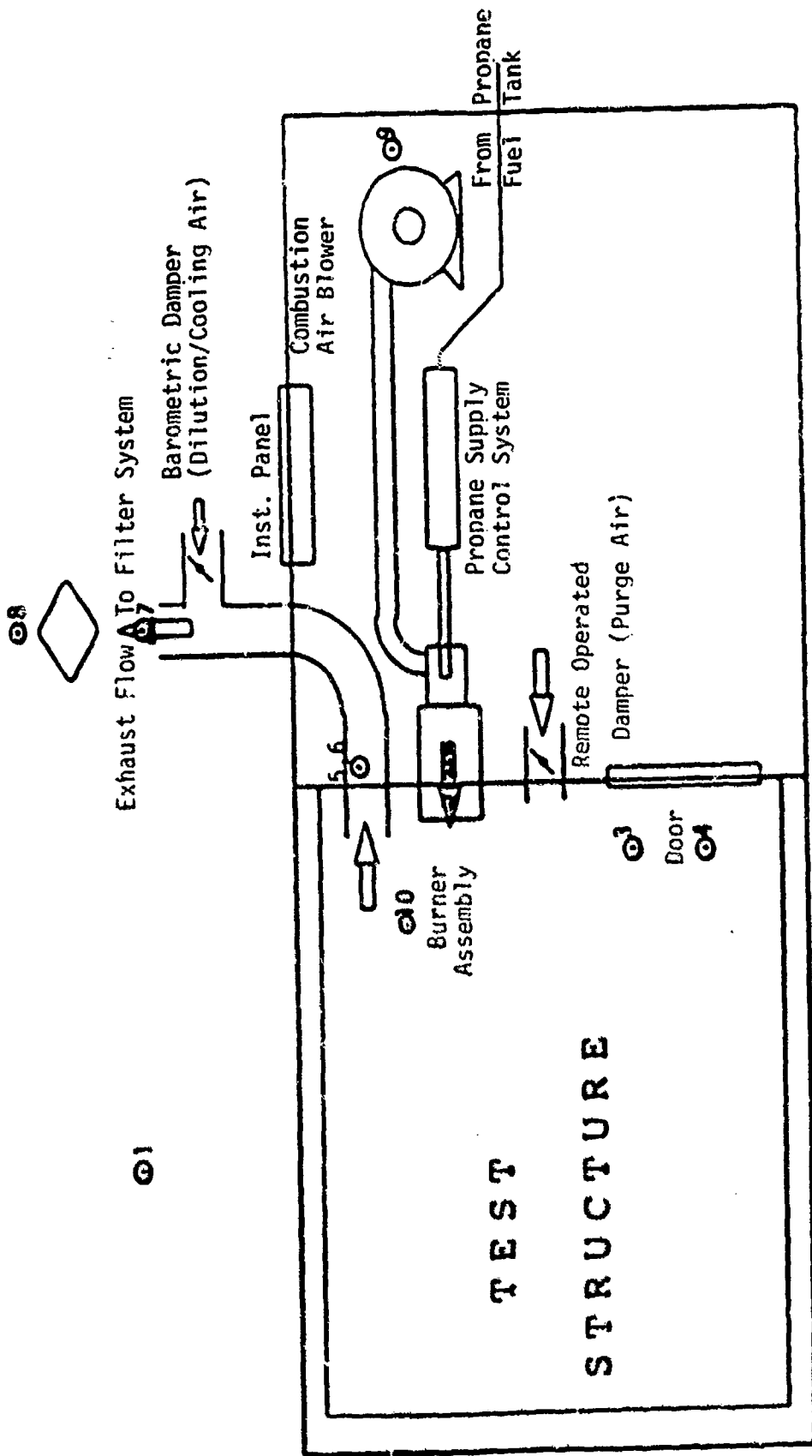
7.1 Background Data

Before the testing began, the thermocouple and pressure transducers were calibrated and background data such as temperature, pressure, and air flow rates were taken by the data acquisition system. The calibration provided confidence in the readings and the background data provided a point of reference for the balance of the test. Background chemical analysis samples were also taken on July 16, 1987. These samples included concrete floor samples (powdered concrete from drilling 3/8 inch diameter holes 3/8 inch to 1/2 inch deep) next to the floor spill areas and wipe samples from the floor and wall. Air samples were taken using bubblers and an ACAMS.

The bubblers were placed at the following locations (Figure 7-1):

- East Floor--air next to test chamber on solid block wall side
- West Floor--air next to test chamber on burner side
- Upper Door--air in test structure 2 feet above the floor, collected from the second from bottom pipe nipple in door
- Lower Door--same as above, except collected from the bottom pipe nipple in door
- East Exhaust--east side of exhaust before dilution with water cooled condenser
- West Exhaust--west side of exhaust, same as above
- Pre 1200 acfm--exhaust after cooling air dilution
- 1200 acfm Exhaust--exhaust after carbon filter
- Burner Fan-- air near burner blower air inlet
- Soil Area-- air between the soil base and the test structure's 6-inch thick floor underneath a spiked floor spill area.

Two other bubblers were used for monitoring outside of Building 3008 for safety purposes. These results were also reported.



- AIR SAMPLING LOCATIONS:**
1. EAST FLOOR, TEST CHAMBER
 2. WEST FLOOR, TEST CHAMBER
 3. UPPER DOOR, TEST STRUCTURE
 4. LOWER DOOR, TEST STRUCTURE
 5. EAST EXHAUST
 6. WEST EXHAUST
 7. PRE-1200-CFM FILTER
 8. 1200-CFM EXHAUST
 9. BURNER FAN (TEST CHAMBER)
 10. UNDER FLOOR SPILL (SOIL)

FIGURE 7-1. LOCATION OF BUBBLER STATIONS. (ACAMS ALSO LOCATED AT STATION 5.)

The ACAMS sample was taken using a tee in the sample line to the East Exhaust bubbler after the condenser. The ACAMS is an on-line monitor that provides an updated reading every 8 minutes. The bubblers used 10 milliliters of hexylene glycol to collect material for an approximate 6-hour sampling period of approximately 1 liter per minute of air pulled through the bubbler (0.1 mg/ml equals approximately 0.003 mg/m³).

The background data collection continued during the coupon spiking operation and during the three days after spiking before the burner was fired.

7.2 Decon Testing

On July 17, 1987, between 0900-1000, the coupons were spiked with approximately 7.6 ml of a 2.0 weight percent solution of HD in hexane to deliver 95 mg of HD to each coupon. After the coupons were placed into their holders in the test structure, the floor spill areas were spiked. The test structure was then closed. The ventilation blowers remained on to keep the test structure at negative pressure and pass the exhaust through the 1200 acfm activated carbon filter. Data and air samples were taken over the weekend while the HD was allowed to soak into the test structure. The ACAMS responded to the HD in the test structure. The reading was over 20.0* after spiking. Before the burner was started July 20, 1987, the ACAMS reading had decreased to 0.23*.

The burner was started July 20, 1987, at 0910 at a controller set point temperature setting of 200°F. The ACAMS reading rose rapidly, responding to the start of the burner. At 1000 the burner control set point was raised to 250°F and the ACAMS reading was 3.00*. The test exhaust temperature was 192°F. The burner control set point was raised 50°F every hour until it reached 400°F, after which it was held at this setting for 6 hours. During

*Reading is number times 0.003 mg/m³.

this heat-up period, no steam was observed coming from the exterior of the test structure. The ACAMS reading peaked at a value of 4.30* at 1022 and decreased after this point. The exhaust bubblers and ACAMS condenser cooling water were turned on at 1200 when the exhaust temperature reached 284°F.

The burner control set point was raised in increments of 50°F every hour to 750°F, starting at 1900. The ACAMS responded to the temperature increase with an increase from 0.3* to about 0.9* at 2200.

When the bubblers were changed at 2400 it was noted that the bubblers that were sampling from the exhaust and from the test structure air were filled and overflowing with water that had condensed in the condenser and/or sampling lines. The water also had flowed into the ACAMS, which made the ACAMS readings questionable for the balance of the test. The water in the bubblers prevented those samples from being analyzed. Water traps were installed in the bubbler sample lines to help alleviate this problem for the rest of the test.

Also at this time, 2400, July 20, 1987, steam was visible at the seams of the metal covering the insulation on the exterior of the test structure. The outside wall temperatures were near 200-212°F at this time, indicating that moisture was being driven out of the concrete. Water had been collecting in the bubbler that was sampling the air beneath the test structure floor since about 6 hours into the heatup phase. This water was probably released from the soil in the test structure base as the temperature increased.

During the evening of July 20, 1987 and the morning of July 21, 1987, a severe electrical storm caused three short disruptions of commercial power to the GP-DAS data collection system. No serious damage to the data integrity was sustained. The burner, powered by its own generator, continued uninterrupted.

The final burner control setting of 750°F was attained at 0100, July 21, 1987. The burner was maintained at this temperature until all the building

temperatures exceeded 300°F for a minimum time of 1 hour. This condition was reached by 2000 on July 21, 1987, 35 hours after the burner was fired. The burner was shut off at 2020 at the same time that the bubblers were switched. This started the cooldown period. The cooldown period was ended 38 hours later when all the test structure internal temperatures dropped to 105°F or less.

The burner air blower was kept operating to blow cooling air into the test structure along with the cooling air entering the structure from the cooling air inlet duct. As the exhaust temperature leaving the test structure decreased, the dilution air inlet damper was closed to pull as much cooling air through the test structure as possible without exceeding the temperature limit to the inlet of the carbon filter. Cooldown ended at 1000 July 23, 1987, and the test structure door was opened.

7.3 Post Testing

Photographs were taken of the test structure after opening and before the coupons were removed. After photographing the test structure, the coupons were removed and placed in unused plexiglass boxes for transport to the chemical laboratory for analysis, which included analysis of surface wipes of the coupons and also analysis of selected concrete coupons that were pulverized and extracted.

Swab samples were collected from the test structure at the following locations:

- in each of the floor spill areas
- near the spill areas on the floor, painted and unpainted
- each wall, painted and unpainted
- ceiling.

Concrete drill samples were taken from each of the floor spill areas and areas outside the spills. Concrete drill samples were also taken on the walls under coupons No. 15, 25 and 35, which were placed with the spiked face toward the wall. Other samples taken included 2 vials of paint chips that were removed from the metal wall and floor beam, and four soil samples taken about 18 inches in from the wall under the test structure. Both pre-test and post test samples collected and analyzed are listed in Tables 7-1 and 7-2.

TABLE 7-1. PRE-TEST BLANK SAMPLES

<u>LABORATORY SAMPLE NO.</u>	<u>LOCATION OF SAMPLE/ TYPE OF MATERIAL</u>	<u>TYPE OF SAMPLE</u>
71	Steel, coupon	Swab
72	Painted steel, coupon	Swab
81	Concrete, coupon	Swab
82	Painted concrete, coupon	Swab
C1	Painted concrete, floor	Swab
C2	Painted concrete, solid block wall	Swab
C3	Painted concrete, hollow block wall	Swab
C4	Concrete	Swab
C5	Concrete, floor	Swab
C6	Painted steel, ceiling	Swab
C7	Steel, ceiling	Swab
C8	Concrete, solid block wall	Swab
C9	Painted concrete	Swab
C10	Concrete, hollow block wall	Swab

TABLE 7-2. POST-TEST SAMPLES

<u>LABORATORY SAMPLE NO.</u>	<u>LOCATION AND TYPE OF MATERIAL</u>	<u>SAMPLE TYPE</u>	<u>SOLVENT EXTRACT</u>
1	Steel, unpainted	Swab	Hexane
2	Steel, painted	Swab	Hexane
3	Painted Floor Spill	Swab	Hexane
4	Painted Steel	Swab	Hexane
5	Unpainted hollow concrete block	Swab	Hexane
6	Unpainted concrete	Swab	Hexane
7	Hollow concrete block, painted	Swab	Hexane
8	Floor spill, unpainted	Swab	Hexane
9	Concrete, painted	Swab	Hexane
10	Solid concrete block,	Swab	Hexane
11	Steel, ceiling	Swab	Hexane
12	Solid block, painted	Swab	Hexane
13	Soil, painted	Soil	Chloroform
14	Soil, unpainted (under spill area)	Soil	Chloroform
15	Wall, solid concrete, under #35	Drill	Chloroform
16	Wall, concrete, under #25	Drill	Chloroform
17	Wall, hollow concrete, under #15	Drill	Chloroform
18	Unpainted concrete, floor spill area	Drill	Chloroform
19	Painted concrete floor spill area	Drill	Chloroform
20	Soil, painted under spill area	Soil	Chloroform

TABLE 7-2. POST-TEST SAMPLES (continued)

<u>LABORATORY SAMPLE NO.</u>	<u>LOCATION AND TYPE OF MATERIAL</u>	<u>SAMPLE TYPE</u>	<u>SOLVENT EXTRACT</u>
21	Soil, unpainted	Soil	Chloroform
22	Paint from center beam in floor	Paint	Chloroform
23	Steel door paint sample	Paint	Chloroform
24	Standard, HD in cyclohexane		
25	Coupon #05, concrete circular (floor)	Swab*	Hexane
26	Coupon 02, painted concrete	Swab	Hexane
27	Coupon 22, painted concrete	Swab	Hexane
28	Coupon 32, painted concrete	Swab	Hexane
29	Coupon 35, concrete	Swab*	Hexane
30	Coupon 10, concrete	Swab	Hexane
31	Coupon 41, steel	Swab	Hexane
32	Coupon 43, steel	Swab	Hexane
33	Coupon 04, painted concrete	Swab*	Hexane
34	Coupon 04A, painted concrete	Swab	Hexane
35	Coupon 24, painted concrete (broken)	Swab	Hexane
36	Coupon 34, painted concrete	Swab	Hexane
37	Coupon 15, concrete	Swab	Hexane
38	Coupon 21, concrete	Swab	Hexane
39	Coupon 11, concrete	Swab	Hexane
40	Coupon 01A, concrete	Swab	Hexane
41	Coupon 33, concrete	Swab	Hexane
42	Coupon 42, painted steel	Swab	Hexane

TABLE 7-2. POST-TEST SAMPLES (continued)

<u>LABORATORY SAMPLE NO.</u>	<u>LOCATION AND TYPE OF MATERIAL</u>	<u>SAMPLE TYPE</u>	<u>SOLVENT EXTRACT</u>
43	Coupon 14, painted concrete	Swab*	Hexane
44	Coupon 44, painted steel	Swab	Hexane
45	Coupon 45, concrete	Swab*	Hexane
46	Coupon 31, concrete	Swab	Hexane
47	Coupon 12, painted concrete	Swab	Hexane
48	Coupon 13, concrete	Swab*	Hexane
49	Coupon 23, concrete	Swab*	Hexane
50	Coupon 25, painted concrete	Swab	Hexane
51	Floor, painted background	drill	Chloroform
52	Floor, unpained background	drill	Chloroform

*These coupons were also pulverized, extracted and analyzed after swab samples were collected.

8.0 PILOT TEST RESULTS

8.1 Test Structure Integrity

The condition of the test structure after the pilot test is shown in Figures 8-1 and 8-2. The photographs show the interior of the structure from the doorway and from the interior looking toward the doorway.

The test structure appeared to be structurally sound and in excellent condition, although no tests for structural soundness were performed. The structure showed no visible signs of structural damage as a result of the thermal decontamination test. There were no cracks in the wall, no separation of the walls at the corners or ceiling. The concrete did not appear to be weakened by the heating based on the time required to drill 3/8 inch diameter holes after the test. The alkyd (Sani-flat) paint used on sections of the floor and walls remained intact on the concrete but chalked and flaked off the metal. This difference in performance may be related to the higher temperatures attained on the metal surfaces compared with those attained on the concrete surfaces.

Several concrete coupons fell to the floor during the test. They remained relatively intact, with only corners chipped off most of them. The coupons fell during the test due to the melting of the lead anchors that were used to install the coupon mounting clips on the concrete block walls and the concrete wall. In some places, lead was splattered on the floor of the test structure.

The following was the status of the coupons:

<u>Coupon No.</u>	<u>Comments</u>
11	Fallen to floor, 1 large piece, 2 small pieces
13,15	In place on wall, loose
14,22,33	Fallen to floor, intact
12	Fallen to floor, 1 large piece, 1 small piece
23,21	In place on wall, loose

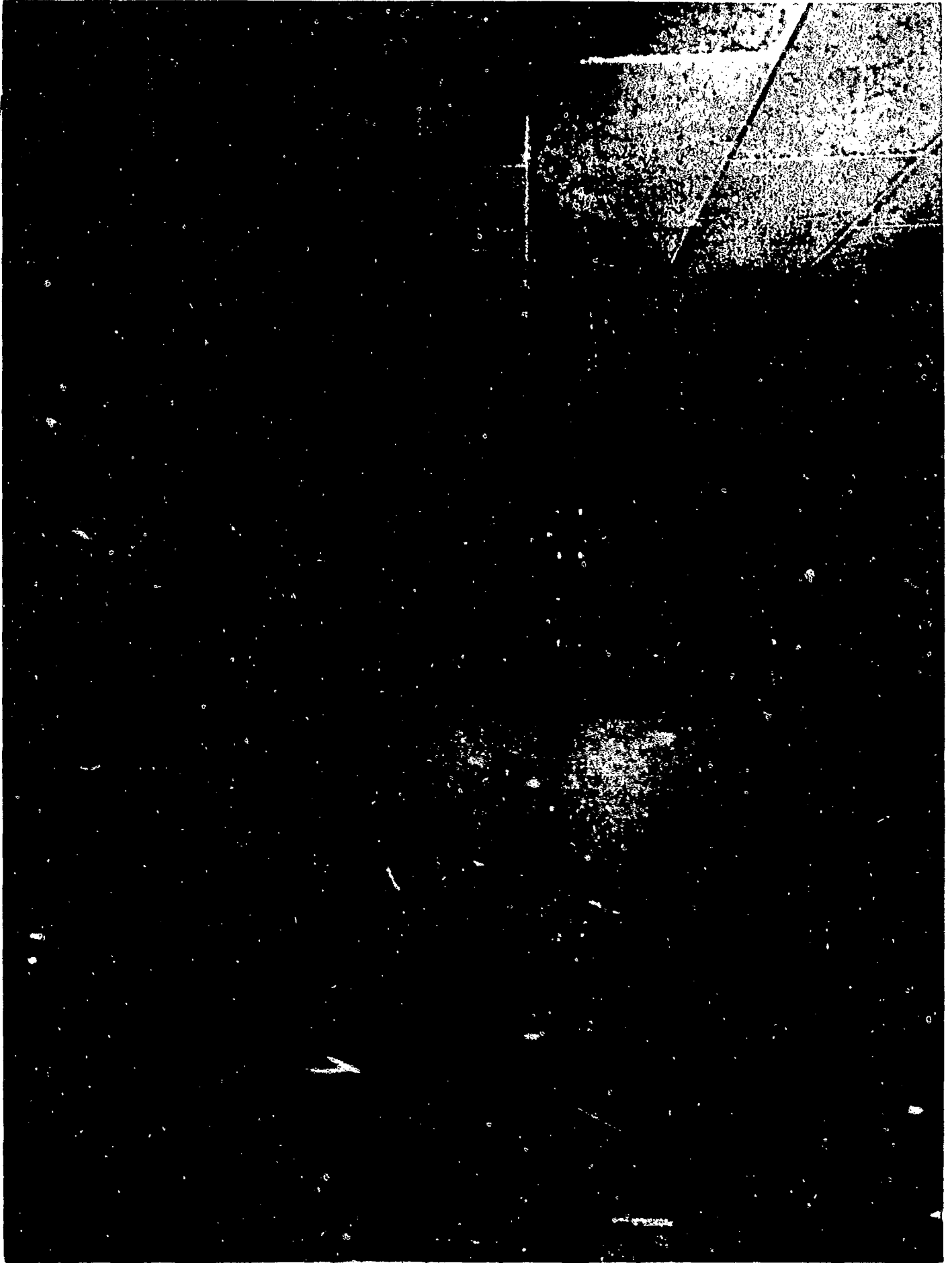


Figure 8-1. Photo of Interior of Test Structure from Doorway



Figure 8-2. Photo of Interior Floor of Test Structure

25	Fallen to floor, 1 large piece, 1 very small corner piece
24	Fallen to floor, approximately 5 pieces
31	Fallen to floor, 2 small corners missing
35,32,34	In place on wall, loose
41,42,43,44	In place, tight.

8.2 Heat-up Profiles

The temperature heat-up and cool-down profiles of each of the walls and floor sections are shown in Figures 8-3 to 8-8. The plots begin with the startup of the burner, which was turned on 80 hours after the test structure was spiked. The graphs for the poured concrete wall, the solid concrete block wall, and the hollow concrete block wall compare thermocouple readings from the interior surface of each wall, middle of each wall, and three locations on the exterior surface of each wall. The plot for the steel wall displays the temperature profiles for halfway up the wall and near the ceiling. The profiles for the two floor sections contain readings from the interior surface of the floor, the middle of the floor, the exterior surface of the floor, and the soil beneath the floor ("sand box"). Each graph also contains the temperature profile of the thermocouple used for burner control for comparison with the other thermal profiles displayed.

The thermal profiles show that for the concrete walls and the floors, the interior surface temperatures of the walls heated faster and attained higher temperatures than did either the middle of the walls or the exterior surfaces. The thermal profiles of the steel wall paralleled the burner air temperature profile very closely.

Figures 8-9 and 8-10 compare all interior and exterior wall surface temperatures, respectively. The steel wall reached the highest temperatures during the test. The poured concrete wall interior surface reached the highest temperatures of all the non-steel walls during the heatup phase, and the hollow block wall interior surface was the least responsive to the burner air

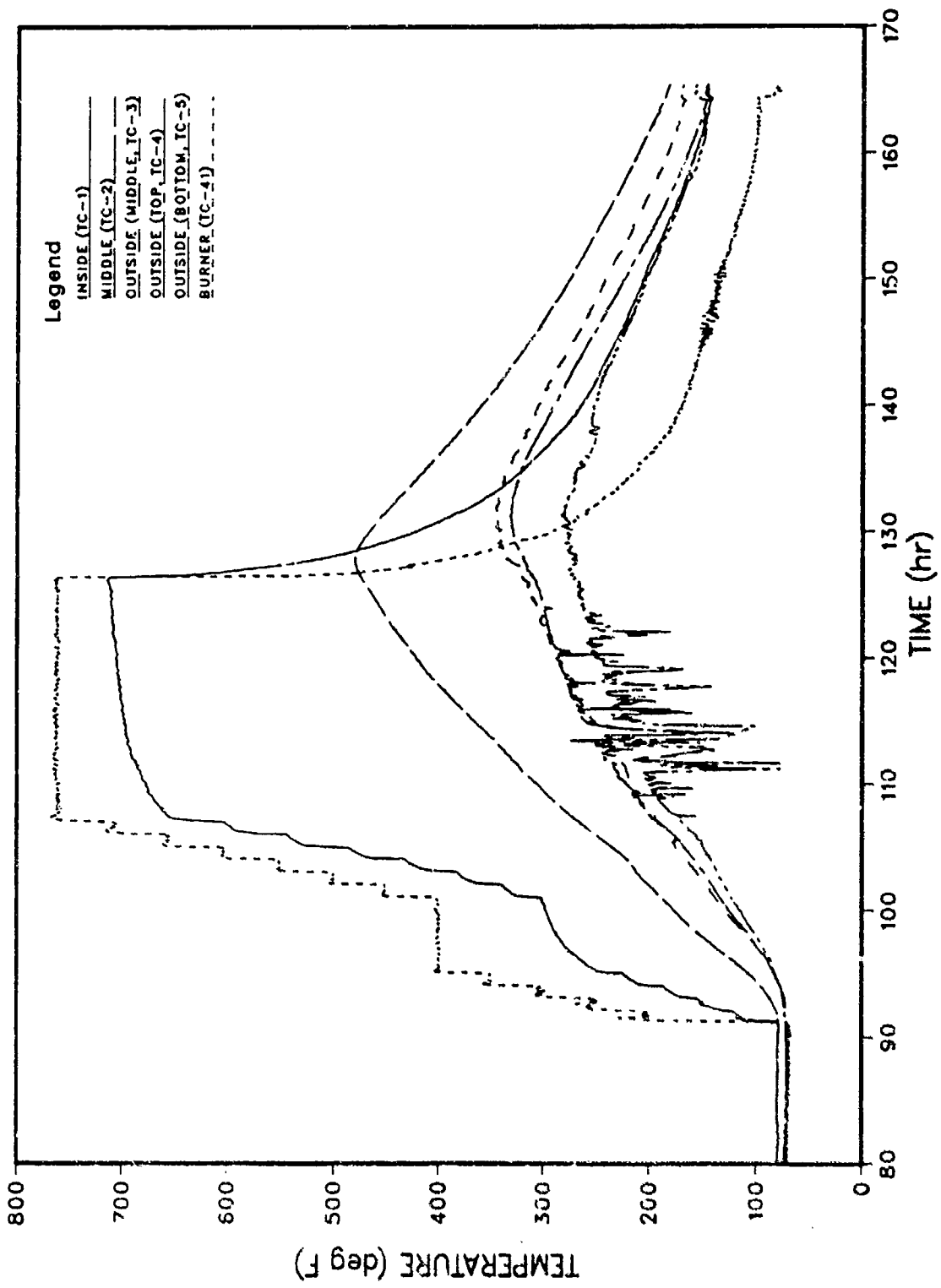


FIGURE 8-3. HEATUP PROFILE OF THE CONCRETE WALL

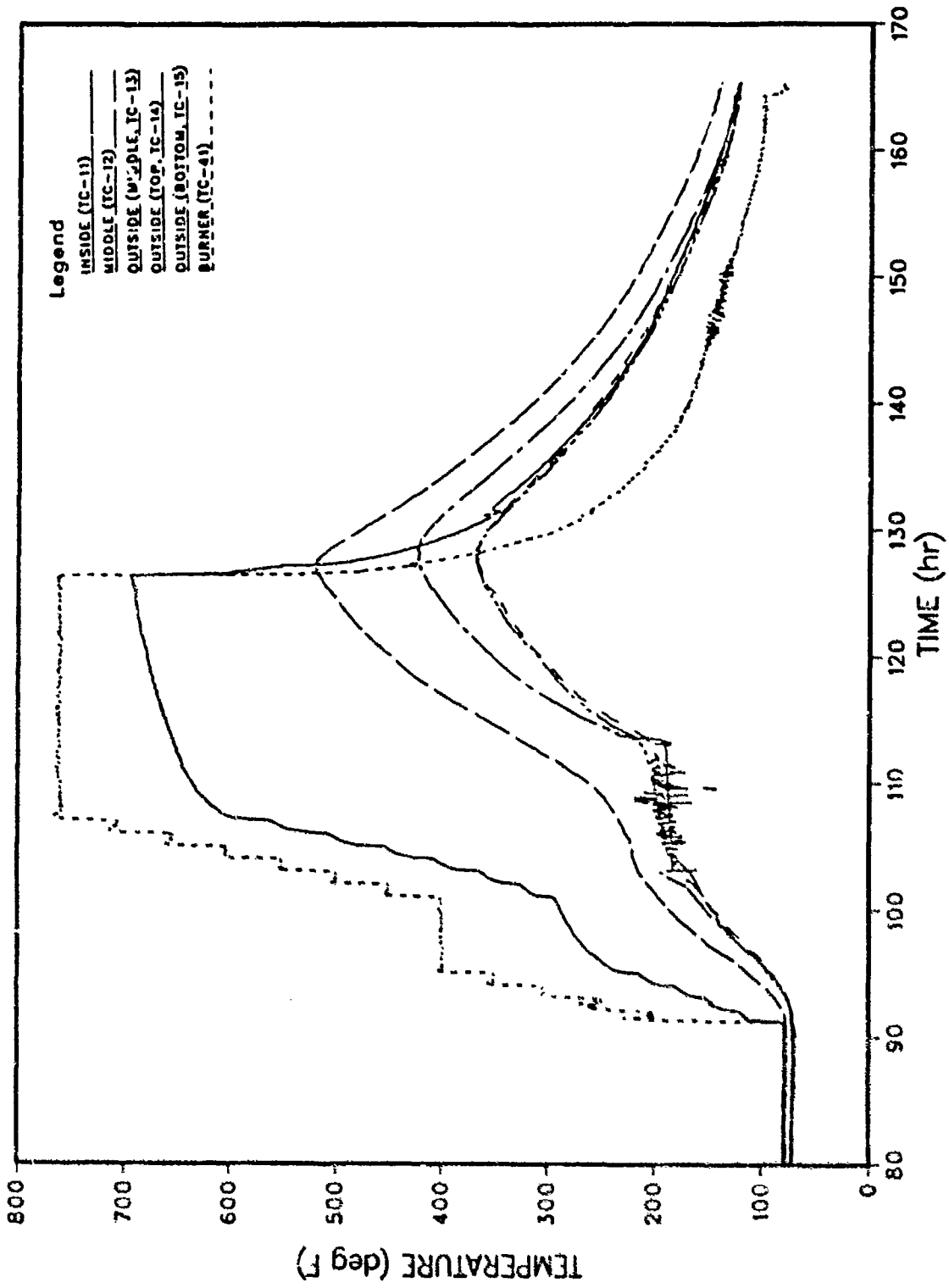


FIGURE 8-4. HEATUP PROFILE OF SOLID BLOCK WALL

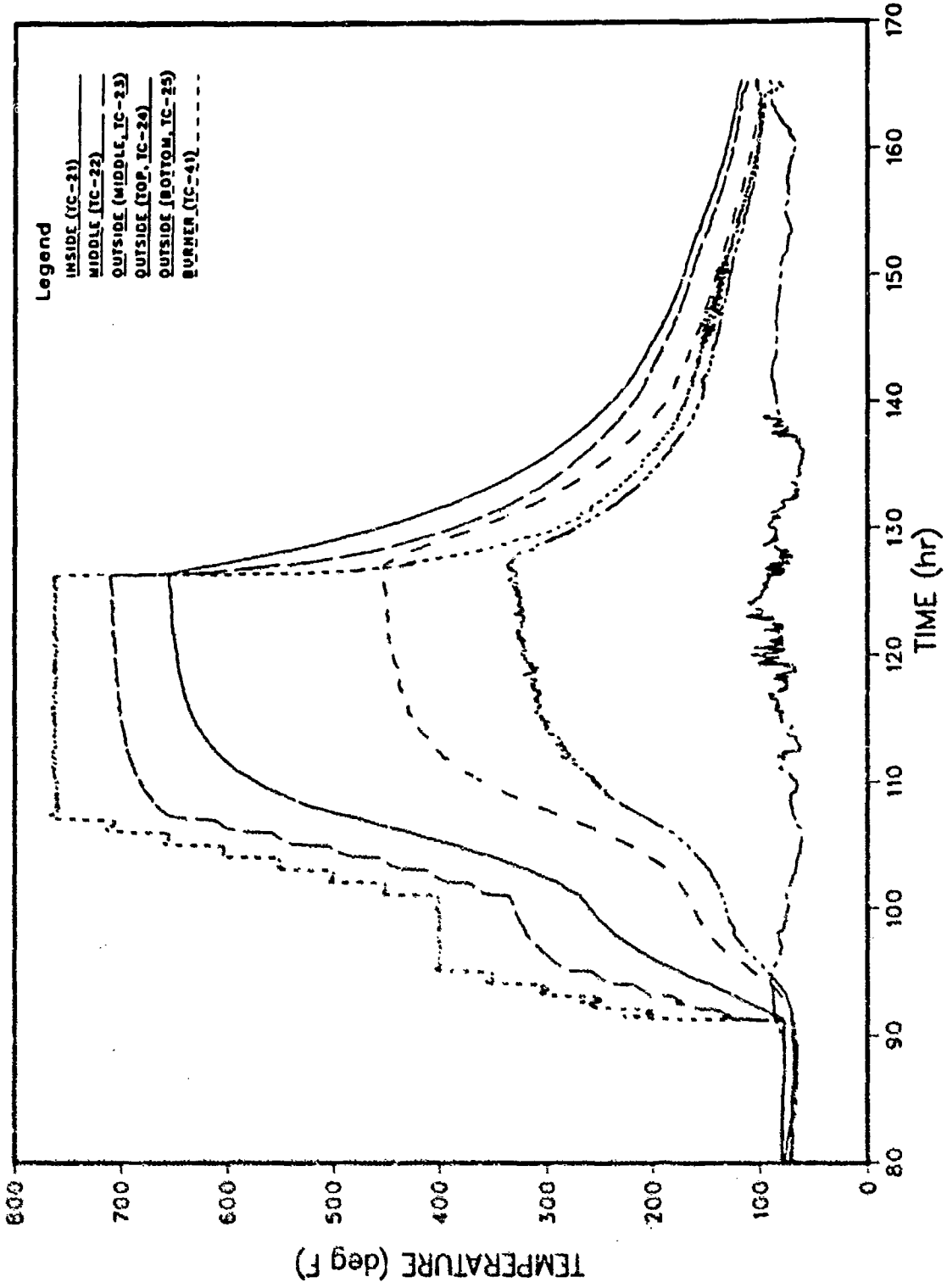


FIGURE 8-5. HEATUP PROFILE OF HOLLOW BLOCK WALL

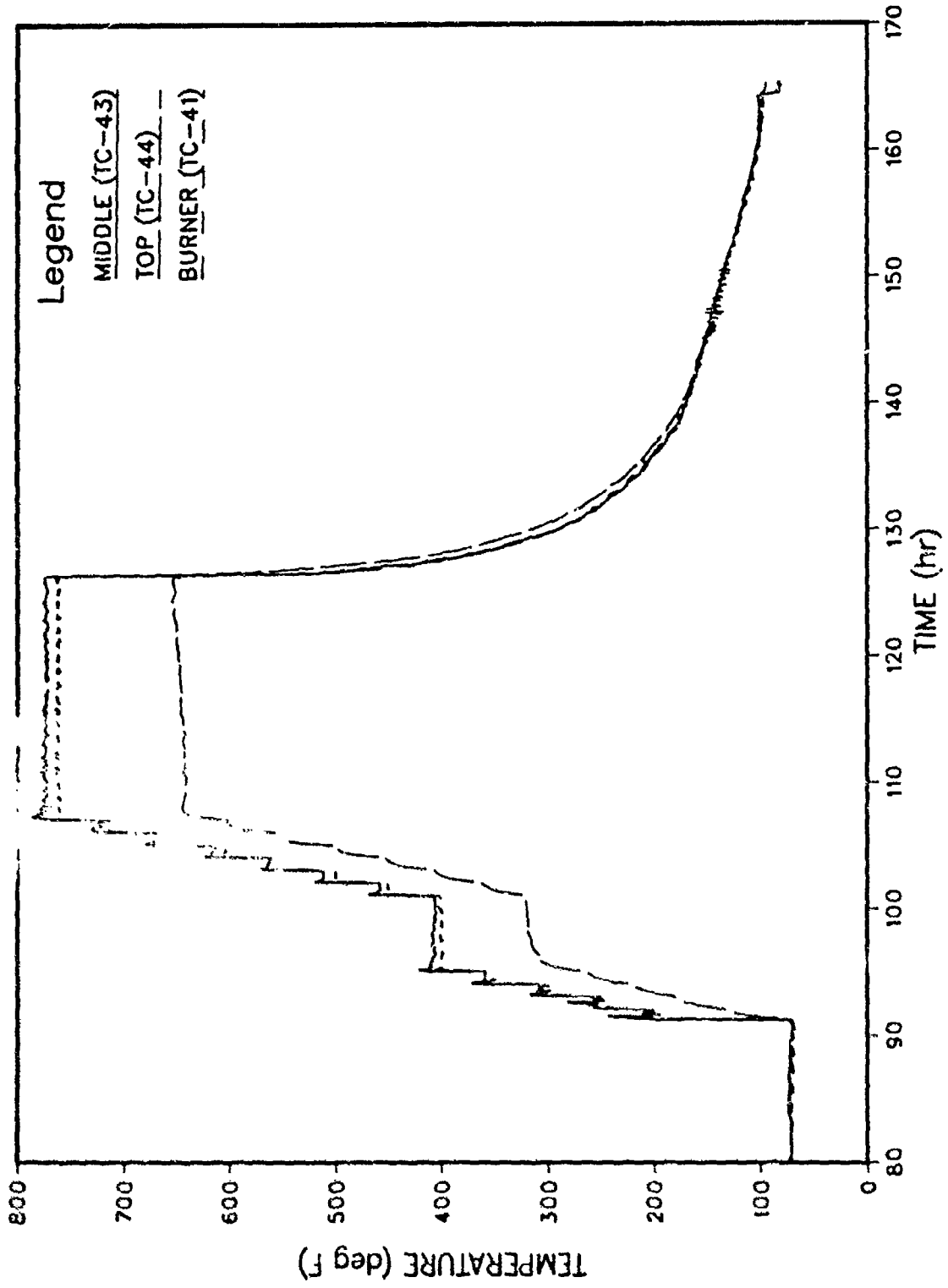


FIGURE 8-6. HEATUP PROFILE OF STEEL PLATE WALL

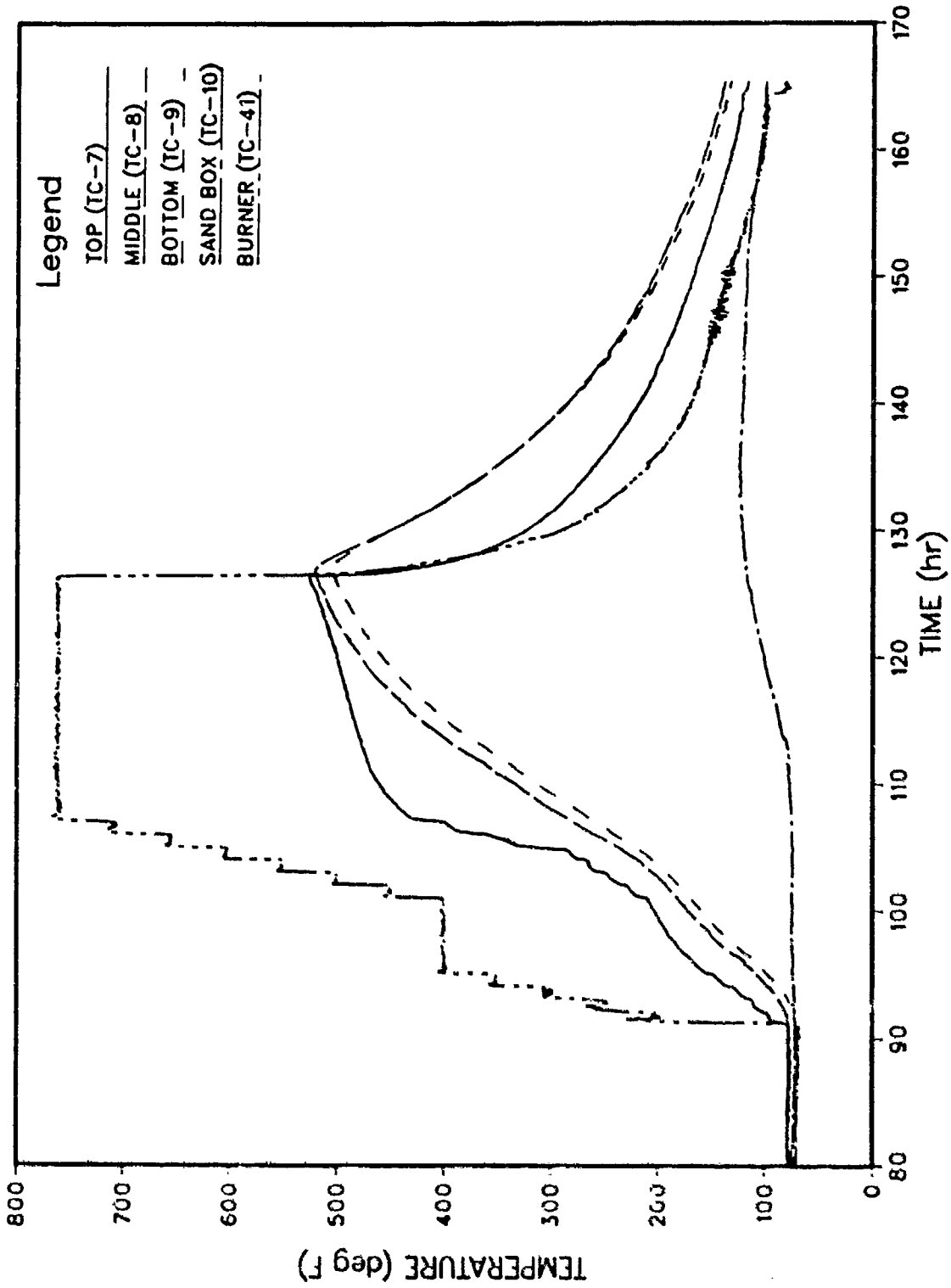


FIGURE 8-7. HEATUP PROFILE OF 6-INCH THICK FLOOR

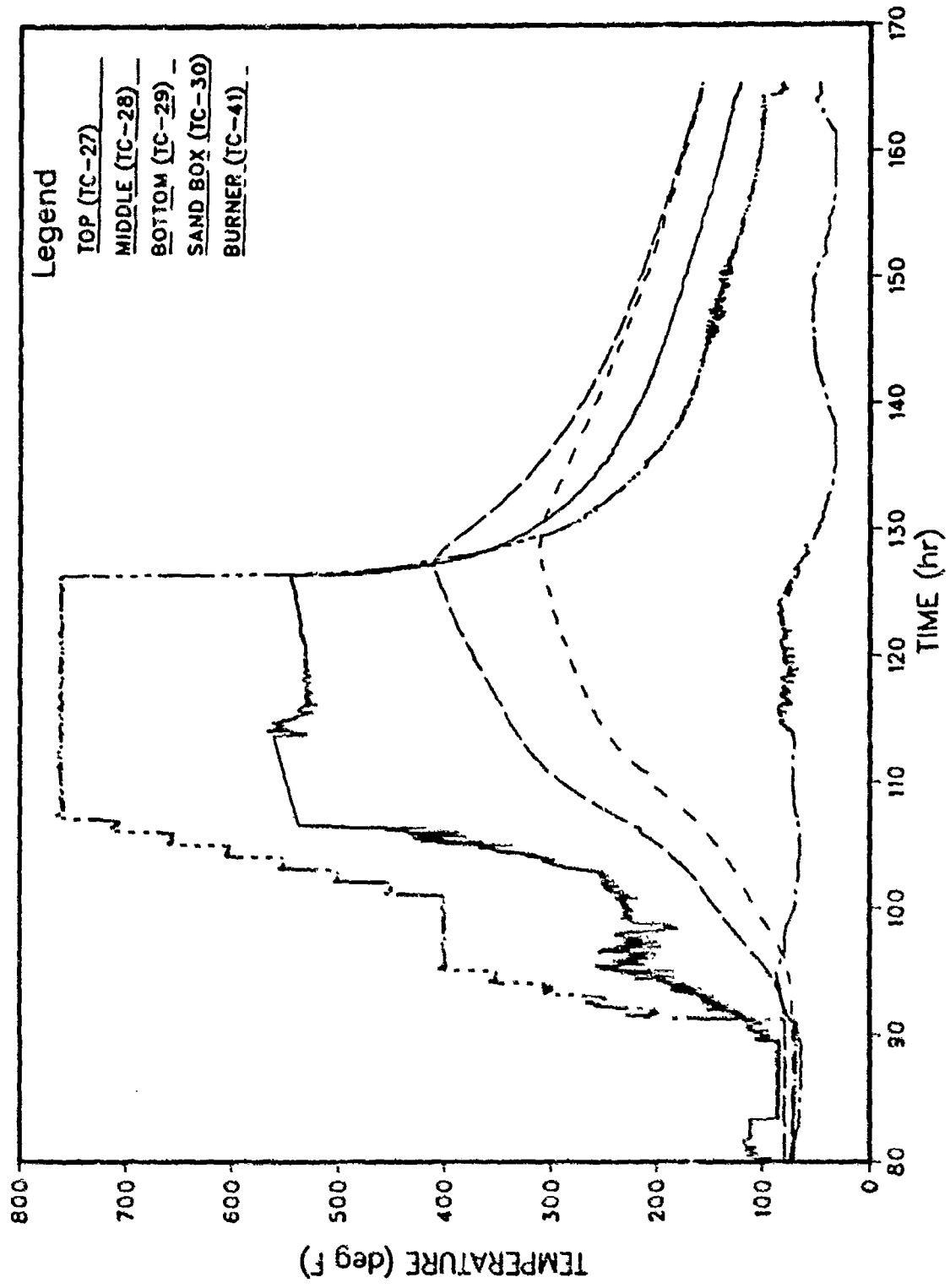


FIGURE 8-8. HEATUP PROFILE OF 12-INCH THICK FLOOR

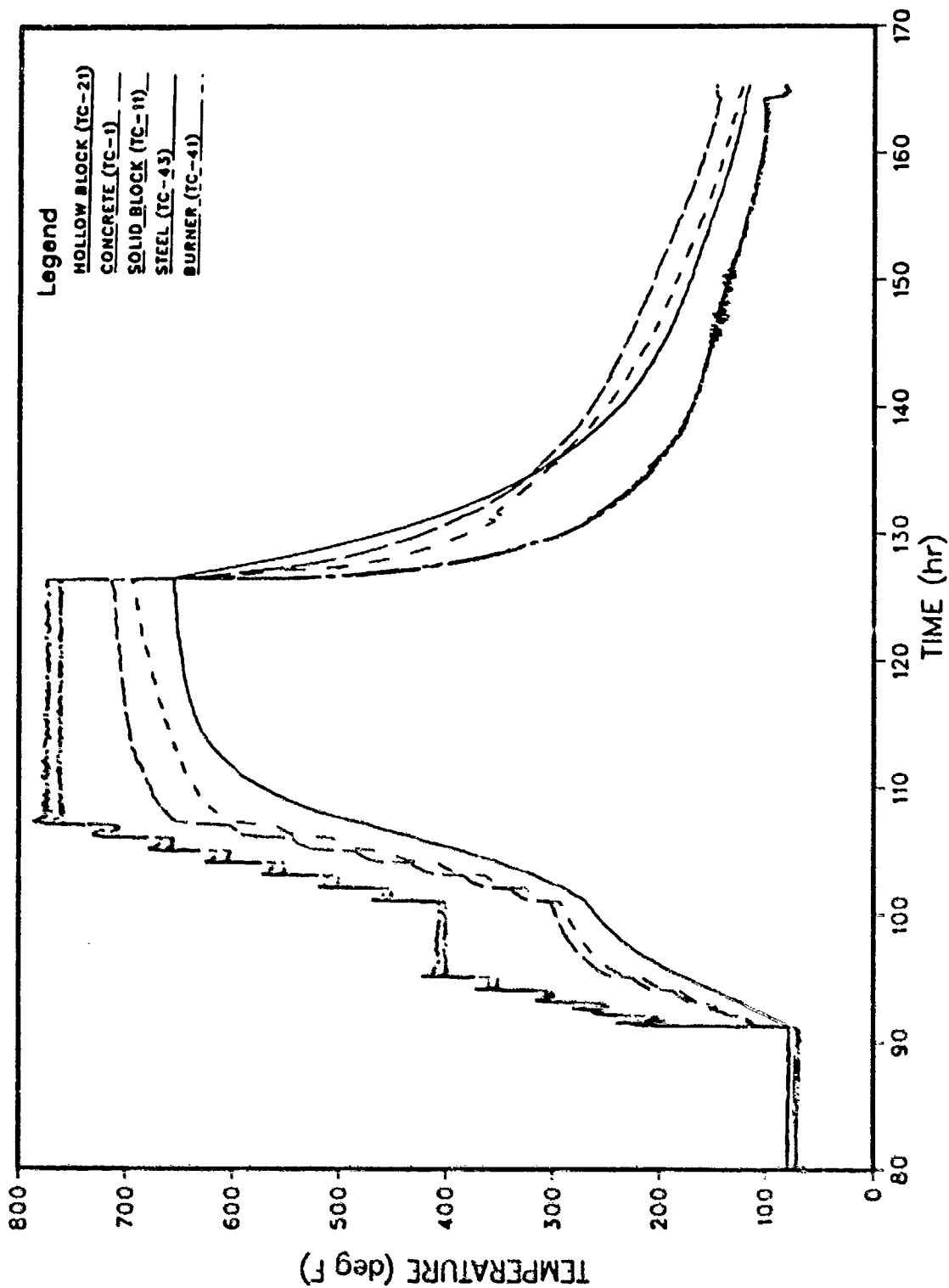


FIGURE 8-9. HEATUP PROFILE FOR INTERIOR WALL SURFACES

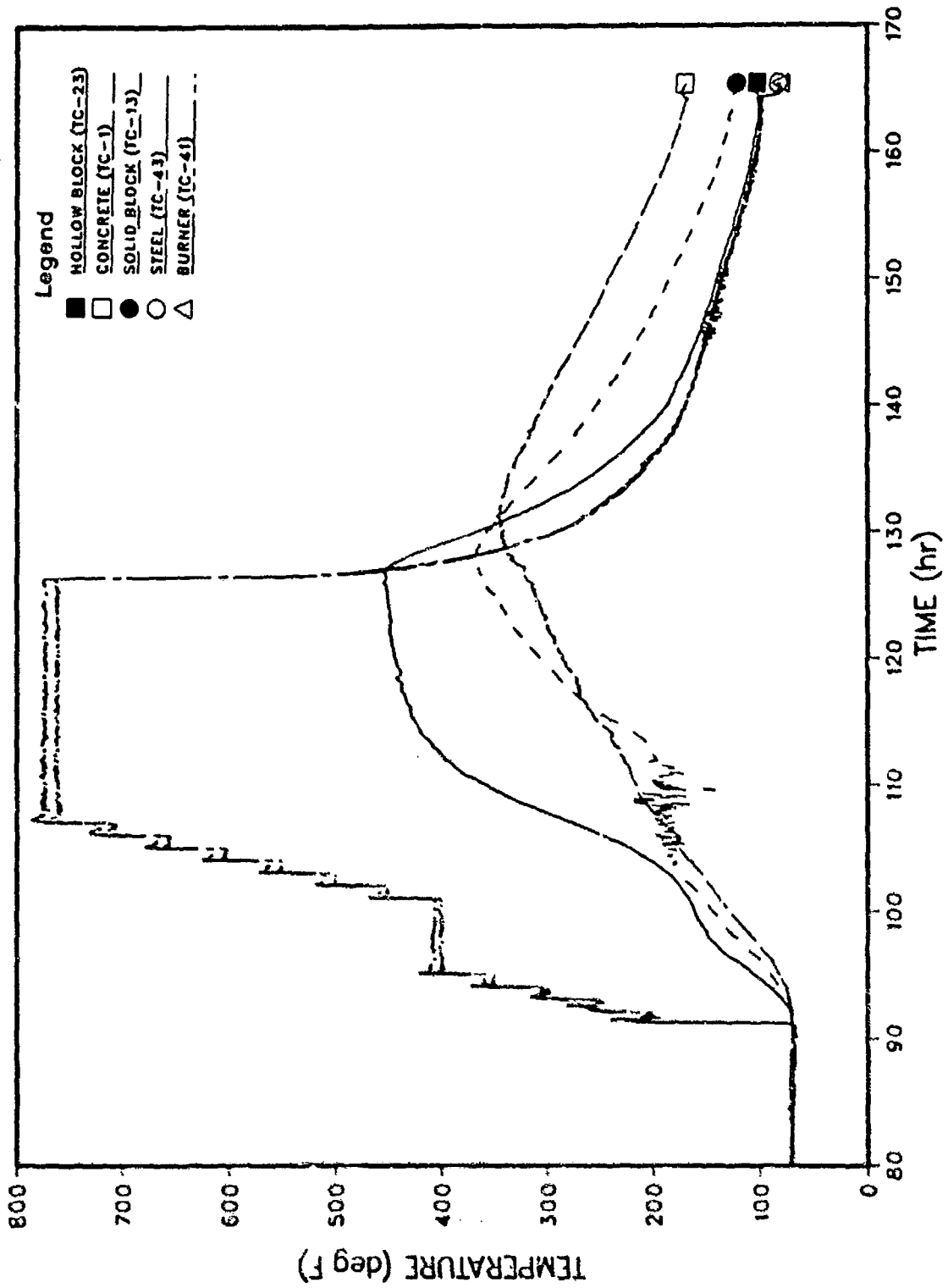


FIGURE 8-10. HEATUP PROFILE FOR HALFWAY UP EXTERIOR WALL SURFACES

temperature. The exterior or the hollow block wall heated up faster and hotter than both the poured concrete wall and the solid block wall. The different thermal characteristics of the building materials heated in this pilot test imply that building construction will be an important factor to the site-specific design of hot gas decontamination system field operations.

Hourly averages for all thermocouples are tabulated in Appendix F.

8.3 Air Analyses

8.3.1 ACAMS Readings

On July 17, 1987, 0900-1000, the test structure was spiked. The background reading before spiking of the test structure was 0.02 on the ACAMS, which is 0.02 times the time weighted average (TWA) value of 0.003 mg/m³. The ACAMS monitored the air in the test structure after it had passed through the exhaust but before the entry of the dilution air. The digital readouts (read by remote video camera) were updated every 8 minutes. After the spiking of the test structure, the ACAMS readings rose to greater than 20 times the 0.003 mg/m³ value, or approximately 0.060 mg/m³. The ACAMS was responding to the HD spike. By 0830 on July 20, 1987, approximately 0.5 hr before the startup of the burner, the ACAMS readings had decreased to 0.0007 mg/m³, or about one-fourth of the TWA (0.003 mg/m³).

Figure 8-11 is a graph of the ACAMS data collected during the major portion of the heatup phase of the test where most of the ACAMS activity took place. At 0900 on July 20, 1987, when the burner air blower was started, the ACAMS readings rose from 0.0007 to 0.0013 mg/m³. At 0922, the readings had risen to 0.002 mg/m³, and the exhaust temperature was 200°F. The ACAMS readings reached a maximum at 1022 of 0.013 mg/m³. The building exhaust temperature at this time was approximately 226°F. From that time until approximately 1700 on July 20, 1987, (the next 7 hours), the ACAMS readings

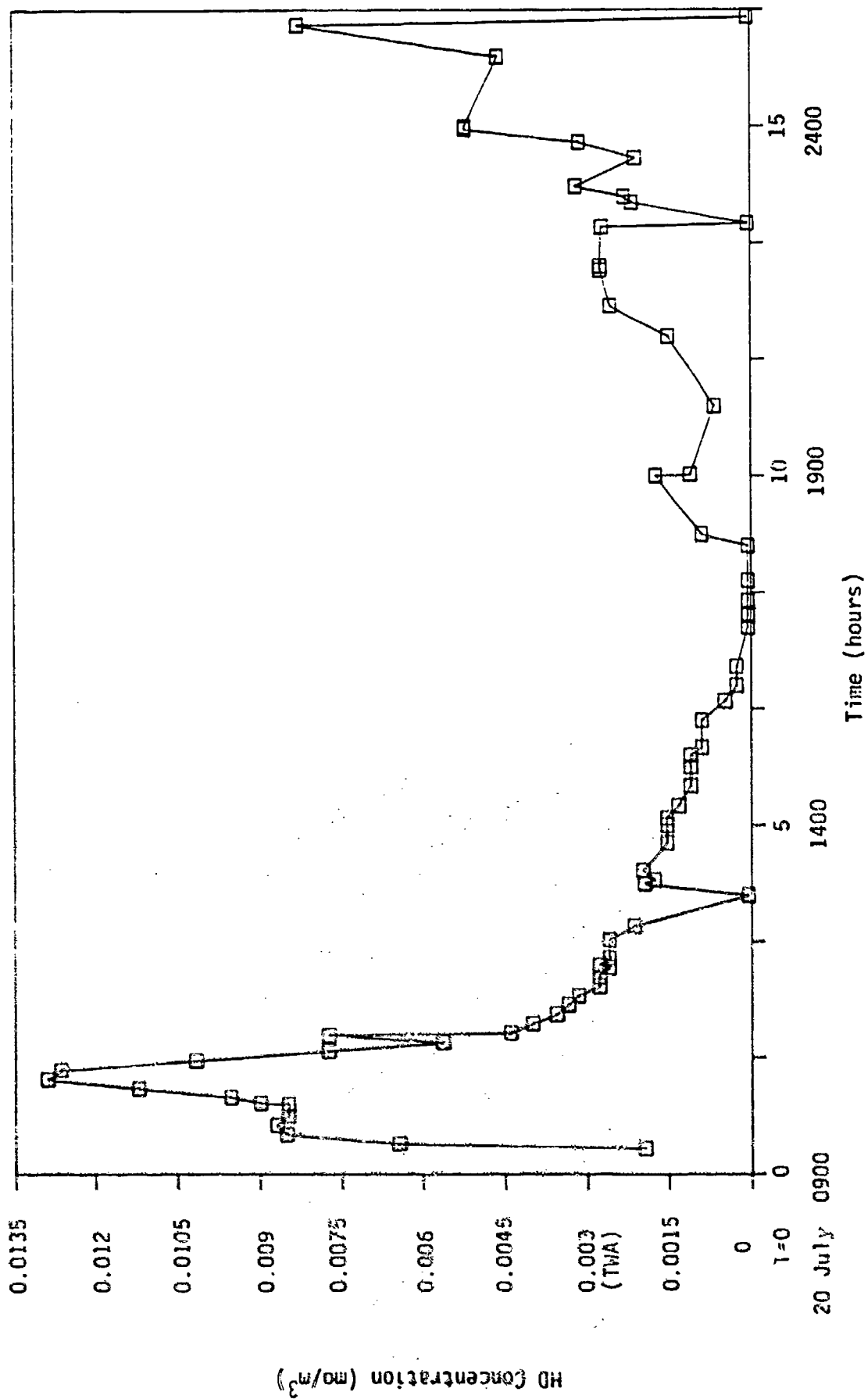


Figure 8-11. Concentrations of HD* in Test Structure Air as Measured by ACAMS (Real Time Monitor) During Heatup Phase of Pilot Test, 0900 July 20 through 0134 July 21, 1987.

*Includes HD and/or HD Breakdown Products

Zfell in a fairly smooth curve to the background level. The building exhaust temperature at 1700 was 374°F.

After 1800 on July 20, 1987, the readings began to rise and fall erratically during the remainder of the test (until 1000 on July 23, 1987). Some of the increases may be explained by an increase in temperature of the burner, but, for the most part, the readings were inexplicable. At 2400 on July 20, water was discovered in the ACAMS, which casts doubt on the readings after this time and may explain the erratic readings during the previous 6 hours. A water-cooled condenser had been installed in the line that sampled the exhaust stream to cool the air entering the ACAMS (and bubblers) to avoid damaging the instruments and evaporating the bubbler solvents. However, the water released from the concrete as it was heated and the water produced as the propane fuel was burned condensed in the sample lines.

8.3.2 Bubbler Analyses

Of the 12 bubbler stations serviced during the total operations, only 5 showed any values above the TWA of 0.003 mg/m³ during any time of the operations. These stations were as follows: Lower Door, Upper Door, East Exhaust, West Exhaust, and the Under Spill Area.

The Lower Door station graph is shown in Figure 8-12. This station drew air samples from inside the test structure near the floor at a location approximately four feet from a spiked floor spill area. The bubbler sample during the spiking operation showed a sharp, 30-fold increase above background, to approximately 0.060 mg/m³, then declined to the detection limit by about 48 hours after the spiking operation. The bubbler showed a positive reading (approximately 0.006 mg/m³) during the burner startup period and during the second six hour period (12 hours total). After this time, water produced by the dehydration of the concrete and from the burner entered the bubbler and the samples could not be analyzed. Water traps were eventually placed in the sample lines, but condensate may have trapped HD or HD breakdown products so that an accurate collection of contaminants would not be possible.

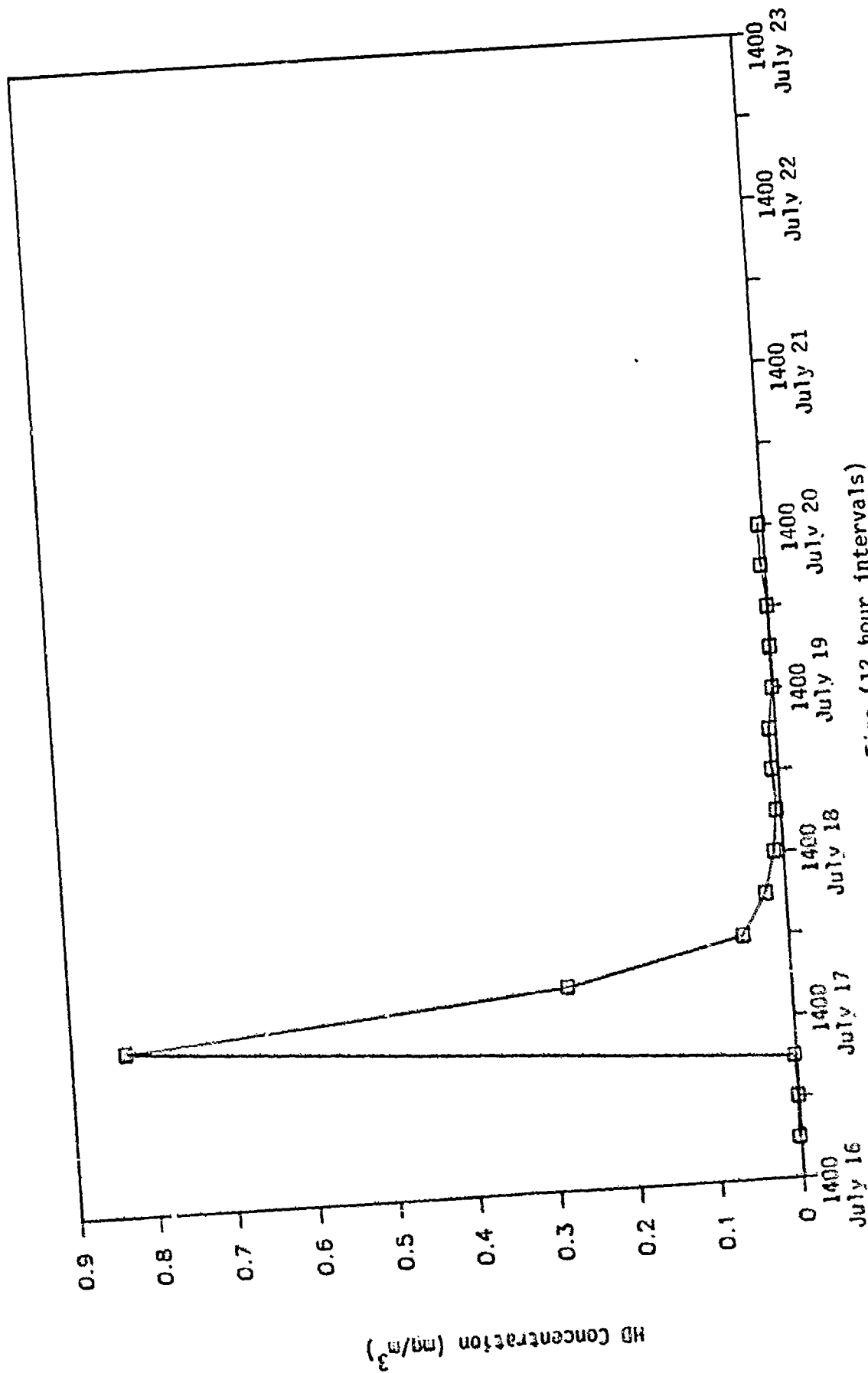


Figure 8-12. HD Concentration in Test Structure Air as Measured by 6-Hour
Bubble Samples, Lower Door Station, 1400 July 16 to 1400 July 23, 1987.

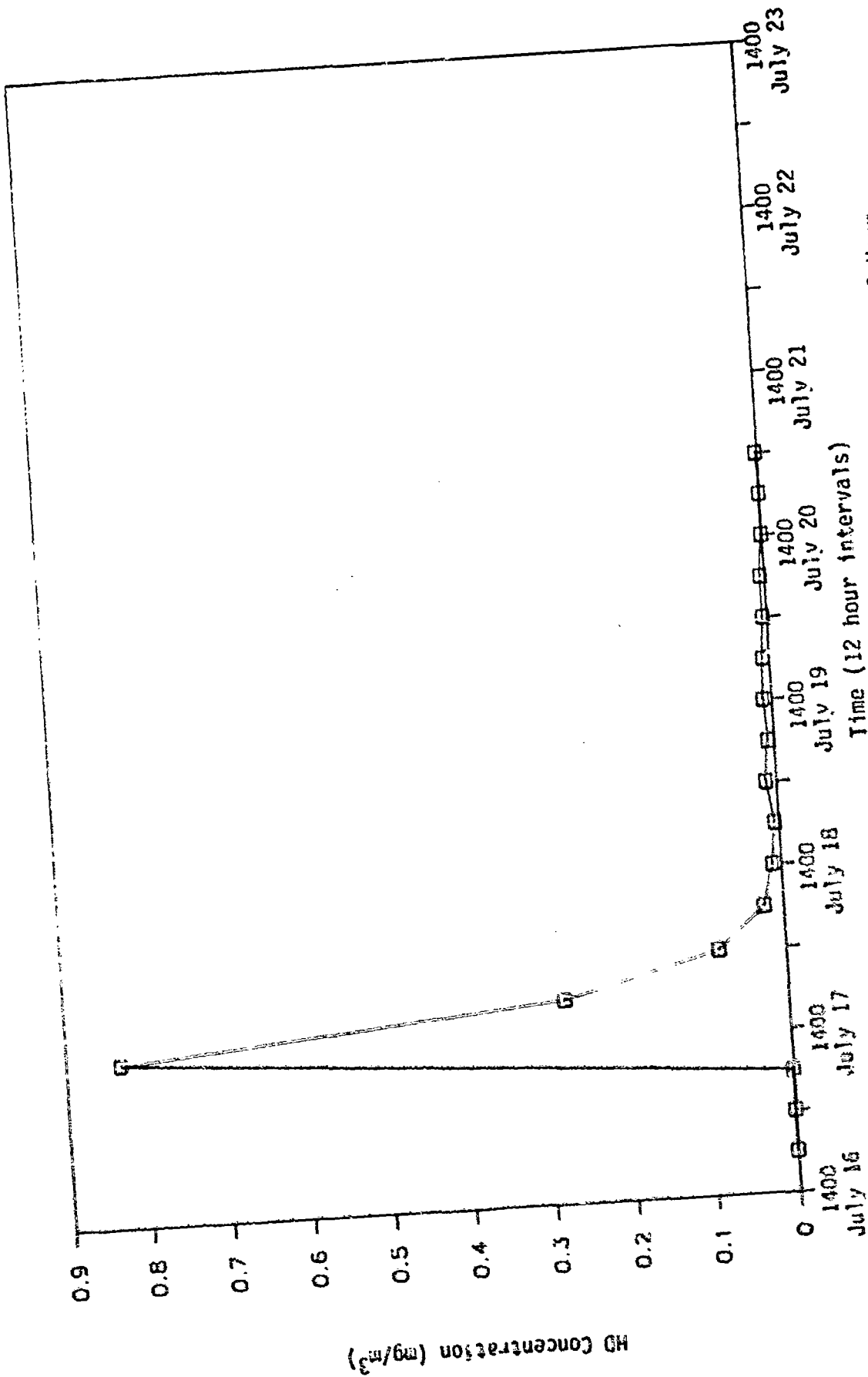


Figure 8-13. HD Concentration in Test Structure as Measured by 6-Hour Bubbler Samples. Upper Door Station, 1400 July 16 to 1400 July 23, 1987.

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100

Therefore, analytical values obtained for periods after the initial discovery of water in the bubbler were treated as suspect and are not plotted on the graph.

The Upper Door station was about two feet above the lower door location. The graph for that station (Figure 8-13) is similar to the one shown in the Lower Door station in that the concentrations during the spiking period were high and the subsequent concentrations decreased to almost the TWA by the time the burner was started. By the second six hours of the heatup phase, the samples were registering only background levels of HD or HD breakdown products. Water entered the bubbler during the 0800-1400 period of July 21, 1987, approximately 24 hours after the test began, rendering the rest of the data suspect.

The East Exhaust station sampled the exhaust of the test chamber before it was diluted with cooling air. The HD concentrations - time curve (Figure 8-14) showed a more subdued response, approximately a 7-fold decrease, to the spike than the Lower and Upper Door stations. This decrease, confirmed by the West Exhaust station results (see below), may be due to lack of mixing of the purge air entering the test structure and the air within the structure when the floor was spiked. The blower air entered the test structure by way of the damper near the top of the door, and left the test structure through the exhaust port next to it (see Figure 7-1). The negative pressure in the test structure may have caused the inlet air to exit without mixing fully with the test structure air. More thorough mixing of the test structure air probably occurred during the test because the heated air was added at the bottom of the door, not at the top next to the exhaust port.

The concentrations of HD in the bubbler samples of the East Exhaust station diminished to close to background by the start of the test and did not significantly change during the period of burner startup and the heatup phase. However, the period of 2000-0200 on July 21 & 22, 1987, approximately 35 hours after the test began and just after the cooldown phase had started, produced a bubbler sample concentration that had increased from background levels to

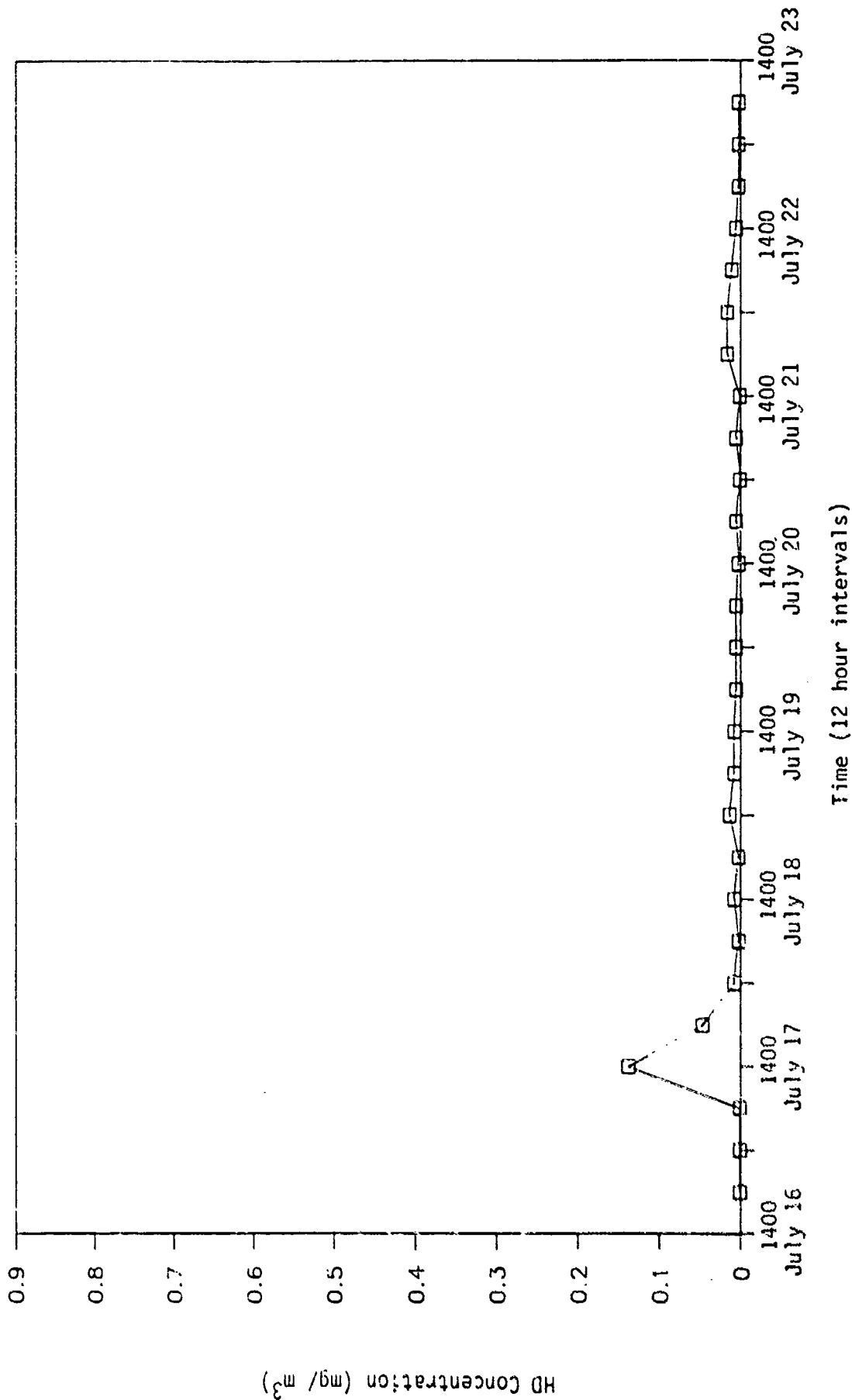


Figure 8-14. HD Concentration in Test Structure Air as Measured by 6-Hour Bubbler Samples, East Exhaust Station, 1400 July 16 to 1400 July 23, 1987.

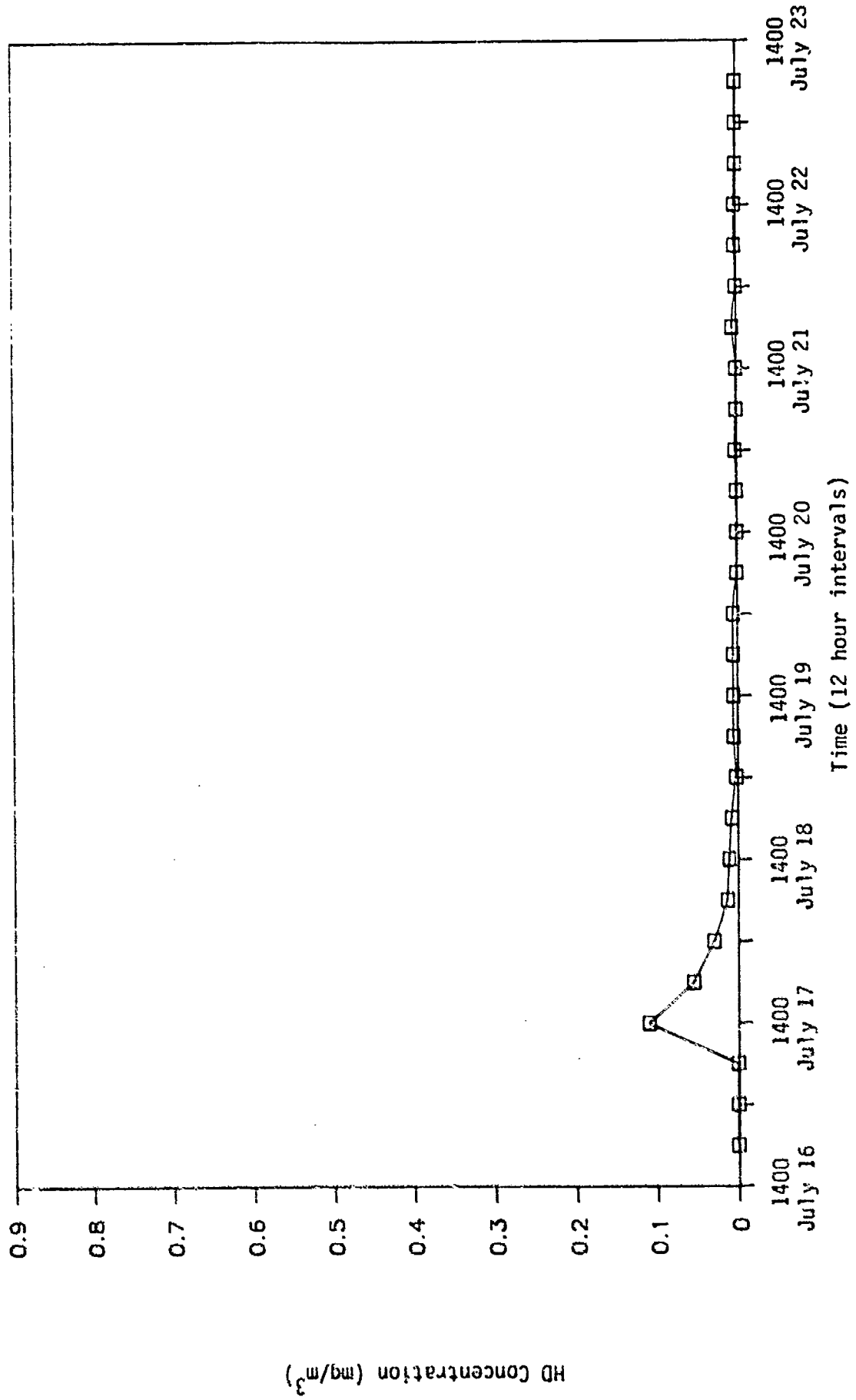


Figure 8-15. HD Concentration in Test Structure Air as Measured by 6-Hour Bubbler Samples, West Exhaust Station, 1400 July 16 to 1400 July 23, 1987.

0.018 mg/m³, or about six times the TWA. Because of these rather high and unexpected values, several samples of the bubbler solutions were analyzed by gas chromatography (GC) to determine the chemical composition of the contaminant. HD breakdown products were found, but no HD was discovered.

Sample results from the West Exhaust station showed a similar graph to the East Exhaust station (Figure 8-15). Samples collected during the first period of the cooldown phase increased from background to about two times the TWA like the East Exhaust station readings, but immediately declined to below the detectable limit for the remainder of the test. As with the ACAMS and the Lower and Upper Door bubbler stations, both the East and West Exhaust stations eventually filled with water and were unable to be analyzed.

The Under Spill Area station, which sampled air between the soil base and the concrete floor just under a floor spill area on the six inch thick floor, yielded a unique profile (Figure 8-16). During the 18 hours before the spiking operation took place, three consecutive bubbler samples showed concentrations of contaminant of two times the TWA (about 0.006 mg/m³). This value remained constant throughout the spiking operation, increased during a six hour period approximately 50 hours after the spike, and then diminished to background levels. Water entered the bubbler during the period of initial test heatup, so most of the remaining samples were not analyzed. This profile suggests that something from the soil used for the soil base of the test structure gave the bubbler a high background level. Bubbler samples collected during the last hours of the cooldown (after water traps in the line were installed) showed no detectable contamination from this station.

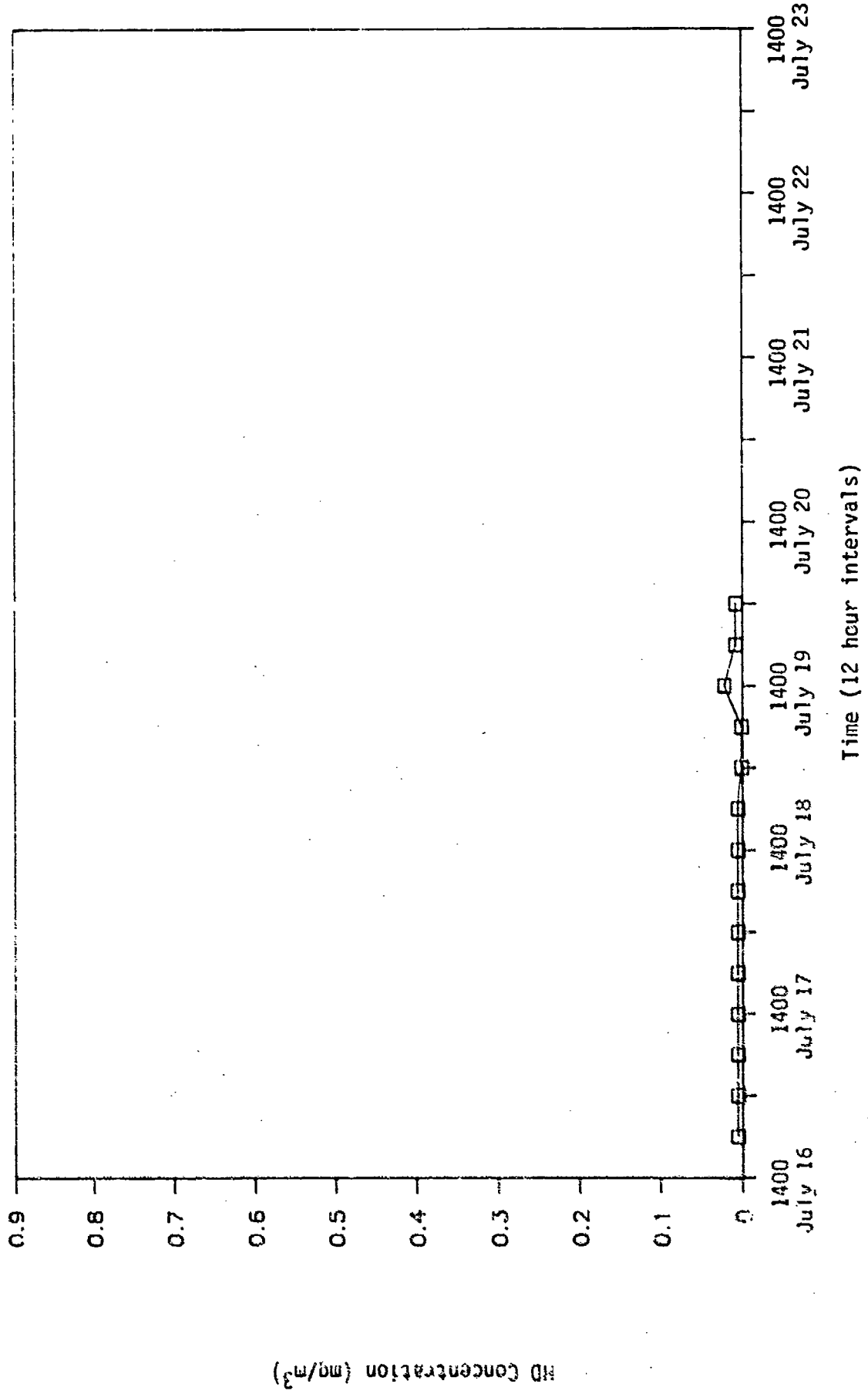


Figure 8-16. HD Concentration in Test Structure Air as Measured by 6-Hour Bubbler Samples, Under Spill Area Station, 1400 July 16 to 1400 July 23, 1987.

8.4 Sample Analysis

8.4.1 Verification Results

The final objective of Subtask 3, Analytical/Sampling Methods, as decided by USATHAMA, was to provide analytical methods that could be used to determine the effectiveness of the decontamination process to be evaluated. After the initiation of the program, there was a programmatic change that resulted in two significant alterations of Subtask 3 objectives. The major modification was the deemphasis of the certification testing that was originally proposed. The objective to verify methods for sampling and analysis of HD in building material matrices encountered in an existing structure was altered to verification of methods for sampling and analysis of HD in selected building materials used in the construction of a pilot test facility. Thus the major objective for this task became to affirm that coupons prepared from various materials and spiked with HD would indeed yield verifiable and quantifiable results.

The verification effort required two phases of experimentation: verification that existing published analytical methods, when available, were operationally sound, and development and verification of new sampling methods where no existing methods could be identified. This process was followed for both analytical and sampling techniques for HD contaminated painted and unpainted concrete, mild steel, and soils.

Experiments were conducted for determining the recovery efficiency of the methods using known spiked concentrations of HD from concrete, metal surfaces, and soils. A report documenting methods development and standard operating procedures for sampling and analysis of HD contaminated materials was completed prior to the initiation of the pilot testing (see Appendix B). The methods verified were for the following:

1. Solvent extraction method for HD from pulverized concrete.
2. Sampling procedure for the generation of pulverized concrete samples via drilling solid concrete structures.
3. Analytical screening technique for the detection of HD on the surface of unpainted concrete and steel.
4. Analytical technique for removal of HD from painted steel surfaces.
5. Solvent extraction method for HD in soil.

The method verification testing was conducted at both the Battelle facility and the Dugway Proving Ground facilities yielding the results in Table 8-1. The method for calculating the Theoretical Detection Limit is detailed in Appendix B.

The results of the method verification experiments for assessing the effectiveness of the hot gas decontamination process imply that the sampling and analytical techniques evaluated can detect HD from pulverized concrete, the surface of painted or unpainted concrete and steel, and in soils. The results of the sampling process developed for the collection of pulverized concrete samples at depth (see Appendix B) does not destroy HD as it samples. The methods for extracting HD from pulverized concrete or soil are suitable for quantitative analysis.

The results of surface swab techniques indicates that these methods are suitable only as a screening method to determine if HD is present on a surface. The results of the swab experiments indicate a large discrepancy in the recovery data. This discrepancy was determined to be the result of changes in the length of time after exposure to HD. The initial experiments conducted by Dugway were performed with a minimum length of time between exposure and swab. This was determined not to be representative of test conditions and the recovery experiments were repeated using an approximately

30 minute exposure period and results agreed well with the studies conducted at Battelle.

TABLE 8-1. RESULTS OF METHOD VERIFICATION STUDIES.

<u>Method</u>	<u>Battelle</u>	<u>Dugway #1</u>	<u>Dugway #2</u>	<u>TDL</u>
Pulverized Concrete Extraction	95%	91%	90% & 95%	.5 $\mu\text{g/g}$ concrete
Soil Extraction	96%	75%		.7 $\mu\text{g/g}$ soil
Concrete Swab	4.2%	67%	8%	10 mg/in^2
Painted Concrete Swab		100%	29%	2.76 mg/in^2
Steel, Swab		100%	80%	1.0 mg/in^2
Painted Steel Swab	80%	96%	69%	1.2 mg/in^2

* Recovery From Painted Concrete Surface
 TDL Theoretical Detection Limit

8.4.2 Results of the Post Test Samples

No HD was detected above the theoretical detection limits in any sample subjected to the hot gas decontamination process.

9.0 PILOT TEST ENVIRONMENTAL AND SAFETY REQUIREMENTS

The decision to conduct the pilot test at DPG in an environmentally controlled test chamber eliminated the need for obtaining environmental permits. It was possible to run the exhaust stream through a charcoal filter system, which controlled the release of possible pollutants to the atmosphere and eliminated the need for an afterburner.

Army safety regulations relevant to the pilot test at DPG were identified, collected and reviewed. Since DPG personnel actually ran the pilot test, DPG was responsible for on site safety during the spiking operation and the test operation. DPG policies and other applicable safety and surety regulations were followed by all participants.

10.0 CONCLUSIONS

The following conclusions from the pilot test results may be drawn:

1. The hot gas decontamination process, as demonstrated in this first large-scale pilot scale test, effectively removes chemical agent HD from painted and unpainted concrete and painted and unpainted steel. Low concentrations of HD that have been absorbed into concrete decrease to levels below theoretical detection limits available by the best available technology (500 ppb, see below) when subjected to the hot gas process. Use of the hot gas decontamination process initially results in the release of measurable concentrations of agent in the purge gas exhaust stream. The agent concentration decreases to below detectable levels as the process is continued.
2. The engineering aspects of implementing the hot gas system are well defined. Beyond site-specific modifications, no major changes to the hot gas burner system or setup are needed to make the design field-ready.
3. The laboratory methods verification study shows that the methods for extracting HD from pulverized concrete and soils work well and give verifiable, quantitative results. The theoretical detection limit for this method is approximately 0.5 ug of HD/g concrete or soil, or about 500 ppb.
4. Sampling concrete in the field to depths of about two inches from the surface is possible by pulverizing the concrete with an electric drill and collecting the fines for analysis. This method is appropriate for collecting samples of up to 10 grams of concrete.

5. Sampling the surfaces of unpainted and painted steel and concrete is possible by wiping a cotton swab on the surface and extracting the swab with solvent. However, this method is strictly a screening technique and should not be used for quantitative purposes because the recovery of HD is not statistically verifiable.

6. The hot gas process did not cause visible structural damage to a small concrete and steel building in which the gas temperature was held at 750°F for 19 hours, and the wall temperatures exceeded 300°F for one hour. However, no structural testing was conducted. Paint remains intact on concrete surfaces, but chalks and flakes off the steel surfaces.

11.0 RECOMMENDATIONS

This study of a hot gas decontamination process is the first time that building materials were heated while effluent HD was sampled and analyzed. The study was successful and, as would be expected, many lessons were learned that will be useful in making subsequent work in this area more effective. The nature of the results and conclusions as well as the practical experience gained from this study suggest some recommendations for future activities and further research.

During the pilot study large amounts of water were released from the concrete as it was heated. To avoid air sampling complications, future efforts should utilize specialized stack sampling apparatus designed for ACAMS application to furnace exhaust stacks. Off-gas sampling by solid sorbent technology should also be used in future testing.

In order for the hot gas decontamination technology to be used with a high rate of confidence, a sampling protocol will have to be developed that will reliably demonstrate the complete removal of agent from real buildings. Because of the fact that no residual agent concentration criteria exist for 5x decontamination, the limits eventually decided upon will undoubtedly be based upon the analytical capabilities developed to detect these residuals.

The ability to sample and analyze contaminated (or uncontaminated) building materials adequately needs to be developed and refined during the next phase of the program in order for the technology to gain credibility.

APPENDIX A
SUBTASK 2 - DESIGN REVIEW

SUBTASK II REPORT

DESIGN REVIEW

INTRODUCTION

The purpose of this subtask was to review the Report of Phase II of this effort as it relates to thermal decontamination of structures, particularly Appendix VI in Report AMX-TE-TR-85012. As part of this review, a tentative design was prepared for the equipment needed for the tests on building decontamination. The tentative design used the generic information in the Phase II and converts it into a specific design for the present program.

This subtask report was prepared early in the program and is intended to provide interface information to the other subtask leaders.

In the present program a small room will be built and contaminated with mustard. The hot gas concept will then be used to decontaminate the building. The test will be performed in the Defensive Testing Center at Dugway Proving Grounds.

The major difference in the decontamination requirements of this test and the generic buildings described in the previous report is that the size of the building to be decontaminated in this test is much smaller than the buildings listed in the report. Because of the smaller size, the heating rate of the building can be much faster than the rates suggested in the Phase II report.

DESIGN REVIEWCombustion Systems

The combustion systems recommended in the report are acceptable and adequate for the generic buildings they were designed to

decontaminate. The design did not address the needs for controls on the burners or for heat distribution within the building, important items for a specific design but beyond the scope of the Phase II design.

Temperature Limit

The maximum heating of the building was not addressed, nor was the rate of heating. Other parts of the report indicated concrete was not seriously damaged when heated to temperatures below 750°F. Overheating of the concrete could make the building unusable or in some cases, dangerous to enter following decontamination.

Heating Rate

Various parts of the building will heat at different rates. Thin walls and equipment within the building will heat to the gas temperature rapidly. The outside of thick walls and the floor may take days to heat to decontamination temperature. The Phase II report did not address methods of preventing overheating of parts of the building while other parts were still below decontamination temperature.

Afterburner

The afterburner design parameters have been standardized for agent destruction (2 seconds at 2,000°F) and the Phase II report follows these standards.

An afterburner is not required for the proposed experiments because the DTC at Dugway prevents agent release to the environment by the use of carbon filters.

Hot Gas Cooling

In the Phase II report, the three variations of the hot gas concept shown in Figure 1 were evaluated. The quencher concept, Figure 1C, was selected because it provided for redundant agent capture. The

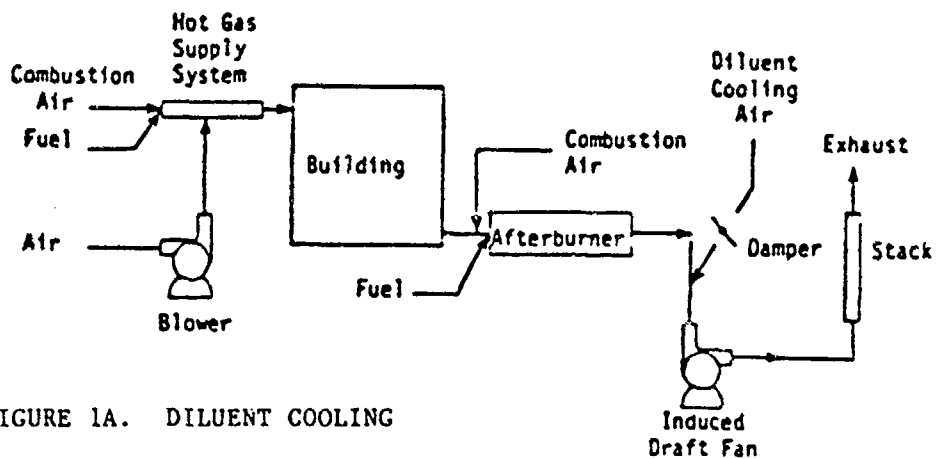


FIGURE 1A. DILUENT COOLING

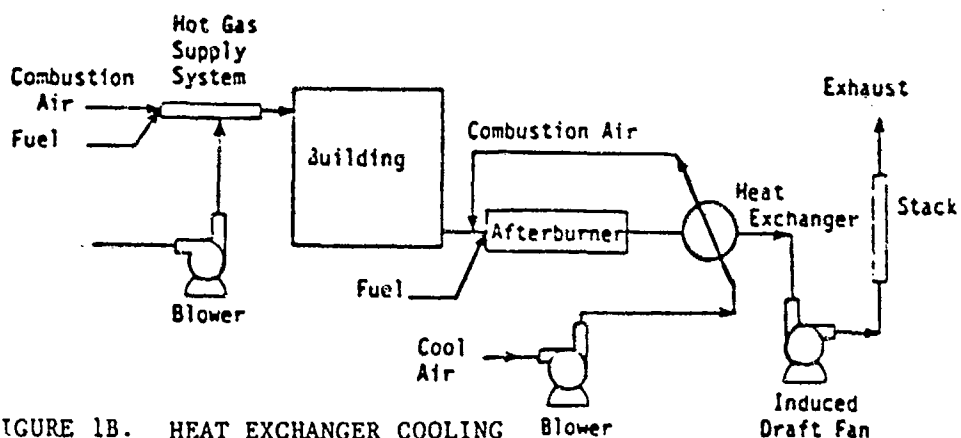


FIGURE 1B. HEAT EXCHANGER COOLING

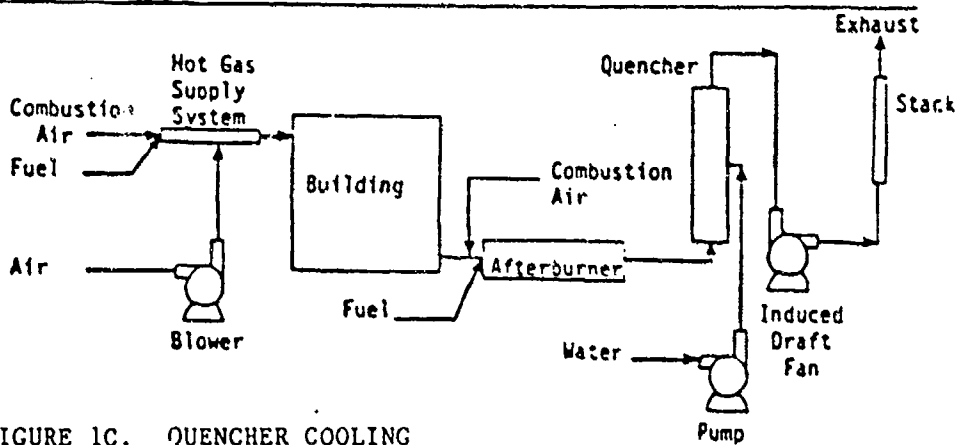


FIGURE 1C. QUENCHER COOLING

FIGURE 1. HOT GAS APPLICATION CONCEPTS SELECTED FOR ENGINEERING EVALUATION

cost differences among the three methods were less than the probable errors in the estimating techniques.

An afterburner will be needed when the hot gas decontamination method is used in the field. The hot gas from the afterburner requires cooling because an induced draft fan is necessary (for safety reasons) to maintain a negative pressure on the building being treated, as well as on the afterburner. Fans that operate at temperatures over about 600°F are very expensive. Therefore, the flue gas must be cooled below 600°F before it flows into the induced draft fan. The Phase II report evaluated three methods of cooling the flue gas: quencher cooling, diluent air cooling, and heat exchanger cooling. The quencher method of cooling was recommended. However, in this review, we reevaluated these three methods and found that dilution air is to be preferred. Dilution air is the simplest method and requires minimum capital cost. It requires the least equipment and it could be moved with minimum difficulty. Since the decontamination equipment will probably be moved to each building to be contaminated, portability is a major advantage. The quencher requires somewhat more equipment and also requires disposal of a liquid waste stream. Disposal of a liquid waste may be a problem at some sites and was not considered in the Phase II analysis. The heat exchange method of cooling requires considerably more equipment than air dilution cooling and, therefore, is rejected in this application. The preferred cooling method is application specific and this recommendation for air dilution cooling should not be extrapolated to other applications. The dilution air method of cooling is not usually preferred in applications where long term continuous operation is expected or in applications where equipment other than a low pressure induced draft fan is downstream of the cooler.

DESCRIPTION OF DEFENSIVE TEST CENTER

A structure built for the test will be decontaminated at Dugway Proving Ground in the Defensive Test Center (DTC). The DTC is in a remote area at least a mile from other structures. The DTC

is a building with inside dimensions of about 25 feet wide, 50 feet long and 20 feet high. A door at one end opens the full 25 feet and is 19 feet high. The entire building is lined with stainless steel. Allowable floor loading is not available. The floor is on grade and is stainless steel over concrete. Concrete floors on grade typically have an allowable loading of 650 pounds per square foot. The walls are stainless steel on the inside and galvanized steel on the outside. Six inches of foam (polystyrene) insulation separates the two walls.

The building is ventilated through HEPA-carbon filter systems in both the inlet and outlet. The outlet has a nominal capacity of 6,000 cfm and the inlet, 4,000 cfm. A control system balances the flow to maintain a predetermined negative pressure. The inlet filter is to prevent agent from escaping in the case of power failure. The inlet air can be temperature controlled between -40°F and +160°F. A 60 ton air conditioner is used to cool the building. Electricity is used for heating. In addition, a venturi scrubber can be used to remove agent from the chamber. Dugway personnel could not provide information on the flow rate. However, 12-inch ducting and a 4x9-inch venturi throat would indicate a flow of about 5,000 cfm. The exhaust is returned to the test chamber.

Rooms adjacent to the main room are used for equipment and controls. Many electrical and thermocouple leads penetrate the wall between the test chamber and the control room. The control room contains a computer used largely for data recording. Agent monitors (RTM and Morans) could be placed in this room.

The 500 KW power used by the test facility is supplied by a long power line. Two 100 KW portable generators are used as backup. Since we will not require refrigeration, additional generators probably will not be required.

Water is trucked to the site. Agent contaminated water must be trucked to CAMDS for incineration. Other water can be treated by Dugway and discharged.

Fuel is not available. Presumably, a tanker loaded with propane will be brought to the site and used during the test.

The elevation at Dugway is about 5,000 feet. Therefore, all gas flow calculations will require pressure correction.

Since Dugway will perform all operations using agent, Battelle personnel should not require protection; however, Dugway can furnish masks and clothing. Dugway's regulation on mustard is that if the concentration in air is less than 0.003 mg per cubic meter, the area can be entered without protection. If the concentration is between 0.003 and 0.5 mg per cubic meter, the area can be entered in Level A clothing. The time of entry is limited by heat stress rather than agent. If the concentration is above 0.5 mg per cubic meter, entry is prohibited. These limits may effect the method of contaminating our test structure.

Once the test structure is contaminated, it will have to be heated to 1,000°F to be 5X decontaminated.

Dugway has a chemical lab and has capability for sampling and analysis.

SERVICES REQUIRED

The major services required are electricity, fuel, compressed air and water.

The electric service is required during operation for the burner blower, for ignition of the burner and for controls. A 1 1/2 HP motor probably will be specified for the blower. The controls should take less than 5 KW. Construction will require welding and, therefore, about a 10 KW power source. The DTC building is equipped with a 500 KW source. During these tests the DTC equipment is expected to use less than 100 KW so no additional power should be required.

A fuel source is required to supply up to 25 lb/hr of propane. The total usage is expected to be about 500 pounds. However, 1,000 pounds should be on site at the beginning of a test unless a method of rapid resupply is available.

Compressed air is required for instrumentation. About 10 cfm at 100 PSI is required. If an instrument air supply is not available at the DTC, a supply can be purchased for about \$3,000 or perhaps rented.

Water is required for the venturi. About 100 pounds/hour will be evaporated during the test. In addition, an initial charge for the venturi system of several hundred gallons will be required.

TENTATIVE DESIGN

Two major items are required for the project, a building to be contaminated, and a method for decontaminating the building.

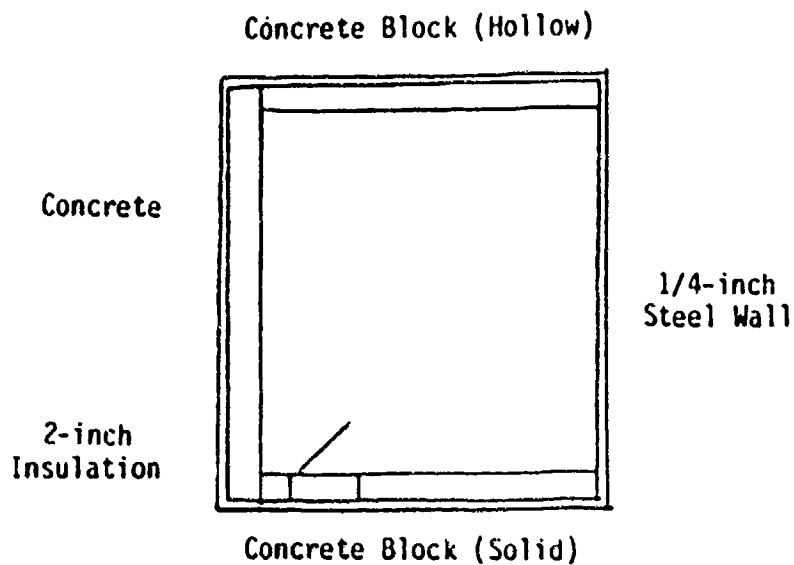
Building

The proposed structure is a 10-x 10-x 8-foot building with a steel door frame in one wall as sketched in Figure 2. Each wall of the building will be made of a different material, one of 12-inch thick concrete, one of solid cement block, one of hollow cement block, and one of 1/4-inch steel plate. The floor will be one-half 12-inch thick concrete and one-half 6-inch thick concrete. The entire structure will be mounted on a sand base. The top of the sand and the building floor will be 2 feet above the floor of the DTC. A steel door frame will be built into one of the concrete block walls. Penetrations for heating, cooling, and room exhaust will be through the steel wall or ceiling. The entire structure will be insulated with high temperature fiberglass insulation of at least a R-10 rating. Thermocouples for measuring heating rates will be located at a later design stage.

Heater Design

Figure 3 is a sketch of the floor and controls needed for heating the room. The inlet and outlet filters, as well as the venturi are part of the DTC. The room heater is a burner mounted on the wall of the test chamber. A high velocity nozzle is selected to stir the air in the room eliminating the need for heat distribution baffles. A thermocouple in the room actuates a control to modulate the fuel flow to the burner. A constant air flow to the burner will be used. The burner maximum capacity will be in the 500,000 Btu/hr range. Its

TOP VIEW



SIDE VIEW

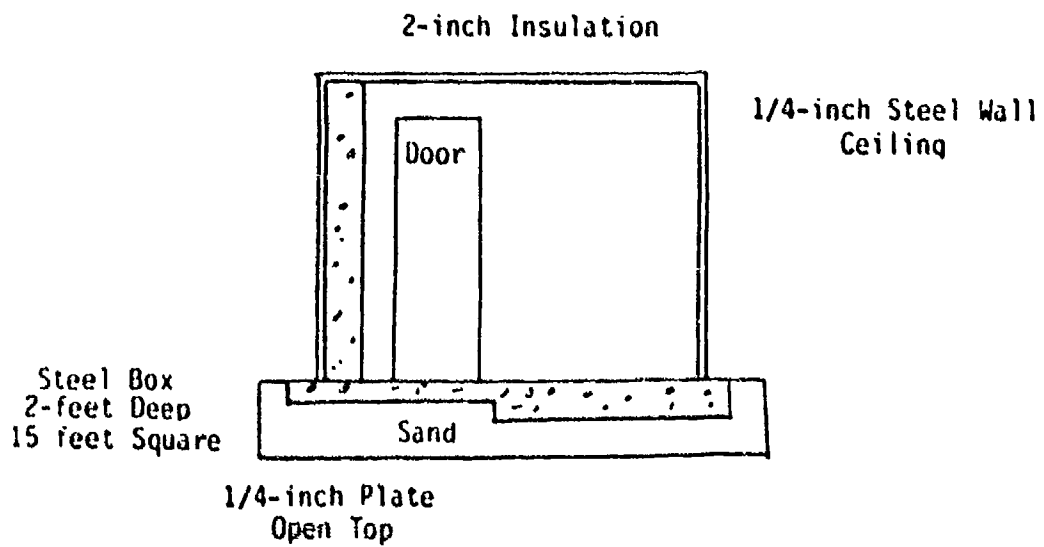


FIGURE 2. SKETCH OF TEST STRUCTURE

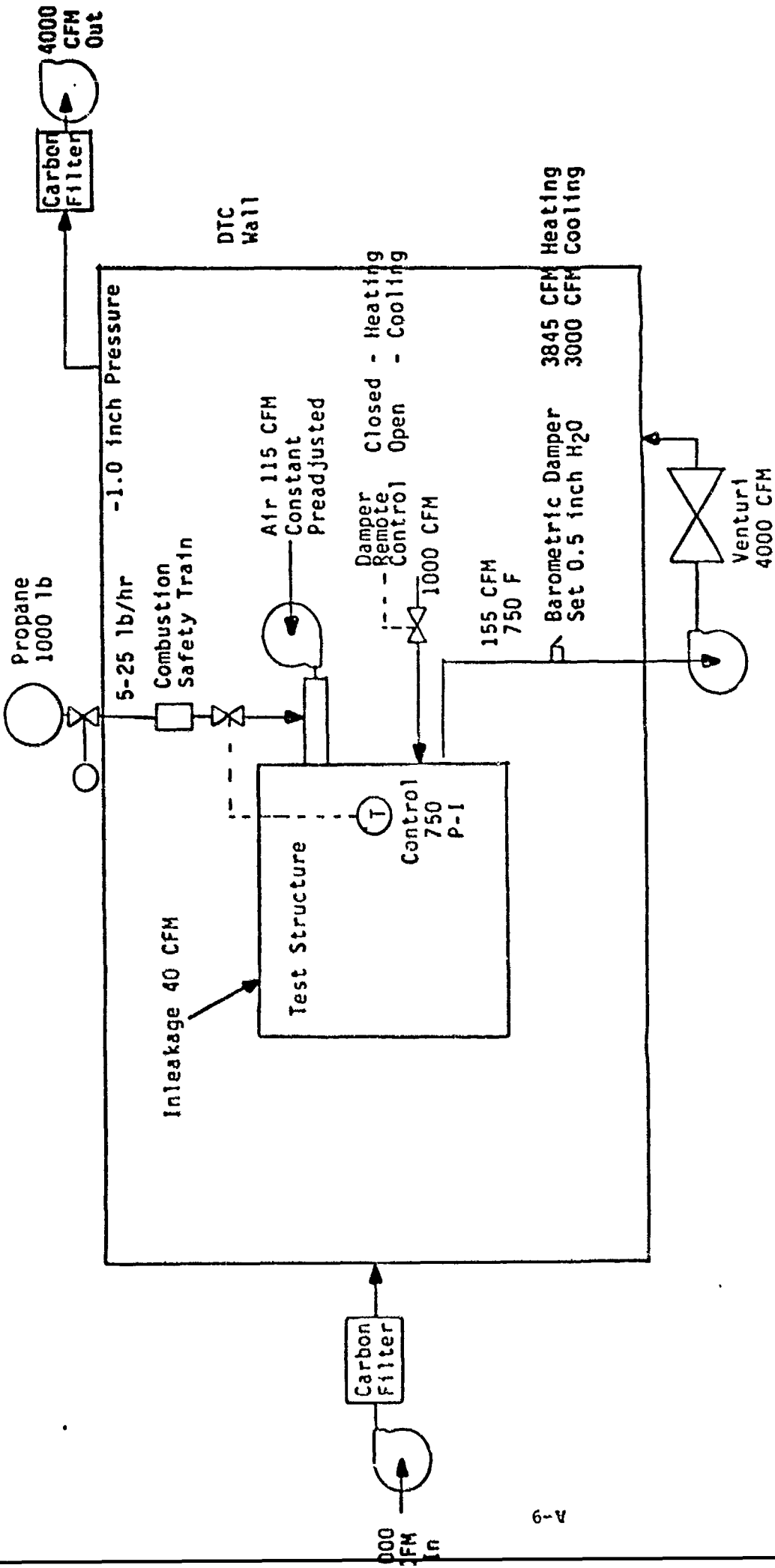


FIGURE 3. LINE DIAGRAM OF FLOWS AND CONTROLS

minimum capacity will be in the 100,000 Btu/hr range. In operation the temperature control setpoint will be ramped up to 750°F at a rate (100°F per hour) which should not damage the concrete and then controlled at 750°F until the outside walls and floor of the building reach 300°F. A 300°F temperature will be maintained for one hour and then the burner will be turned off. A damper to allow cool air to enter the test structure will be opened and the building cooled. The damper will be sized to allow about 1,000 cfm to enter the chamber, but the flow will not be otherwise controlled.

The major design parameters are:

Burner rating	- 500,000 Btu/hr
Burner Gas Velocity	- 500 ft/min at rating
Burner Excess Air	- Stoichiometric at max fire
Building Pressure	- 0.2 in. negative
Propane	- minimum 1000 lb on site
Cooling Air	- 1,000 cfm
Max Room Temperature	- 750°F
Min Outside Wall Temperature	- 300°F

The only design parameter that is critical is the 300°F on the outside wall, a temperature which must be maintained for one hour to assure decontamination. Most of the other parameters can vary by 25 to 50 percent without affecting equipment performance or test results.

Gas Cooler Design

The gas from the test chamber will be at 750°F, too hot for the fan in the venturi scrubber. However, for efficient operation the venturi needs a high air velocity and, therefore, a high flow rate. The air required for efficient venturi operation is much greater than needed for cooling. The dilution air will be controlled by a barometric damper which will be adjusted to maintain a negative pressure in the test chamber.

Equipment with Long Lead Time

The equipment required is all conventional and should be close to off-the-shelf. The major time required is expected to be for assembly and checkout. The concrete and mortar in the building should cure for at least 30 days with 90 days preferred.

WASTES GENERATED

The major waste generated will be the building following the test. While it is not expected to be contaminated, present rules require that it be heated to 1,000°F to assure agent destruction. This will not be done during the test. For decontamination it should be heated to 1,000°F and then buried. The structure will weigh about 30-40 tons. The outlet filters on the DTC may need changing following the tests.

Waste water will be generated in the venturi scrubber, from building washdown if needed and from personnel decontamination. The quantity generated should be small and base disposal should handle it.

The waste from sampling should also be small and it should be disposable at Dugway.

DATA REQUIREMENTS

The operating data requirements are of several types: control data, data to determine that the equipment is operating satisfactorily, and data to correlate with agent removal from the structure. These data will be temperature, pressure, and agent concentration data. Table 1 lists temperature data required. Table 2 lists pressure data required. Table 3 lists flow data required. Table 4 lists agent concentration data required. Table 5 lists miscellaneous data. These data are not expected to change rapidly. If recorded manually they should be taken hourly. If computer recorded, they should be taken at about 10 minute

TABLE 1. TEMPERATURE DATA REQUIRED

Location	Function
Inside Room	Control Burner
Inside Concrete Wall	Agent Removal
Middle Concrete Wall	Agent Removal
Outside Concrete Wall	End Test
Inside Solid Block Wall	Agent Removal
Middle Solid Block Wall	Agent Removal
Outside Solid Block Wall	Agent Removal
Inside Hollow Block Wall	Agent Removal
Middle Hollow Block Wall	Agent Removal
Outside Hollow Block Wall	Agent Removal
Steel Wall	Agent Removal
Steel Ceiling	Agent Removal
Inside 6-inch Floor	Agent Removal
Outside 6-inch Floor	Agent Removal
Inside 12-inch Floor	Agent Removal
Middle 12-inch Floor	Agent Removal
Outside 12-inch Floor	Agent Removal
Bottom Sand	Agent Removal
DTC Room	Flow Calculations
Duct from Test Room	Flow Calculations
Duct to Scrubber	Flow Calculations
Duct from Scrubber	Flow Calculations

TABLE 2. PRESSURE DATA REQUIRED

Barometric Pressure
Pressure in DTC
Pressure in Test Room
Pressure in Blower Fan Outlet
Pressure in Propane Tank
Pressure in Venturi Blower Outlet
Pressure Drop DTC Inlet Filter
Pressure Drop DTC Outlet Filter

TABLE 3. FLOW DATA

Air	- Burner Blower
Propane	- Burner Blower
Air	- From Test Chamber
Air	- Cooling--To Test Chamber

TABLE 4. AGENT CONCENTRATION

Test Chamber - Gas Outlet
DTC Room

TABLE 5. MISCELLANEOUS DATA

Weight - Propane in Tank
Level - Fluid in Venturi Storage Tank

intervals. Data on propane level, venturi water level, test chamber temperature, and DTC pressure should be manually recorded to assure that the operators are monitoring the process.

HEAT TRANSFER ANALYSIS

The heat transfer analysis was made on a preliminary basis to determine the equilibrium temperature in the walls and floor and to determine the time needed to heat the outside of the walls and floor to above 300°F. In each case the gas temperature in the room is assumed to be controlled at 750°F, a temperature limit imposed because higher temperatures significantly damage the concrete. In addition it is assumed that the outside of the sand under the building and the air in the DTC are maintained at 80°F. The gas-solid heat transfer coefficient is assumed 3.0 Btu/hr ft²°F both inside and outside of the building. The insulation is assumed to have a R-10 value.

Equilibrium Temperatures

Table 6 lists the equilibrium temperatures in the structure.

Transient Temperature

Table 7 lists the time required for the entire wall or floor to reach 300°F. The time required to heat the outside of the wall to 300°F was estimated using a Hottel chart. This method assumes the insulation has zero conductivity. The temperature on the outside of the floor was estimated using a Gurney-Lurie chart and assuming the sand has the same properties as concrete. This assumption should result in a time to heat the floor longer than actually required.

As seen from the Table 7, heating the floor takes longer than any other surface. In addition, the time to heat is sensitive to the soil underneath it. If the soil has a high thermal conductivity

TABLE 6. EQUILIBRIUM TEMPERATURE IN STRUCTURE, °F

	Gas	Inside Wall	Outside Wall	Outside Insulation
Ceiling	750	730	730	100
Steel Wall	750	730	730	100
Concrete Wall	750	733	606	97
Solid block Wall	750	732	641	98
Hollow Block Wall	750	732	626	98
Floor, 12-inch	750	722	415	80

TABLE 7. TIME REQUIRED TO HEAT OUTSIDE OF STRUCTURE TO 300°F

Surface	Time, hr.
Steel Ceiling	0.25
Steel Wall	0.25
12-inch Concrete Wall	16
8-inch Concrete Block Wall	8
Hollow Concrete Block Wall	2
12-inch Floor	56

typical of wet soils, the floor may never reach temperature. If the soil is a good thermal insulator, the floor could reach temperature in less than 24 hours.

Burner Duty

The heat required of the burner is the sum of the heat lost through the test structure walls and in the flue gas from the building. Assuming 150 SCFM of flue gas is exhausted, the heat in the flue gas is about 115,000 Btu/hr. This allows for 40 SCFM of inleakage air. The building must be very tight to limit the inleakage to 40 SCFM. Each additional SCFM of inleakage will increase the heat required by about 750 Btu/hr. At equilibrium, the heat loss through the building is about 30,000 Btu/hr. The heat stored in the building is about 4,000,000 Btu. To heat the structure in about 12 hours, a 500,000 Btu/hr burner is required.

Afterburner

The afterburner shown in the Phase II design is not needed because of the venturi and carbon filter agent removal system built into the DTC. The maximum burner output is 500,000 Btu/hr. With 4,000 cfm ventilation this heat input would result in a temperature rise of about 110°F in the DTC. An afterburner would be needed if the heat release into the DTC caused excessive heating of the DTC. The DTC has a 60 ton air conditioner which could be used for cooling if needed. For the type of test needed, a heat release of about 1,000,000 Btu per hour could be tolerated when using the air conditioner or about 300,000 Btu/hr without the air conditioner. For greater heat releases an afterburner would be required and the exhaust from the afterburner would be exhausted directly to outside of the DTC.

Venturi

Because of the wet venturi scrubber, water will evaporate and the temperature should not rise more than 10-20°F with a 500,000

Btu/hr input. Also during the heating cycle only about 150,000 Btu/hr is released into the room and the rest of the energy goes into heating the test structure. This heat release should be completely controlled by evaporative cooling in the venturi. At the start of the cooling cycle the heat release is on the order of 500,000 Btu/hr. This heat release rate would last for less than an hour. During this part of the cycle, the DTC temperature would rise, or if desired, it could be controlled using the DTC refrigeration system.

APPENDIX B
SUBTASK 3 - REPORT ON THE DEVELOPMENT
OF ANALYTICAL METHODS FOR DETERMINATION
OF HD CONTAMINATION IN OR ON
BUILDING MATERIALS

TECHNICAL REPORT

on

**THE DEVELOPMENT OF ANALYTICAL METHODS FOR DETERMINATION OF HD
CONTAMINATION IN OR ON BUILDING MATERIALS**

to

**US ARMY TOXIC AND HAZARDOUS
MATERIALS AGENCY**

August 15, 1986

by

T.L. Hayes

**NOVEL PROCESSES
CONTRACT NO. DAAA15-86-D-0001
TASK NO. 1, SUBTASK 3**

**BATTELLE
Columbus Division
505 King Avenue
Columbus, Ohio 43201-2693**

INTRODUCTION

Since World War II, the U.S. Army has been involved with the production, testing, and demilitarization of chemical agents. As a result of these activities, Government facilities including a variety of process equipment and permanent structures were contaminated with Chemical Surety Materiel (CSM). When the U.S. Army stopped production of chemical agents, these facilities were decontaminated using existing technology and put on an inactive status. These structures are presently candidates for excessing actions. This technical report discusses the experimental approach followed to develop analytical procedures to sample structures that have been contaminated during previous agent activities. The experimental process was conducted as five individual subtasks. These subtasks were:

- * Method development of a solvent extraction process for HD in pulverized concrete.
- * Method development of an analytical procedure for HD in a solid concrete matrix.
- * Method development of an analytical technique for the removal of HD from concrete surfaces.
- * Method development of an analytical technique for the removal of HD on painted steel surfaces.
- * Method development of a solvent extraction process for HD in soil.

A series of tests were designed within each subtask to evaluate the method of sampling and the proposed analytical method to be developed. The data enclosed were generated using the experimental approach described in each subtask and are the validation data for each of the proposed test methods.

In addition to validation data for the analytical methods, the method of sampling used in the laboratory is discussed to provide a basis for the development of the actual field sampling methods. The sampling methods evaluated in the laboratory were designed so they could be readily adapted to field sampling with little or no modification. In addition, the sampling methods were selected in anticipation of the types of samples necessary for the documentation of the decontamination process testing to be conducted at Dugway Proving Ground. The testing to be conducted at Dugway will involve sampling of a structure designed to evaluate the hot-gas decontamination process.

This study was performed for the U. S. Army Toxic and Hazardous Materials Agency under contract No. DAAA15-86-D-0001.

OBJECTIVE

The objective of this study was to develop and test analytical methods to be used in conjunction with surface and subsurface sampling methods of concrete, surface sampling methods of painted metal, and solvent extraction methods for HD within soil. The analytical procedures developed were designed so they could be used to perform an evaluation of the hot-gas decontamination process for HD at Dugway Proving Ground.

EXPERIMENTAL

The experimental effort, described within this technical report, was performed using laboratory-scale operations and neat HD as the testing CSM. The effort was conducted to determine the analytical recovery of the methods for HD under similar sampling conditions as those anticipated for field use and to examine sampling and analysis artifacts that could present a problem in the analysis of these collected samples. The recovery for each method was evaluated on a minimum of two days and the data combined to establish a method recovery and theoretical detection limit. The analytical instrumentation used for these tests was calibrated daily using the chromatographic conditions given in Table 1.

Table 1. Gas Chromatographic Conditions for HD Analysis

Gas Chromatograph: Hewlett Packard Model 5880A
Detector: Flame Photometric Detector equipped with a
393 nm filter for sulfur analysis
Column: SE-54 25 m fused silica column X 0.22 mm I.D.
2 um film thickness
Carrier Gas: Helium
Carrier Velocity: 28 cm/min.
Temperature Program: 50 C to 150 C programed at 16 C/min.
Injector Temp.: 200 C
Detector Temp.: 200 C
Transfer Line Temp.: 200 C
Injection Volume: 2 uL, splitless injection

The experimental protocols followed are attached in appendix A. give specific details followed for each experiment. A summary of each effort is presented in the following sections.

1. Method Development of a Solvent Extraction Process for HD in Pulverized Concrete

The objective of the initial experiment performed was to evaluate the feasibility of using an organic solvent to extract HD from concrete. The concrete was pulverized for two reasons, one to simulate the samples generated by drilling, and second to facilitate the extraction process by increasing the extraction surface area. The concrete was pulverized, using an grinding mill, to a particle size that would pass through a #4 standard sieve (600 μ M). Individual 10 g samples of the concrete were weighed into 25 mL Erlenmeyer flasks with ground glass stoppers. The weighed concrete samples were spiked with a 0.1 mg/g loading of HD. The spiked samples were mixed for approximately 15 seconds by shaking and permitted to stand for 10 minutes before being extracted. To extract the samples, a 5 mL aliquot of chloroform was added to each flask and the flasks were again shaken by hand for 15 seconds. The samples were then permitted to stand for 10 minutes. After the 10-minute extraction period, a 2 mL aliquot of the hexane extract was removed, filtered, and placed in a GC vial for analysis. The samples were analyzed by GC/flame photometry using the conditions listed in Table 1. These experiments were conducted over a four-day period. The experiment on each of the four days consisted of a sample set of five spiked concrete replicates, a single blank concrete sample, and a recovery sample. The recovery sample was prepared by spiking 1 mg HD in 5 mL chloroform.

2. Method Development of an Analysis Procedure for HD in or on Solid Concrete

The second experimental effort was conducted following validation of the previous effort. This effort determine whether HD could be detected in contaminated concrete by a sampling and sample analysis process. Data collected indicate that HD is a persistent chemical agent and may be captured inside concrete structures contaminated by military operations. The experimental effort developed a procedure to permit sampling of solid structures for the presence of residual HD. A major consideration for the sampling effort conducted in the laboratory was to develop a method that could be readily transferred to the field. The second consideration was that the sampling procedures examined produce a minimum amount of dust during the sampling process. This is a necessary requirement to ensure safety of the sampling personnel and to prevent the spread of possible contamination. A third consideration was to minimize the amount of structure dismantling or destruction needed in order to collect samples. A fourth consideration was to develop a

procedure that delivered the concrete in a pulverized state ready for extraction so additional sample preparation was not needed.

After considering the possible sampling alternatives, the decision to use a drilling technique was made based upon previous success using this technique for other compounds, and because the sampling technique satisfied the above considerations. The decision was made to use a 1/4 inch masonry drill bit instead of a larger one to reduce heat caused by drilling. The reduction in sample size generated due to the smaller drill size was resolved by making multiple samplings to collect enough concrete sample for extraction. The smaller drill size was selected due to a concern that the heat generated while using a larger drill may decompose the HD during the sampling process.

The laboratory concrete coupons were prepared using Portland cement as a 1 inch thick, 5 X 5 inch square coupon. The concrete samples were marked with a 1 inch square in the center of the 5 X 5 inch coupons prior to spiking. The 1 inch square area was then spiked with 63.5 mg (50 uL) of neat HD and allowed to stand for 30 minutes. The spiked coupons were then placed in a lucite box to prevent the drillings from being entrained in the fume hood exhaust. The marked sample area was then drilled in five different locations to a 1/4 inch depth using a drill press. The concrete drillings were collected by tapping the side of the lucite box and transferred to a 4-dram vial containing 10 mL of chloroform as the extraction solvent. The samples were shaken for at least 15 seconds to facilitate the extraction and allowed to stand for 10 minutes.

After the extraction period, a 1.5 mL of the sample extract was transferred to a GC vial for analysis, using the conditions described in Table 1. The sample vials were left open in a hood to permit evaporation of the hexane so that a concrete sample weight could be taken. The weight of the concrete dust collected was used to calculate the average HD contamination level in the samples. The effort was conducted on two separate days with five spiked replicates per day. In addition to the five spiked samples, five blank concrete samples were also processed to determine whether interferences would pose an analysis problem. A recovery sample was also prepared by spiking HD in hexane (on 1/4 the volume spiked on the coupon (12.5 uL) was used because only 1/4 the total spiked surface was extracted).

3. Method Development of an Analytical Technique for the Removal of HD from the Surface of Concrete

This experiment provided a method for removing and detecting residual HD on concrete surfaces by a non-invasive technique. The sampling method employed was a surface swab technique followed by GC analysis. The swab method has been successfully used as a screening method to evaluate surface contamination of other compounds and provides an excellent field testing method. To perform this task, a series of 5 X 5 inch square concrete

coupons were spiked with 95 mg (75 uL) of HD divided into 16 equally spaced drops of neat agent. The coupons were immediately swabbed in two perpendicular directions, using a single 2 X 2 inch square cotton gauze pad which had been previously extracted using a soxhlet extractor with hexane as the solvent. The cleaned gauze pad was saturated with hexane prior to swabbing the coupon surface. The gauze was handled by forceps during all of the sampling steps to prevent contamination after the solvent extraction clean-up of the gauze pads.

After the swabbing process was completed, the gauze pad was placed into 10 mL of hexane in a 4-dram vial. The vial was shaken to facilitate the extraction of the cotton swab. An aliquot was removed from the 10 mL extraction volume and placed into a GC vial for analysis, using conditions described in Table 1. This process was repeated on two days with five replicates per day of spiked coupons. In addition to the spiked coupons, five recovery samples were generated by spiking 95 mg of neat HD into a 4-dram vial containing 10 mL of hexane. Five swab blanks were also analyzed to determine whether interferences would pose an analytical problem. Blanks were generated by swabbing and analyzing a non-spiked concrete coupon as describe above for the spiked samples.

4. Method Development of an Analytical Technique for Removal of HD from Painted Steel Surfaces

This method provided a means for removing and detecting residual HD on painted metal surfaces. The sampling method employed was a surface swab technique followed by GC analysis. As previously stated, the swab method has been successfully used as a screening method to evaluate surface contamination of other compounds and provides a proven field sampling method. To perform this task, 5 X 5 inch square painted metal coupons were spiked with 16 equally spaced drops of neat agent. The coupons were swabbed in two perpendicular directions, using a single 2 X 2 inch square cotton gauze pad which was previously extracted using a soxhlet extractor with hexane as the solvent. The cleaned gauze pad was soaked in hexane prior to swabbing the coupon surface. The gauze was handled by forceps to prevent contamination after the clean-up extraction of the gauze pad.

After the swabbing process, the gauze was placed into 10 mL of hexane contained in a 4-dram vial. The vial was shaken to facilitate the extraction of the cotton swab. An aliquot was removed from the sample extract and placed into a GC vial for analysis, using conditions described in Table 1. This process was repeated on two days with five replicates of spiked coupons per day. In addition to the spiked coupons, five recovery samples were generated by spiking 95 mg of neat HD into a 4-dram vial containing 10 mL of hexane. Five swab blanks were also analyzed to determine if interferences would pose an analytical problem. The blanks were generated by swabbing and analyzing a

non-spiked painted metal coupon as described above for the spiked samples.

5. Method Development of a Solvent Extraction Process for HD in Soil

This experiment evaluated the feasibility of using an organic solvent to extract HD from soil. The soil was pulverized using a mortar and pestle to a homogeneous finely divided sample. The soil was then passed through a #4 standard sieve to remove any particles larger 600 μm . The sieved and blended soil was split into individual 10 g samples and placed in 25 mL Erlenmeyer flasks with ground glass stoppers. The soil was then spiked with a 0.1 mg/g loading of HD. The spiked samples were mixed for approximately 15 seconds by shaking and permitted to stand for 10 minutes before being extracted. To extract the samples, 5 mL of chloroform was added to each flask and the flasks were shaken by hand for 15 seconds. Samples were permitted to stand for 10 minutes prior to removing a 2 mL aliquot of the hexane extract. The 2 mL aliquot was filtered and placed in a GC vial for analysis. The samples were analyzed by GC/flame photometry using the conditions listed in Table 1. The experiment was conducted over a three-day period, using five spiked replicates, a single blank sample, and a recovery sample. The recovery sample was prepared by spiking 1 mg of neat HD in 5 mL of chloroform.

RESULTS

The results of the experiments described in the previous section are presented in the following tables. The data for each experiment include the daily experimental results, the average (\bar{X}), standard deviation (SD), and the calculated recovery for the method. In addition to the calculated recovery, a theoretical method detection limit and method relative standard deviation (% RSD) are also listed. The theoretical method detection limit includes the method recovery and the instrumental detection limit as indicated below:

Instrumental Detection Limit (IDL) = 0.5 $\mu\text{g/mL}$ by FPD

$$\text{Theoretical Detection Limit} = \frac{(\text{IDL}) \times (\text{sample volume})}{(\text{method \% recovery}) \times (\text{sample weight}) \times (\text{multiplied by } 100)}$$

The results of the solvent extraction process for HD in pulverized concrete (Method #1) are presented in Table 2. The percent recovery compares the HD concrete sample recovery (determined by using the extraction method and the measured result) to the measured recovery of HD for an identical volume of solvent in the absence of the concrete.

Table 2. Results of the Solvent Extraction Process for HD in Pulverized Concrete

Sample	Percent Recovery	
Concrete #1-6-18	96	
Concrete #2-6-18	93	$\bar{X} = 92$
Concrete #3-6-18	99	SD = 5.1
Concrete #4-6-18	88	%RSD = 5.5
Concrete #5-6-18	85	
Concrete Blank-6-18	BDL	
Concrete #1-6-19	87	
Concrete #2-6-19	86	$\bar{X} = 90$
Concrete #3-6-19	93	SD = 3.2
Concrete #4-6-19	89	%RSD = 3.5
Concrete #5-6-19	94	
Concrete Blank-6-19	BDL	
Concrete #1-6-20	97	
Concrete #2-6-20	100	$\bar{X} = 99$
Concrete #3-6-20	99	SD = 1.2
Concrete #4-6-20	100	%RSD = 1.2
Concrete #5-6-20	100	
Concrete Blank-6-20	BDL	
Concrete #1-6-24	94	
Concrete #2-6-24	99	$\bar{X} = 98$
Concrete #3-6-24	98	SD = 2.2
Concrete #4-6-24	100	%RSD = 2.3
Concrete #5-6-24	100	
Concrete Blank-6-24	BDL	

Method Recovery = 95 %

Method %RSD = 4.0

BDL = Below Detection Limit

Theoretical Method Detection Limit = 0.3 ug/g Concrete

The results of the concrete drill sampling study (Method #2) are presented in Table 3.

Table 3. Results of the Concrete Drill Sampling Study for HD

Sample	Total mg Recovered		Contamination level (mg HD/concrete)
DS-1-RECOVERY	13		
DS-1-7-28	14		5.4
DS-2-7-28	11	$\bar{X} = 14$	5.1
DS-3-7-28	10	SD = 4.3	3.4
DS-4-7-28	12	%RSD = 31	5.7
DS-5-7-28	22	106 % RECOVERY	9.0
DS-1-7-29	17		10
DS-2-7-29	9	$\bar{X} = 12$	5.7
DS-3-7-29	12	SD = 2.8	9.1
DS-4-7-29	10	%RSD = 22	4.0
DS-5-7-29	13	94 % RECOVERY	5.2

$\bar{X} = 6.3$ mg HD / g concrete

DS-1-BLANK	BDL
DS-2-BLANK	BDL
DS-3-BLANK	BDL
DS-4-BLANK	BDL
DS-5-BLANK	BDL

Method Recovery = 100 % Method %RSD = 29

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 5 mg/sq. inch of surface

The results of the concrete swab sampling study Method #3 are presented in Table 4.

Table 4. Results of the Concrete Swab Sampling Study

Sample	Total mg Recovered		
SS-1-7-28	5	$\bar{X} = 5.4$ SD= 1.4 %RSD= 25	6 % RECOVERY
SS-2-7-28	8		
SS-3-7-28	5		
SS-4-7-28	4		
SS-5-7-28	5		
SS-1-7-29	3	$\bar{X} = 3$ SD= 0.89 %RSD= 30	3 % RECOVERY
SS-2-7-29	4		
SS-3-7-29	2		
SS-4-7-29	2		
SS-5-7-29	4		
SS-1-BLANK	BDL		
SS-2-BLANK	BDL		
SS-3-BLANK	BDL		
SS-4-BLANK	BDL		
SS-5-BLANK	BDL		

Method Recovery = 4.2 % Method %RSD = 40

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 4.4 mg / sq. inch of surface

The results of the painted steel swab sampling study Method #4 are presented in Table 5.

Table 5. Results of the Painted Steel Swab Sampling Study for HD

Sample	Total mg Recovered		
SPS-1	63		
SPS-2	74	$\bar{X} = 71$	
SPS-3	79	SD = 6.8	75 % RECOVERY
SPS-4	62	%RSD = 10	
SPS-5	75		
SPS-6	77		
SPS-7	86	$\bar{X} = 82$	
SPS-8	84	SD = 3.3	86 % RECOVERY
SPS-9	83	%RSD = 4.0	
SPS-10	79		
SPS-Blank	BDL		
SPS-Blank	BDL		
SPS-Blank	BDL		
SPS-Blank	BDL		
SPS-Blank	BDL		

Method Recovery = 80 % Method %RSD = 10

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 0.25 mg/sq. inch of surface

The results of the solvent extraction process for HD in soil study Method #5 are presented in Table 6.

Table 6. Results of the Solvent Extraction Process for HD in Soil

Sample	Percent Recovery	
Soil #1-6-19	75	
Soil #2-6-19	89	\bar{X} = 88.2
Soil #3-6-19	95	SD = 6.9
Soil #4-6-19	93	$\%RSD$ = 7.8
Soil #5-6-19	89	
Soil Blank-6-19	BDL	
Soil #1-6-20	100	
Soil #2-6-20	100	\bar{X} = 100
Soil #3-6-20	100	SD = 0.0
Soil #4-6-20	100	$\%RSD$ = 0.0
Soil #5-6-20	100	
Soil Blank-6-20	BDL	
Soil #1-6-24	100	
Soil #2-6-24	100	\bar{X} = 99
Soil #3-6-24	100	SD = 1.5
Soil #4-6-24	99	$\%RSD$ = 1.6
Soil #5-6-24	96	
Soil Blank-6-24	BDL	

Method Recovery = 96 %

Method $\%RSD$ = 5.6

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 0.5 ug/g Soil

DISCUSSION

The results of the solvent extraction processes for both concrete and soil indicate that HD can be quantitatively extracted from these matrices and analyzed by gas chromatographic techniques. The results presented in Table 2 indicate that an expected recovery from concrete is 95 % at the 200 ug HD/g concrete spike level and with a instrumental detection limit of 0.5 ug HD/mL hexane. It is expected that 0.3 ug HD/g concrete could be detected using this method. In addition, the percent relative standard deviation over the four-day period of 4.0 % indicates the method has suitable reproducibility for a

quantitative analyses.

The results of the soil extraction presented in Table 6 indicate that a similar recovery of 96% is expected at a 200 ug HD/g soil spike level. Using the same instrumental detection limit of 0.5 ug HD/mL hexane, it is expected that a 0.5 ug HD/g soil could be detected using this method. The soil extraction method yields a slightly higher percent relative standard deviation (5.6 %) but still within the acceptable 10 % level for a GC method. Both of the above theoretical method detection limits are based on a 10 g sample and 5 mL extraction volume.

The results of the concrete drilling experiments presented in Table 3 indicate that a 100 % recovery is expected using this method of analysis at a concrete contamination level of 63.5 mg HD/sq. inch of surface area. The percent relative standard deviation for this method was 29 %. This level of variability is relatively high for a typical analytical method, however with the involved spiking and sampling process the value is appropriate. The method has been developed to determine if HD is present and is expected to be suitable for this measurement. The theoretical method detection limit was calculated as 5 mg/sq inch of surface, assuming a 0.5 ug/mL Instrumental Detection Limit (IDL). The validation data for weight of sample collected indicate the average concentration level of HD found in the concrete validation samples equalled 6.3 mg HD/g concrete. This concentration level data (g/g concrete) may be more appropriate for the field tests as the method of reporting the amount of HD measured for a concrete sample during the analytical process. The experimental approach of using a 1/4 inch masonry drill bit was very successful and posed no problems in the laboratory tests. The samples were easily handled and provided no analytical problems using this method. The theoretical detection limit for the drill dust concrete samples taken in the field should be the same as that for the pulverized concrete study of 1 ug HD/g concrete sampled (wt./wt. measurement) since a similar method recovery was observed.

The results of the concrete swab sampling study are presented in Table 4. The data indicates a low recovery can be expected from surface swab samples. These data verify that the concrete is extremely porous and absorbs the CSM immediately upon spiking the surface. The HD measured is that amount that did not penetrate the concrete in approximately 5 minutes (the time required for spiking). The test does show that, if HD is present on the surface, the swab technique of sampling will remove at least some of the HD from the surface for analysis. The blanks showed no interferences for this method and the method should transfer easily to the field for screening surface contamination levels. Based upon the data collected, at least 4.4 mg/sq inch must be present in a 5 X 5 sq inch sampling area to be detected with an expected percent relative standard deviation of at least 40%. This high level of variation (40 % RSD) precludes this method from being used as more than a screening method. The cotton swabs used for the sampling must be pre-extracted by soxhlet extraction prior to use to prevent interferences. The method as used in the laboratory should transfer easily to the

field for use as a screening method for HD contamination on surfaces of concrete.

The results of the painted steel swab sampling study are presented in Table 5. The data indicate a major increase in recovery and reduced level of variability over the same technique used on the porous concrete. This was expected since the metal does not present the physical characteristics of absorption toward the CSM demonstrated by concrete. It is expected that painted concrete (to reduce the porosity) will present similar recovery results. The swabbing of painted metal produced an 80 % recovery and a lower theoretical detection limit of 0.25 mg/sq. inch based on a 5 X 5 sq inch sampling surface. The percent relative standard deviation of 10% RSD represents a much improved method over that of the unpainted concrete samples. The detection limit can be reduced further by increasing the surface area swabbed. The analytical method presented no analytical interferences and should be easily transferred to the field application.

CONCLUSIONS

The methods evaluated for this study all appear field ready and should be able to generate samples capable of assessing the decontamination effectiveness of the hot-gas process. The methods for extracting HD from concrete or soil are suitable for quantitative validation. The surface swab techniques are more of a screening method and can be used to detect residual agent on machinery or building surfaces caused by spills or contamination due to the demilitarization processes that were performed. It should be noted that the HD contamination on untreated concrete has probably penetrated below the surface and the drill technique will prove to be a more feasible method of sampling. The drill technique, although limited in depth in the laboratory, should not present a problem in doing depth profiles through the concrete. The 1/4 inch drill size does not present a heating problem when 1/4 to 1/2 inch depths are drilled at appropriate intervals. No observations were made during the laboratory experiments that would prevent these methods from being used to sample the designated facility at Dugway Proving Ground.

APPENDIX A.

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Key words: HD
Gas Chromatography

Title: STANDARD OPERATING PROCEDURE
FOR ANALYSIS OF HD IN SOILS AND CONCRETE
(G-8875-0134)

Originated by: Timothy L. Hayes Date: 8/28/86

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Circulation List:

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HML

Record of Reviews:

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STANDARD OPERATING PROCEDURE
 FOR ANALYSIS OF HD IN SOILS AND CONCRETE

I. Statement of Work

Since World War II, the U.S. Army has been involved with the production, testing, and demilitarization of chemical agents. As a result of these activities, Government facilities including a variety of process equipment and permanent structures were contaminated with GB, VX, and HD. When the Army stopped production of chemical agents, these facilities were decontaminated and put on an inactive status and are candidates for excessing actions.

As part of this test program, a method of on-site building material sampling and CSN analysis must be developed and tested to determine the level of decontamination of the structure and the surrounding area.

A series of tests has been designed to test a method of sampling soil and pulverized concrete challenged with dilute HD. The samples obtained will then be analyzed for residual HD.

II. Objective

The objective of this study is to develop a sampling and analytical method to determine the presence of HD in solid matrices such as concrete or soil after they have been finely divided to allow for efficient extraction. This SOP is for the analysis of samples after collection using any sampling method that produces a finely divided concrete or soil sample.

III. Materials and Equipment Design

A. Materials

- | | | |
|----|---|-------------|
| 1. | Pulverized Concrete,
< #4 sieve size | as required |
| 2. | Pulverized soil,
< #4 sieve size | as required |
| 3. | Hexane (ACS),
glass distilled, reagent grade | as required |
| 4. | Chloroform (ACS), | as required |

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glass distilled, reagent grade

5.	Erlenmeyer Flask, 25 mL with ground-glass stopper	12 each
6.	Pasteur Pipets	as required
7.	GC Auto-sample Vials	as required
8.	4 Dram Vials	as required
9.	Drummond Positive Displacement Pipet 0 - 25 uL, 0 -100 uL	1 each.
10.	Hamilton Syringe, 10 uL, 500 uL	2 each
11.	Gelman Syringe Filters	as required
12.	Balance	1 each
13.	Volumetric, 10 mL	as required
14.	Alconox Solution, 5%	as required
15.	Distilled Water	as required
16.	Acetone	as required
17.	M17 Mask	1 / person
18.	Butyl Rubber Aprons	1 / person
19.	Latex Rubber Gloves	as required
20.	15 mil Butyl Rubber Gloves	as required
21.	Nitriles/Soft Cotes	as required
22.	Microwipes	as required
23.	Pyrex Baking Dish	as required
24.	Wash Bottle Containing Bleach	as required
25.	4-Liter Econ Bucket Containing Bleach	as required

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B. Apparatus Design

The only test apparatus required for these tests are the 25 mL Erlenmeyer flasks and the balance. All work, except analysis of the extract samples, will be conducted within a fume hood.

IV. Analytical Technique

A Hewlett Packard Model 5880A gas chromatograph or equivalent, equipped with either a Flame Photometric Detector with the 393 nm window for sulfur or an Electrolytic Conductivity Detector, and an appropriate data system and printer and/or plotter is required to perform this method.

A data system is interfaced to the GC to allow acquisition of the signal from the detector for the duration of the chromatographic program. The computer system is equipped with storage devices for saving the data from the GC runs. Computer software is used to integrate the area under chromatographic peaks for quantification. An automatic sample injector and its associated data system will allow unattended analysis of samples.

Parameters

Chromatographic Conditions:

Column:	0.530 um ID x 10 meters DB-5 column
Carrier:	Helium
Flow:	30 ml/min
Injection Volume:	3 ul
Column Temp:	approximately 130°C isothermal
Detector Temp:	200°C
Injector Temp:	200°C

V. Test Conditions and Matrix

- A. CEM: Next MD
- B. Temperature: Ambient - approximately 68°F
- C. Test Material: Concrete, Soil, & #4 Sieve
- D. Extraction Solvent: Chloroform

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E. Test Matrix:

Day 1

5 concrete samples spiked with
100 uL of 10 mg/mL HD solution

1 concrete blank/no HD spike

5 soil samples spiked with
100 uL of 10 mg/mL HD solution

1 soil blank/no HD spike

Day 2

Repeat Day 1 tests

Day 3

Repeat day 1 tests

VI. General Procedure

A. Concrete/Soil Extraction

The following procedure will be used to obtain extraction samples from both pulverized concrete and pulverized soil. Each day, 5 concrete and 5 soil samples will be run, along with 1 blank of each material.

The test will be conducted by placing 10 g of pulverized concrete/soil in a 25 mL Erlenmeyer flask. The concrete soil will then be challenged with 100 uL of a 10 mg/mL HD solution and the flask shaken by hand for approximately 15 seconds. The sample will be allowed to stand for 10 minutes. After that time period, 3 mL of chloroform will be added and the sample will again be shaken by hand for approximately 15 seconds. The sample will then be allowed to stand for another 10 minutes. Following that, an aliquot of the extract will be withdrawn from the flask, filtered, and placed in a GC auto-sample vial. The extract will then be analyzed for HD.

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B. Analytical Procedure

1. Sample Handling and Preservation

Upon receipt, the XCSM samples will be stored in a hood or secured container at 4°C until the time of analysis. The samples will be allowed to warm to room temperature prior to analysis. The samples must be analyzed within five days of collection. After analysis, the sample is returned to the hood or secured refrigerator in the event re-analysis is required. Otherwise, the sample is decontaminated prior to proper disposal. Standard solutions will be stored at 4 C from the start of the analyses and periodically analyzed to verify that noticeable decomposition does not occur upon extended storage.

2. GC Calibration

The instrument is initially calibrated with five standards. A calibration curve is made by plotting the peak areas versus concentration and a line is constructed using linear regression analysis. Daily calibration checks are executed by analyzing three standards of differing concentrations at the beginning of each day. The peak areas are compared against those obtained from the initial calibration. Recalibration with the five standards is required when the GC system has not been operated for one week or longer or when the peak areas for the calibration checks vary more than $\pm 10\%$ from the values obtained for the standards during the initial calibration.

3. Sample Analysis

Once the instrument has been properly calibrated, analysis of the samples can begin. The samples are analyzed by injecting 3 ul onto the GC column. As a minimum, a standard/control is run after every 6 samples during daily operation. Standard deviation is analyzed after every six samples and the day's analyses are completed. The peak areas obtained from analyzing the standards are compared against the initial calibration data and should agree within $\pm 10\%$. If this threshold is not met, the instrument is considered to be out of calibration and data for the samples are discarded. The system is recalibrated and the samples are then reanalyzed.

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4. GC Calculations

A calibration curve is generated by plotting the data obtained from the initial calibration. The concentration of HD in the standard (x-axis) is plotted versus the appropriate peak area (y-axis). A graph is plotted by subjecting the data to linear regression analysis.

The concentration of HD in the samples is determined by extrapolation of the peak areas to the concentration of HD along the x-axis. The concentration, in ug/mg, of HD in the original sample is given by:

$$\text{HD, ug/g} = \frac{A \times 5 \text{ ml}}{10 \text{ g concrete sample}}$$

where A = the amount of HD in the extract determined from GC analysis.

VII. Specific Procedure

A. 10 mg/mL HD Stock Solution

1. Mark a 4-dram vial with Teflon-lined cap appropriately.
2. Pipet 9.9 mL chloroform into 4-dram vial and seal with Teflon-lined cap.
3. Prepare for neat HD transfer in accordance with the procedures defined in SOP HML-001. The HD stock solution is made by pipetting 91.0 uL HD into the 9.9 mL chloroform in the 4-dram vial.

B. GC Calibration Standards

1. Mark 5 4-dram vials with Teflon-lined cap appropriately.
2. Prepare the following GC calibration standards:

100% - 200 ug/mL HD

75% - 150 ug/mL HD

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50% - 100 ug/mL HD

25% - 50 ug/mL HD

10% - 20 ug/mL HD

C. Concrete/Soil Sample Extraction Procedure

1. Obtain 12 25-mL Erlenmeyer flasks and mark appropriately.
2. Weigh 10 g of pulverized concrete or pulverized soil and place in flask. Repeat until 6 flasks contain concrete and 6 flasks contain soil.
3. Place the flasks containing concrete in the fume hood. Hood workers don protective equipment and the two-man rule is in effect. Obtain the 10 mg/mL HD stock solution.
4. Challenge the first flask containing concrete with 100 uL of the 10 mg/mL HD stock solution.
5. Stopper the flask and hand-shake the Erlenmeyer for approximately 15 seconds to cover the HD challenge. Set flask aside within the fume hood.
6. Repeat steps 4 and 5 for the next 4 flasks containing concrete.
7. Allow the flasks to stand for 10 minutes.
8. After the flasks have stood for 10 minutes, add 5 mL of chloroform to the first flask challenged with HD. Stopper the flask and hand-shake for approximately 15 seconds. Set flask aside within the fume hood. Allow flask to stand for 10 minutes.
9. Repeat step 8 for the remaining HD challenged flasks and the single concrete blank for a total of 6 flasks containing concrete.
10. Repeat steps 4 through 9 for the flasks containing soil.
11. Withdraw an aliquot of the extract with a pasteur pipet and place in a 500 uL syringe with a Gelman filter attached to the end. Filter the extract into a GC auto-sample vial and submit it for HD analysis.

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12. Repeat step 11 for the remaining concrete and soil samples.

13. Upon completion of the test run, decontaminate all equipment used in accordance with the procedures defined in SOP HML-10 and dispose of waste in accordance with SOP HML-11. Following decontamination, the Erlenmeyer flasks are to be washed in the following manner:

- a. Wash with 5% Alconox solution
- b. Rinse with hot water
- c. Rinse with distilled water
- d. Rinse with acetone.
- e. Air dry.

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APPENDIX B

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Key words: CONCRETE
HD
SURFACE SWAB
SUBSURFACE DRILLING
DECONTAMINATION

Title: STANDARD OPERATING PROCEDURE
FOR NOVEL PROCESSES CONCRETE SURFACE AND SUBSURFACE SAMPLING
DECONTAMINATION STUDY
G-8875-0134

Originated by: [Signature] Date: 8/28/86

Approved by: [Signature] Date: 8/29/86
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Record of Reviews:

Date	Reviewed by	Date	Reviewed by
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STANDARD OPERATING PROCEDURE
 FOR NOVEL PROCESSES CONCRETE SURFACE AND SUBSURFACE SAMPLING
 DECONTAMINATION STUDY

I. Statement of Work

Since World War II, the U.S. Army has been involved with the production, testing, and demilitarization of chemical agents. As a result of these activities, Government facilities including a variety of process equipment and permanent structures were contaminated with GB, VX, and HD. When the Army stopped production of chemical agents, these facilities were decontaminated and put on an inactive status and are candidates for excessing actions.

As part of this test program, a method of on-site building material sampling and CSM analysis must be developed and tested to determine the level of decontamination of the structure.

A series of tests has been designed to test a method of obtaining surface swab samples and subsurface drill samples of concrete challenged with neat HD. The samples obtained will then be analyzed for residual HD.

II. Objective

The objective of this study is to develop and test a method of surface and subsurface sampling to be used in the decontamination confirmation tests on existing Government buildings that were once contaminated with CSM.

III. Materials and Equipment Design

A. Materials

- | | | |
|----|---------------------------------|-----------|
| 1. | Concrete Test Coupons | as needed |
| | a. 5" X 5" X 1/4" - Swab Tests | |
| | b. 5" X 5" X 1" - Drill Tests | |
| 2. | Stainless Steel Coupons | as needed |
| | a. 5" X 5" X 1/8" | |
| 3. | Alkyd Painted Metal Coupons | as needed |
| | a. 5" X 5" X 1/8" | |
| 4. | Coupon Holder Box - Drill Tests | 5 each |

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5.	3/8" Power Drill Press with 1/4" tungsten carbide bit	1 each
6.	Coupon Templates	
	a. Subsurface Template	1 each
	b. Surface Template	1 each
7.	Solvents	
	a. Chloroform	as needed
	b. Hexane	as needed
8.	Red Marking Pens	as needed
9.	4-Dram Sample Vials	as needed
10.	Pyrex. Glass Baking Dishes	as needed
11.	Gauze Pads, 2" X 2" X 12 Ply	as needed
12.	Forceps	2 each
13.	GC Auto-injector Vials	as needed
14.	Pasteur Pipets	as needed
15.	Stop Watch	1 each
16.	Laboratory Notebook	1 each
17.	Hamilton Syringe with Stepper, 10 uL	1 each
18.	Hamilton Syringe, 1 mL	1 each
19.	Micro wipes	as needed
20.	Styrofoam Vial Holder	1 each
21.	Acid Brush	as needed
22.	Tape, Duct	as needed
23.	Soft-Cotes	as needed
24.	4-Liter Decon Bucket containing Bleach	1 each
25.	Metal Vial Holder	1 each
26.	Wash Bottle containing Bleach	1 each

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- | | |
|---|-------------|
| 27. Butyl Rubber Aprons | 1 / person. |
| 28. M17 Mask | 1 / person |
| 29. Latex Rubber Gloves | as needed |
| 30. 13 mil Butyl Rubber Gloves | as needed |
| 31. Balance | 1 each |
| 32. Plastic Secondary Container | 1 each |
| 33. Chemical surety material used in the testing is authorized under Bailment Agreement DAAK11-84-H-0003. The CSM used in these tests will be HD. | |

E. Apparatus Design

The test apparatus to be used in these tests will be a lucite box that will serve to collect the dust generated by drilling into the concrete coupon (Figure 1.) and a small drill press to afford easy drilling of the coupons.

IV. Analytical Technique

The surface swab samples and the drill dust extraction samples will be analyzed on a Hewlett Packard 5730A Gas Chromatograph. The extraction of the surface swab samples will be done using hexane and the drill dust extraction will be done using chloroform. The extract samples will be pipetted into GC auto-sample vials and submitted for analysis.

V. Test Conditions

- A. CSM: Neat HD
- B. Temperature: Ambient - approximately 68°F
- C. Test Material: Concrete/Painted metal coupons
- D. Solvent: chloroform, hexane
- E. 5 coupons will be tested each day on two successive days for each test method and surface.

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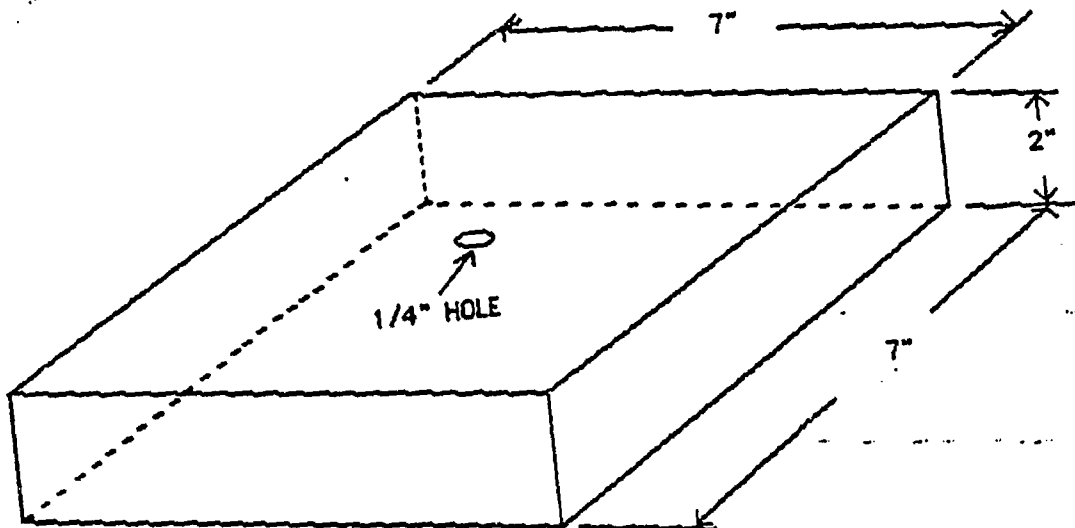


FIGURE 1. SUBSURFACE DRILLING COUPON BOX

F. Test Matrix:

Safety Inspection:

Dry run will be used to obtain background levels of the unchallenged coupons:

Swab tests on 5 blank concrete coupons

Drill tests on 5 blank concrete coupons

Day 1

Maximum recovery tests of the surface swab test using 5 stainless steel coupons.

Maximum recovery test of drill test: spike 25% of actual challenge into 10 mL chloroform

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Challenge 5 concrete coupons with HD, Swab Test.

Challenge 5 concrete coupons with HD, Drill Test.

Day 2

Challenge 5 concrete coupons with HD, Swab Test.

Challenge 5 concrete coupons with HD, Drill Test.

Day 3

Challenge 5 painted metal coupons with HD, Swab Test

Challenge 1 stainless steel coupon with HD, Swab Test, Maximum Recovery

Day 4

Challenge 5 painted metal coupons with HD, Swab Test

Challenge 1 stainless steel coupon with HD, Swab Test, Maximum Recovery.

VI General Procedure

A. Surface Swab Sampling Test

The test will be conducted by placing 75 μ L (95.25 mg) of HD on the surface of a 5" X 5" X 1/4" concrete coupon in a 16 dot pattern. The coupon will immediately be swabbed in 2 directions with a gauze pad that has been dipped in hexane. The swab is then placed into a 4-dram vial containing 10 mL of hexane. The bottle is shaken for a period of time and an aliquot of the extract is placed into a GC auto-sample vial and submitted for GC analysis. Duplicate tests of 5 coupons will be conducted on 2 successive days.

B. Subsurface Drill Sampling Test

The test will be conducted by placing a 5" X 5" X 1"

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concrete coupon into a Lucite box (Figure 1.). 50 uL (63.5 mg) of HD will be applied to the center of the coupon within a defined area and the coupon will be allowed to stand for 30 minutes. Then the lid of the box will be taped into place and 5 holes will be drilled to a depth of 1/4" depth using the drill press. The drill dust will be transferred into a 4-dram vial. A known quantity of chloroform will then be added to the 4-dram vial and shaken for a period of time. An aliquot of the extract will then be placed into a GC auto-injection vial and submitted for analysis. The weight of each concrete sample will also be obtained. Duplicate tests of 5 coupons will be conducted on 2 successive days.

VII. Specific Procedure

A. Surface Swab Sampling

- a. Obtain the required number of 5" X 5" X 1/4" concrete/painted metal coupons.
- b. Place surface template (Figure 2) on surface of coupon and mark the center of the holes with a red marking pen. Mark sample code on coupon. Repeat for all coupons.
- c. Mark 4-dram vials and GC auto-injection vials with the appropriate code.
- d. Place 10 mL hexane in each 4-dram vial and seal with a Teflon-lined cap.
- e. Place coupons in glass baking dishes located within the fume hood.
- f. Position gauze pads, forceps, 4-dram vials, GC vials, pasteur pipets, and 2 2-oz. jars filled with hexane inside the fume hood.
- g. Prepare for 75 uL HD challenge and conduct the challenge of a single coupon according to the procedures defined in HML SOP-21.
- h. Grasp a gauze pad with forceps and dip the pad in hexane from a 2-oz. glass jar. Wipe the challenged area of the coupon in one direction, fold the pad over and swab the challenged area perpendicular to the first swabbing.

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i. Mount 4-dram vial in styrofoam holder. Fold the pad again and place the gauze pad into the appropriately marked 4-dram vial and cap vial. Shake the vial for 15 seconds and set the vial in holder.

j. Rinse forceps in second 2-oz. jar containing hexane.

k. Repeat steps g - j for each of the coupons to tested.

l. Following the extraction of all swabs, place a 1.5 mL aliquot of each 4-dram vial into an appropriately marked GC auto-injection vial. Submit the samples for analysis.

m. Upon completion of the test runs, the test materials and test apparatus are decontaminated according to the procedures defined in HML SOP-10 and waste is disposed of in accordance with HML SOP-11.

E. Subsurface Drill Sampling

a. Obtain the required number of 5" X 5" X 1" concrete coupons.

b. Place subsurface template (Figure 3) on surface of coupon and mark the outline of the 1" square hole on the surface of the coupon with a red marking pen. Mark sample code on coupon. Repeat for all coupons.

c. Mark 4-dram vials and GC auto-injection vials with the appropriate code. Tare each 4-dram vial in the secondary container.

d. Place 10 mL of chloroform in each 4-dram vial and seal with a Teflon-lined cap.

e. Place a concrete coupon into each of the five plastic boxes (Figure 1.), place lids on boxes, and place boxes into fume hood.

f. Position 4-dram vials, GC auto-injection vials, and drill into fume hood.

g. Prepare for 50 uL HD challenge and conduct the challenge according to the procedures defined in HML SOP-21.

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- h. Allow HD to stand and soak into the coupons for 30 minutes.
- i. At the completion of the soak period, secure the lids to the boxes with tape. Position a single box in a large glass baking dish.
- j. Using the drill press, insert drill into the hole located in the top of the lucite box and drill a 1/4" deep hole into the coupon. Pull the drill bit out of the coupon and maneuver the coupon into a different position and drill another 1/4" deep hole. Repeat until 5 holes are drilled into the surface of the coupon.
- k. Remove drill from the hole in the box and tape drill hole. Shake the drill powder off the surface of the coupon and remove the tape from the lid of the box. Position the appropriate 4-dram vial in fume hood.
- l. Open the box and remove the concrete coupon from the box and place it in desicc.
- m. Pour the drill powder from the box into the 4-dram vial and cap with a Teflon-lined cap.
- n. Set box aside within fume hood. Transfer a 1.5 mL aliquot of the extract to an appropriately marked GC vial.
- o. Repeat steps i - n for each of the coupons and DSM to be tested.
- p. Upon completion of the extract analysis, open the 4-dram vials and allow the solvent to evaporate.
- q. Cap the 4-dram vials and place them individually into the secondary container and obtain weight. Calculate the amount of concrete in each sample.
- r. Wipe the boxes with solvent and allow them to dry.
- s. Upon completion of the test runs, the materials and test apparatus are decontaminated according to the procedures defined in SOP HML-10 and waste is disposed of as outlined in SOP HML-11.

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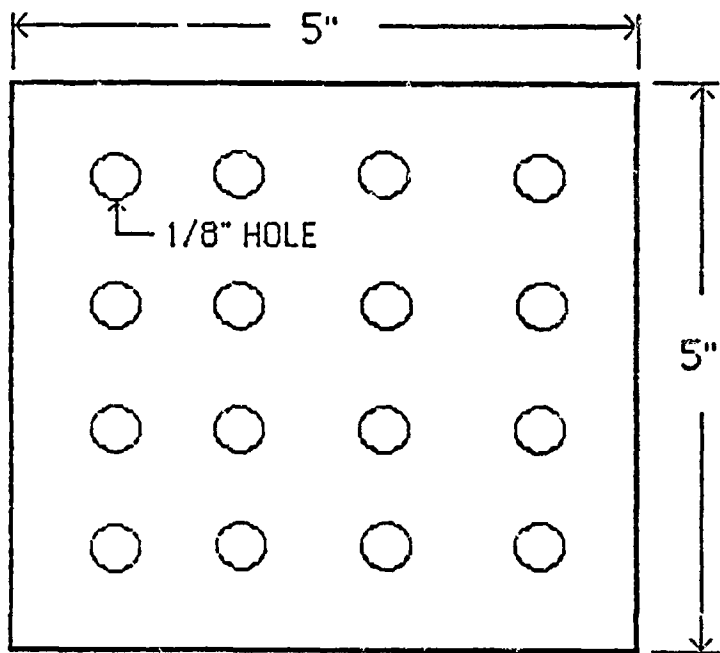


FIGURE 2. SURFACE TEMPLATE

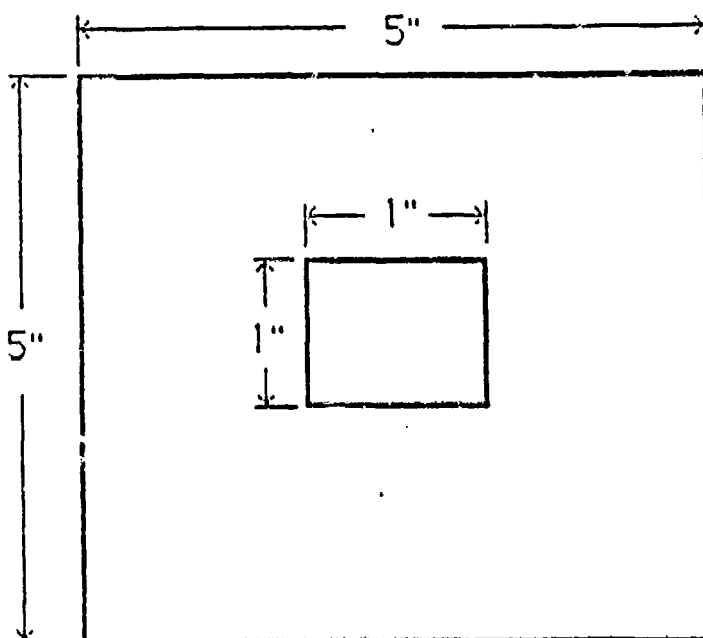


FIGURE 3. SUBSURFACE TEMPLATE

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APPENDIX C
TEST PLAN FROM NOVEL PROCESS DEMONSTRATION AT DPG

REVISED
FINAL REPORT

on

TEST PLAN FOR NOVEL
PROCESS DEMONSTRATION AT
DIGWAY PROVING GROUNDS

to

U.S. ARMY TOXIC AND HAZARDOUS
MATERIALS AGENCY
ABERDEEN PROVING GROUND, MARYLAND

April 24, 1987

under contract
Novel Processes
Contract No. DAAA 15-86-D-0001
Task No. 1, Subtask 6

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**U.S. ARMY TOXIC AND HAZARDOUS
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ABERDEEN PROVING GROUND, MARYLAND****I. General Information****1.1 Project Description**

The objective of this task, Novel Processing Phase III is to conduct a pilot test of the hot gas decontamination concept as investigated in Phase II by Battelle. The Phase II experimental and engineering evaluations demonstrated the feasibility of the hot gas decontamination using laboratory samples. Pilot scale test under controlled conditions are necessary to further evaluate the hot gas decontamination process prior to full-scale implementation.

The part of their responsibilities in DoD property disposal, USATHAMA must identify, contain and eliminate toxic and hazardous materials where facilities, potentially available for alternate government or private use, have been declared excess or are candidates for excessing. With this mandate, USATHAMA must provide not only the technical basis to implement decontamination but also the standards to ensure decontamination has been effective.

Battelle generated, experimentally evaluated, and performed engineering/economic analyses on novel building decontamination concepts under separate contract. Based on the results, Battelle and USATHAMA

selected the hot gas decontamination concept for field evaluation. Maintaining a temperature of 150 C for 60 minutes was effective in decontaminating HD, GB, and VX contaminated painted and unpainted steels and porous materials. A steam decontamination concepts and an OPAB* decontamination concept were also effective decontaminants. However, the engineering analysis indicated OPAB would be less effective than either steam or hot gas, while the economic analysis indicated the hot gas concept is preferred over the steam concept.

For this project a structure will be constructed of 4 different building materials including poured concrete, hollow block wall, solid block wall and steel plate. The structure is described in detail in monthly progress reports to USATHAMA under this contract. The structure will be constructed by a subcontractor, transported to Dugway Proving Grounds (DPG) and prior to testing, the structure will be moved into the test chamber. The original plan (August 20, 1986) requested that the Defensive Testing Center (DTC) be used as the test chamber. This facility was visited on May 14 and 15, 1986 by Battelle engineers and was considered to be the size and level of control required for this test program. Due to delays in availability of the DTC the alternative chamber in Building 3008 will be used for this program.

The test structure will be spiked with HD solution prior to hot gas decontamination demonstration. The idea is to simulate a structure which is essentially at a 3x level of contamination and is typical of structures which may be encountered in field decontamination.

A hot gas generation system will be designed by Battelle engineers and then coupled to the building. The exhaust gases from the building will be cooled and evacuated from the chamber through the HEPA/Charcoal type exhaust filters which are integral to the chamber operations.

In order to provide validation of the decontamination efficiency, it will be necessary to collect samples in the structure both before decontamination and after decontamination. Gaseous monitoring for HD agent will also be required to demonstrate the release rate of HD from the surfaces and also for safety monitoring. Finally, physical

*octyl pyridinium 4-aldoxime bromide.

data of the system will be monitored for characterization of the heat transfer effectiveness of the materials as related to agent release.

1.2 Location of Testing

As a result of the September 30 meeting at DPG, the USATHAMA project officer and the BCD deputy project manager, agreed that 3008 chamber facility at DPG offers an acceptable facility for running a controlled experiment of this nature. By using known spiked coupons, state of the art monitoring, and controlled environmental conditions, the feasibility of hot gas decontamination may be demonstrated. The chamber allows the placement of a large structure within a controlled environment. The coordination of available support facilities at DPG will greatly enhance the success of this project.

1.3 References

The following references are relevant to the background and operations of this project:

"The Development of Analytical Methods for Determination of HD Contamination In or On Building Materials" Technical Report to USATHAMA Contract DAAA 15-86-D-0001, Task 1, Subtask 3, August 15, 1986.

"Advanced Development and Field Testing of Novel Process Technologies to Decontaminated Chemical Agent Contaminated Facilities", Battelle Proposal to AMCCOM Procurement Directorate, Aberdeen Proving Ground, July 19, 1985 on RFP#DAAA-15-85-R-0020

"Development of Novel Decontamination and Inerting Techniques for Explosive Contaminated Facilities, Laboratory Evaluation of Concepts". Report No. AMXTH-TF-TR-85009, USATHAMA, March, 1985.

"Subtask II Report on Design Review" in Battelle Monthly Report to USATHAM, Contract DAAA15-86-D-0001, June 16, 1986.

1.4 Scope

The overall scope of this project is considered as a one time only pilot test with the following objectives:

- Determine the effects of the hot gas process on actual structures and representative materials
- Perform analysis and sampling for agents on or in building materials, soils, and exhaust air streams
- Demonstrate the feasibility of the hot gas process in pilot-scale decontamination tests of contaminated structure
- Confirm/refine the assumptions made in previous engineering and economic evaluations such that a detailed design of a full-scale process can be made.
- Obtain data for use in satisfying regulatory/safety agency requirements for application and validation of a decontamination process.

It is recognized that in order to accomplish the above goals, a joint effort by personnel of DPG and BCD will be required. The purpose of this Test Plan is to delineate the areas of responsibility and the specific requirements for each party.

1.5 Limitations and Responsibilities

The following project limitations and responsibilities are recognized as necessary for the successful operation of this project.

- 1) Overall management of the project will be with BCD under the Battelle project manager Dr. William McNeill, Battelle Denver Operations Office. Dr. McNeill will report all progress and problems directly to the

USATHAMA project officer Mr. Andrew Roach, USATHAMA, Aberdeen Proving Grounds who will have final review authority.

- 2) Operation of the test facility will be the responsibility of DPG personnel. Contractual arrangements for use of the facility will be made between DPG and USATHAMA as required.
- 3) The preparation, handling, and analyses of all samples collected at DPG will be the responsibility of DPG personnel.
- 4) The operation of the hot gas generator will be the responsibility of DPG personnel with the advice of Battelle personnel.
- 5) Construction of the test structure and delivery to Dugway will be by Furst Construction (Salt Lake City Utah) and coordinated with DPG operations. Final approval of construction will be by BCD engineers. Support requirements such as power and fuel will be coordinated with DPG by BCD engineers.
- 6) Installation of the structure into the chamber will be by DPG personnel.
- 7) All agent handling equipment, agent monitoring instruments, real time data monitoring, decontamination of personnel and final disposal of the structure will be by DPG personnel.
- 8) All safety and operating procedures will comply with DPG regulations. All onsite BCD personnel will follow DPG safety regulations and will observe all area access limitations as required by DPG. BCD will prepare a draft of heater operating procedures for DPG.
- 9) During test operations, 24 hours a day monitoring will be required and therefore overtime security and

operations will be required from DPG personnel. BCD personnel will require on-site access in order that a BCD staff person is on site at all times during operations. It is anticipated that during test operations the BCD crew will be limited to 3 persons (1 woman and 2 men). These three people will require housing at Dugway. Additional staff may be required during construction phases but on site housing is not essential for this period.

- .10) All sample handling, sample analyses, and data handling by DPG personnel will be observed by on-site field personnel from BCD. Any questions regarding methodology or safe operating procedures by BCD personnel will be directed to the on-site DPG field manager.

1.6 Calibrations and Quality Assurance

In addition to the above listed general limitations and responsibilities, specific actions related to Quality Assurance will be required as follows with responsibility in parenthesis:

- Maintenance calibration records of all flow devices for air heater operations (BCD)
- Maintenance of records of all HD spike preparations (DPG)
- Maintenance of pre/post calibrations of temperature readouts (DPG)
- Maintenance of calibration records of air sampling instruments (DPG)
- Analyses of necessary internal spikes, and split samples for laboratory quality control (DPG)
- Maintenance of log books during testing operations (BCD and DPG)

- Validation checks of Real Time Monitoring data accuracy (data point checked pretest) and chronometer accuracy (time record checks daily) (DPG).

It is planned to have a QA representative from BCL review specific procedures prior to test operations. The representative will be onsite during testing and analysis. All DPG records will be reviewed internally by required DPG personnel before final release. Records of any corrective action will be noted and included in a final report. Records and data collected by BCD during on site operations will be made available to DPG personnel as required. Copies of all data and calibration records will be collected and stored at the Battelle Denver Operations Office except original BCD laboratory notebooks which will be filed by the Columbus Records Management Office.

1.7 Temporary Chemical Exclusion

The chamber area will require temporary chemical exclusion of 1 working day. Agent diluted for spiking, will be taken from the chemical laboratory to the test area and added to the test coupons. The unused chemical will be returned to the laboratory the same day. Alternatively, the coupons may be spiked in the laboratory but the floor spill solutions will still require transport and delivery onsite.

1.8 Safety

Dugway will be responsible for on site safety during operation of the test. Battelle will provide necessary burner operating guides with safety instruction to Dugway operating personnel.

2. Supplies and Facilities

2.1 Furnished by Dugway

The following supplies, materials, and facilities will be supplied by Dugway in support of this project.

2.1.1 Test Facility

The test facility provided by Dugway will consist of one of the chambers in Bldg. 3008. The test will require access to the control room for monitoring purposes, use of decontamination facility for spiking and sampling, bench work area for coupon spiking, power for operation of burner and monitoring system, filter ventilation of exhaust air, optional use of mobile conditioning unit (MCU) for periods of spiking through cooldown, GP-DAS data system and any required support facilities. In addition, facilities for moving the test structure into the chamber will be required as will parking access for the propane fuel tank outside the test chamber. Installation of thermocouples wiring will be by DPG personnel. Facilities will be included for access to the structure through a door. This door will be sealed with fiberfax and clamps during test operations.

The test structure will require that a bed of soil be placed under the building. This soil should be local soil and can be loaded by a front end loader. No compaction requirement is specified nor is aggregate size a particular concern though large rocks should be avoided in order to allow soil sampling. Provisions for soil sampling through the support structure will be required also. Both loading of soil and sample access will be provided by DPG.

2.1.2 Spiking System

The Battelle test facility will receive HD spiked coupons on four vertical walls. The floor of the structure will receive two spill simulated spikes by means of a syringe type of device. The coupons will be placed in specially designed holders on each surface. The spills will be applied to confined areas of the floor. The floor spikes will consist of

two replicated deliveries to designated areas of the floor. Concrete caulking ridges around the spill area will prevent spillage into adjoining area.

The concentration levels for each coupon spike should be 95 mg of HD per 5" x 5" coupon. For the spill deliveries this amount is multiplied by the area factor to provide 1520 mg of HD. Dugway personnel will design, fabricate and operate the agent spike system as required. Dugway personnel will administer the spike solution to each coupon and floor and will be responsible for placement of each coupon in the test structure.

2.1.3 Sampling Tests

The sampling tests will include 4 sampling programs each of which includes a number of different methods. Each of the four are described in the attached Appendix A of Attachment A.. It is assumed that all sampling will be conducted by Dugway personnel using methods agreed to be acceptable by both Battelle and Dugway management.

2.1.4 Sample Safety

Since all sampling will be conducted by Dugway personnel, any necessary Dugway safety equipment will be supplied by Dugway P. G. Battelle will maintain an operations engineer and/or a field site manager on site at all times. These personnel will be monitoring the test operations and will comply with all Dugway safety requirements. It is assumed that any respiratory or protective clothing, required will be supplied by Dugway. Battelle will supply its own safety shoes, safety glasses, and hearing protection if required.

2.1.5 Decontamination

Dugway will supply all necessary decontamination for personnel involved in sampling using the available decon change house.

The Battelle structure may require decontamination by Dugway personnel and this may be by either solution cleaning on site or disassembly and disposal.

2.1.6 Agent

Agent will be supplied as a solution of HD. It is understood that the agent is currently in a thickened state and it may be necessary to distill the material prior to use. Agent solution will be made using an appropriate solvent as defined by Dugway SOPs. The solvent should be compatible with the delivery system and also should be volatile enough to allow drying after wetting the surface.

The surfaces will be either concrete or steel and will be painted or unpainted. Reaction with painted materials should be minimized. Concentrations will be at workable levels as defined by Dugway regulations but total concentrations should be designed to simulate a contaminated building. All control of agent solutions will be by Dugway but a written record report of concentrations and total mass as delivered in spiking operations will be required by the Battelle on-site field manager. At no time will Battelle personnel be involved in agent handling or transfer.

2.2 Support Equipment Furnished by Battelle

The following support and equipment will be furnished by Battelle Columbus Laboratories.

2.2.1 Personnel

A minimum number of technical personnel will be required onsite at Dugway before, during and after the test. The following levels of personnel have been identified as required for operation of this program:

- Senior Engineer - responsible for design, construction and checkout of test structure system
- Field Test Manager - responsible for communication with Dugway personnel, management of on-site Battelle personnel, and overseeing all test operations
- Operators (2 required for 24 hour shift work) responsible for monitoring of Novel Process system during test, also one person will be on site for pretest checkout and the other for post test dismantling
- Technicians - as required for construction; technicians will be provided prior to actual test operations.
- Alternate Engineer - A person familiar with all aspects of the project will be available for substitution of the above personnel should the need occur.
- Onsite Quality Assurance observer to review analytical methods and data record keeping.

2.2.2 Test Structure

The design of the test structure will be provided by Battelle. The facility was constructed by Furst Construction Inc. The final structure has been delivered to Dugway and final assembly will be by DPG personnel.

The facility does not include 1) the devices to hold coupons in place nor 2) painted surfaces. Both of these will be provided by BCD.

2.2.3 Heating System and Fuel

A propane fired direct type heater will be attached to the test structure by Battelle personnel or Furst. The

acquisition, setup, checkout and operation of this system will be the responsibility of Battelle. The system will require a source of propane fuel outside the chamber at the required setback distance (25 feet from the 3008). The fuel will be a subcontracted item by Battelle.

2.2.4 Controls

Controls for the heating system and thermocouple inputs (30-50) for monitoring the test structure will be provided in the construction. In addition various system pressures will be monitored by Magnehelic type devices and some provision for monitoring and controlling the fuel supply should be included in the fuel handling system. The controls for the chamber and its support systems including the MCU if used will be the responsibility of Dugway personnel and in no way will Battelle attempt to overlay any control system on the Dugway system of operation of the facility.

3.0 Test Procedures

The following outline of test procedures is proposed. All procedures will require review by Dugway and are subject to revision as necessary to meet Dugway operating procedures.

3.1 General Procedures

The overall general purpose of the test procedures is to provide representative information on 1) the amount of HD spike in the test structure, 2) the background matrix of the test structure, 3) the temperature profiles in the various test materials 4) the operation characteristics of the Novel Process Decontamination System, and 5) the level of decontamination achieved in the process.

3.2 System Preparation

A building with a heater unit attached on a separate base will be built in Salt Lake City and delivered to Dugway on one or two flat bed trucks.

Dugway will be responsible for final assembly of the system. Final assembly will include:

- 1) unloading the truck(s)
- 2) Filling the structure base with local soil
- 3) Setting the building on the base
- 4) Repairing damage caused by transport (if any)
- 5) Moving the building into the chamber and blocking wheels on structure (if used)
- 6) Connecting ductwork between the test structure and the exhaust fan
- 7) Connecting duct work for the MCU if used and any required baffling or containment
- 8) Connecting thermocouples to data logger and demonstrating operation and identification of all thermocouples
- 9) Placing combustion control panel in control room and connecting controls to burner
- 10) Checking out blower and combustion controls
- 11) Installing LPG piping and storage tank
- 12) Hooking up instrument air supply if necessary for burner operation (not needed)
- 13) Checking operation of temperature control on burner. Correcting if necessary
- 14) Checking setting on barometer damper (0.5 in) and correcting if necessary
- 15) Checking in leakage into test building. It should be less than 50 cfm at 0.2 inch negative pressure
- 16) If necessary recaulking building (caulking specified by BCD)
- 17) Setup of air monitoring instrument in chamber and air sampling system in exhaust duct.

3.3 System Checkout

The system will be checked out to verify that all systems are operating properly. As a minimum the following checks will be made:

3.3.1 Flames and Pressures

- a. Test structure pressure control by barometric damper. Set to 0.5 in. H₂O
- b. Cooling air damper opens and closes on signal
- c. Exhaust flow meter
- d. Combustion blower fan and gauge
- e. Combustion air flowmeter

3.3.2 Combustion System

- a. Heat control valve operates
- b. Oven temperature safety

3.4 Sampling

Field sampling will include a total of 5 test methods and 4 test periods. As described earlier, there will be a variety of building materials including poured concrete, solid block concrete, steel walls, and soil. Air samples and sample test coupons will be included. Air sampling will be in the exhaust stream during the test and in the chamber when agent is present. The filter inlet may be monitored if required by Dugway but this information is not required by Battelle.

3.4.1 Test Methods

The test methods include 1) wipe samples 2) subsurface drilling, 3) soil samples using Tee type samplers, 4) coupon

collection, and 5) air sampling. The surface wipe samples have been validated by Battelle using concrete coupons and neat HD. The resulting validation SOPs are included in the reference for task 1, subtask 3 report (1986) on this project (Attachment A). A final SOP for each method will be prepared in format necessary for Dugway using the techniques described in the Battelle methods.

The soil sampling will be horizontal borings of the underlying test structure soil using a standard tee type soil sampler furnished by Dugway. These samples will be collected in clean glass containers and then extracted and analyzed by a method similar to the soil SOP developed by Battelle.

Coupons will be included to simulate volatilization of agent from various materials. The coupons will be analyzed following the decontamination test. Each coupon will be retrieved and placed back in its original container. A specific SOP will be developed by Battelle to address any required coupon handling.

The sampling of air for HD will be according to methods developed by Battelle and validated by Dugway. The specific requirements for this project is that the concentration of HD in the exhaust gas should be known at least every work shift during testing and at the start and finish. This data should be made available to the Battelle field manager and the system operator as soon as it is available since the length of time of operation may be based on observed levels of HD in the exhaust gas stream. Extracted samples from the exhaust duct sampling should be saved by storage at 4°C for possible subsequent analysis of GC/MS for breakdown products.

3.4.2 Test Periods

The following four test periods are defined with requirements for samples.

3.4.2.1 Background Sampling

The Battelle structure will require background sampling to establish a baseline for sample collection. The samples to be collected (Sampling procedures are detailed in Appendix A of Attachment A) will include the following:

- surface wipes including the structure floor and four walls will be collected. It is planned to have each wall half painted and half bare with a wipe of each type thus requiring a total of 10 wipe samples (includes 2 floor wipes)
- subsurface samples using a hand drill and collecting the dust. Each of the painted and unpainted floor surface will be sampled and 5 holes approximately $\frac{1}{2}$ to 1 inch deep will be required for each sample. A total of 2 composited floor samples will be collected and each hole will be plugged after sampling using quick setting concrete mix or caulking
- Samples of the underlying soil bed will be taken horizontally at two locations using a tubular sampler.
- Photographs will be taken of the complete structure along with designated sampling locations (minimum 20 photographs).

3.4.2.2 Pretest

The preparation of test coupons will consist of delivering the 95 mg of HD to each of 18 coupons. The coupons will be provided by Battelle and will include the following types:

- painted steel 5" x 5"
- unpainted steel 5" x 5"
- painted cement 5" x 5"
- unpainted cement 5" x 5"
- unpainted cement 5" diameter

Of the above, 1 of each type will be returned to the laboratory as a pretest sample. The remaining spiked coupons will be placed in the test structure as follows:

- unpainted steels on unpainted steel wall (1 coupon)
- painted steels on painted steel wall (1 coupon)
- each painted in each of 3 non-metal walls (3 coupons)
- one painted with spike face towards poured cement wall (painted section)
- each unpainted in each of 3 non metal walls (3 coupons)
- two unpainted with spike faces towards two non-metal-non painted walls
- one unpainted coupon in hole in poured floor (5" diameter)
- one unpainted on surface of unpainted floor
- one painted on surface of painted floor.

Each wall and the floor will include unspiked coupons one painted, one unpainted to act as deposition coupons. The test program will use a total of 32 coupons, 14 of which are spiked and placed in the structure, 4 of which are spiked and analyzed as pretest samples, 10 of which are blank coupons exposed to decontamination and, 4 of which are unexposed blanks for background only.

In addition to the coupon tests two simulated floor spills will be included. These will be administered to a 400 in² area of the floor (both painted and unpainted) by means of a syringe type delivery. The amount of HD should be at least 1520 mg but volume can be adjusted for ease of delivery and handling by diluting with solvent. The solvent should be hexane. The delivery will be administered to the prescribed area and allowed to evaporate dry. The syringe barrel and needle will be solvent rinsed and the rinse will be analyzed to verify percent delivery of agent to each floor test area.

3.4.2.3 Air Monitoring

After the application of the spike it will be necessary to continuously monitor the air environment around the test facility for agent. Specifically the monitoring will include the following:

- Chamber atmosphere
- Exhaust stream from test structure during heating
- Air inside test structure prior to any required entry.
- Air under floor of test structure

The chamber and exhaust gas monitoring will be required at intervals throughout the actual test. The structure environment will need to be monitored only prior to any entry to the structure.

For the chamber atmosphere a bubbler may be used. For hot exhaust stream from the structure bubbler and/or a ACAMS may be used. Inside air sampling may be by bubbler method or other specified by DPG safety requirements. The air under the floor may be monitored by a bubbler.

In actual testing operations the exhaust level of HD should be measured at least once per work shift. It is also necessary to monitor this stream when the experiment is started and terminated. The facility will be considered decontaminated if two consecutive readings concur that (1) the minimum detectable level (MDL) of HD is present and (2) the minimum temperature requirements have been achieved. At this time the facility is to be cooled using ambient air. Monitoring should continue at least 2 more times during cooldown.

3.4.2.4 Post Test Sampling

After the structure is adequately cooled (at least 2 days), an air monitoring test will be conducted inside the structure to assure that the levels are consistent with a minimal risk. At that time, post test sampling will be conducted in a manner similar to that described for 3.4.2.1. During post test sampling it will also be required that samples of any solid material such as blistered paint products or corrosion dust which are deposited on the floor be sampled by scooping into glass vials. All coupons will be collected and placed in individual sample boxes. Photographs of the test structure interior at post test along with condition of each set of coupons upon collection should be provided by DPG (minimum 20 photographs).

3.4.3 Sample Control and Supplies

All samples will be collected by Dugway personnel. Containers can be supplied by Battelle along with any necessary tools or special equipment. All air sampling and monitoring

equipment should be provided by Dugway but if necessary equipment can be provided by Battelle. Solvents would be more conveniently supplied by Dugway but for all sampling operations a minimum of spectro grade or better is required. Appropriate solvent blanks will be taken during each test program.

All samples will remain in the control of Dugway personnel though Battelle on site personnel should be permitted to observe sampling unless prohibited by safety regulations. Data from analyses will be reviewed simultaneously by Battelle and Dugway before release and both will need to agree to release data prior to preparation of a final report. Battelle will supply necessary sample record sheets, sample identification numbers, container labels, and analytical travelers for reporting. Any chain of custody or sample control will be the responsibility of Dugway personnel and a traceable record should be available for each sample for data validation.

3.5 Test Chamber

The operation of the 3008 Chamber will be by Dugway personnel. The conditions to be simulated are normal environmental conditions; constant air temperature with humidity monitored.

3.6 Chamber Systems

The operation of Chamber Systems will be by Dugway personnel. These will include the following:

- Modular conditioning system
- Air filtration system
- Data collection system.

These systems will have to be checked out after installation of the planned operations.

3.7 Novel Process Operation

The operation of the Novel Process Thermal Decontamination system will be by personnel from Dugway. One Battelle observer will be available at all times during the test operations. During the setup and checkout, two Battelle engineers will be onsite to assemble and demonstrate the operation of the system.

3.8 Novel Process Systems

All controls for the burner will be provided by the contractor. It is planned to have a Battelle engineer on site to monitor critical operations during test operations. Any functions which are critical to safe or continuous operation may be alarmed to alert the DPG operators of possible problems. A SOP for burner operation will be provided by Battelle and the plan will include shutdown procedures.

3.9 Test Schedule

A tentative test schedule is shown in Figure 1. The schedule has been planned around a tentative May 4, 1987 test date with completion of the program within 3 weeks. A definition of each task is included below.

- 3.9.1 Setup -
- 1) filling soil and installing soil sampling ports
 - 2) hook up of thermocouples and monitors
 - 3) installation of air monitoring systems
 - 4) setup of fuel delivery system
 - 5) installation of cooling system - dilution air
 - 6) installing test coupon holders in test structure
 - 7) installation of test structure
 - 8) setup of data collection system

0-20

Note: Setup tasks may be completed earlier than dates specified.

- 3.9.2 Checkout-**
- 1) operation of direct fired heater
 - 2) operation of cooling systems
 - 3) operation of data collection
 - 4) identification and replacement or repair of malfunctioning monitors in system
 - 5) setup decon system.
 - 6) calibration checks
- 3.9.3 Background**
- Sample-**
- 1) pretest background air sample test
 - 2) collection and analysis of sample as described in 3.4.2.1
 - 3) photographing of sampling operations and structure interior (approx. 20 photos)
- 3.9.4 Spike**
- 1) review of safety operations
 - 2) check of supplies for test
 - 3) delivery of spike solutions to test chamber
 - 4) setup temporary chemical exclusion
 - 5) test air monitoring systems
 - 6) deliver spikes to coupons
 - 7) fill spill delivery system
 - 8) return any unused solutions
- 3.9.5 Pretest-**
- 1) monitor structure air prior to entry
 - 2) place coupons in designated plates
 - 3) deliver floor spill samples to designated spots
 - 4) collect blank and pretest samples
 - 5) seal and insulate structure door
 - 6) inventory of samples collected delivered to Battelle Test Manager

- 7) return blank coupons and pretest samples to laboratory

- 3.9.6 Analysis-1) Dugway laboratory provides initial screening of HD to verify contamination of structure
- 2) final check of all systems operations
 - 3) validation of data collection system.

- 3.9.7 Test -
- 1) perform thermal decontamination
 - 2) collect manual data
 - 3) collect real time data by data acquisition
 - 4) monitor operation of heater system
 - 5) monitor exhaust gas concentration of HD
 - 6) shut down heating system when two consecutive exhaust air monitor reports are below MDL ($.003 \text{ mg/m}^3$) and all thermocouple points (#'s 1-9, 11-27, 43, 44, 45, 54, 55 from Agent Test Module Drawings) of the test structure are at least $300 \text{ }^\circ\text{F}$ for a minimum of one hour.

- 3.9.8 Cooldown -
- 1) switch to cooldown
 - 2) monitor system by real time data acquisition (automated)

- 3.9.9 Sample -
- 1) monitor structure air prior to entry
 - 2) collect post test samples (3.4.2.4)
 - 3) deliver samples to Dugway lab
 - 4) provide inventory of samples to Battelle field manager
 - 5) Photograph interior of structure and all collected coupons.
 - 6) Calibration checks.

- 3.9.10 Cleanup-
- 1) decontaminate structure with liquid decon solution if required
 - 2) remove test structure
 - 3) disassemble structure
 - 4) dispose of material as required.

3.10 Decontamination

All decontamination will be provided by Dugway. The following may require decontamination.

- personnel taking samples
- test structure after thermal decontamination test
- any sampling or spiking systems exposed to agent
- the test facility if agent vapors are detected outside the test structure
- all ducting, insulation and ancilliary support equipment.

3.11 Decontamination supplies

Supplies for decontamination will be furnished by Dugway. The change out house associated with the facility will be made available and useable for sampling personnel during the pretest and the post test sampling. Backup decontamination will be supplied as required.

3.12 Backup Decon Facilities

Additional facilities for decontamination will be provided as required by Dugway safety regulations. Decontamination will be for HD only.

3.13 Monitoring of Test Chamber

Samples of the test chamber (both the chamber and the test structure) atmospheres will be monitored by Dugway using appropriate methods as required. These two monitoring stations are primarily for safety purposes. The exhaust of the test structure will be monitored at least every day shift during the test as a measurement of the decontamination products and also at the start and finish of the test for indication of initial and final HD concentrations. (continuous 1 hr.)

3.14 Change House

The change house will be operated for use of Dugway sampling personnel during the pre-test and post-test sampling. It is estimated that approximately one day will be required for each sampling period but a reserve day may be included as required. During test operations the change house should be available for any emergency or repair activities but no sampling is planned for the actual test period.

3.15 Operator Training

For the operations of the direct fired heater system, training will be conducted onsite by a Battelle engineer. The trained operators will be responsible for overall system operations and maintenance records.

Operators of the 3008 system and the GP-DAS data collection systems (Dugway) will be trained according to Dugway procedures.

Sample team members (Dugway personnel) will be provided with SOPs for collection of each sample type along with a list of samples to be collected. These SOPs will have been jointly approved by USATHAMA, Dugway Chemistry, Operations, and Safety departments along with the Battelle on site manager, and project manager. Prior to spiking, a practice sample spiking with solvent

will serve as a training exercise. All items related to safety and decon will be specified by Dugway. The methods of collection, labeling, and analyses will be mutually agreed between Battelle and Dugway. An on-site test field manager from Battelle will observe all sampling (without entry into contaminated area) and will provide consultation as required.

3.16 Agent Transfer

All handling of HD agent solution will be by Dugway personnel. Agent handling will include the following:

- distillation of agent
- preparation of spiking solutions
- labeling and record keeping
- transport of agent to test location
- design and operation of floor spill delivery method
- spiking of test coupons placement and recovery of all spiked test coupons
- placement and recovery of all spiked test coupons
- transport of unused solutions and collected samples to laboratory

At no time will Battelle personnel be involved with agent transfer activities.

3.17 Test Procedure

After finalization of a Test Plan, specific test procedures will be provided in a format as specified by Dugway. Battelle will provide the following test procedures.

- collection and analysis of soil samples
- collection of surface wipe samples
- placement and collection of test coupons

- analysis of test coupons
- operation of the direct fired heater systems
- methods for high temperature exhaust air sampling
- requirements for test operations

The necessary operating procedures for the 3008 chamber will be provided by Dugway to their personnel. A standard Operating Plan will be developed by the DPG test officer.

3.18 Contaminated Waste

The test structure can be decontaminated with an appropriate decontamination solution while in the chamber and then moved outside if necessary. The material may be stored on site. If necessary the structure could be demolished and disposed as required. The support materials such as insulation will require on-site disposal. An estimate of the total materials will be provided in the final test structure design report.

3.19 Safety

All safety directives will be according to Dugway operating procedures. Battelle personnel will receive briefings as required. Medical records of Battelle personnel will be provided if required. All Battelle on-site safety will be coordinated through the Field Site Manager who is an industrial hygienist. Any questions regarding questionable activities, liability or other safety related issues will be directed to the Battelle Safety Officer in Columbus, Ohio.

3.20 Protective Equipment

Battelle will provide its personnel with safety shoes, safety glasses, and hard hats if required. Safety equipment related to accidental agent exposure should be provided by Dugway according to their procedures unless specifically requested otherwise by Dugway safety.

3.21 Security

Battelle personnel on-site will have DOD clearances which will have been forwarded to Dugway for site access. In the event that a Battelle team member is not cleared, a cleared Battelle person will act as escort. This will relieve Dugway of responsibility of providing continuous escort.

During test operations, Battelle will maintain at least one person on-site at all times. In the event of the person needing to leave the test site, a contact point will be established with the operator for emergency communications. During cool down periods, Battelle personnel will be provided either on-site or on call as required.

The security of the area will be the responsibility of Dugway. It is especially important that after spiking of the test structure, it be secured until completion of the test in order to assure test data validity.

4.0 Data Requirements

The following data requirements have been tentatively identified as necessary to the operation of this test program. It may be necessary to add additional inputs so provision for additional data should be anticipated.

4.1 Process Monitoring

The following temperatures will be monitored in the test structure. As a minimum interval, temperatures should be scanned every 4 minutes. The TC designations are the corresponding thermocouple numbers from the Agent Test Module Drawings, sheet number 6.

- 1) Inside test structure room (TC 41)
- 2) Inside test structure room, second location (TC 42)

- 3) Inside concrete wall (TC 1)
- 4) Middle concrete wall (TC 2)
- 5) Outside concrete wall (TC 3)
- 6) Inside solid block wall (TC 11)
- 7) Middle solid block wall (TC 12)
- 8) Outside solid block wall (TC 13)
- 9) Inside hollow block wall (TC 21)
- 10) Middle hollow block wall (TC 22)
- 11) Outside hollow block wall (TC 23)
- 12) Steel wall (TC 43)
- 13) Steel ceiling (TC 54)
- 14) Steel ceiling, second location (TC 55)
- 15) Inside 6 inch floor (TC 8)
- 16) Outside 6 inch floor (TC 9)
- 17) Inside 12 inch floor (TC 27)
- 18) Middle 12 inch floor (TC 28)
- 19) Outside 12 inch floor (TC 29)
- 20) Bottom soil, 6 inch floor (TC 10)
- 21) Bottom soil, 12 inch floor (TC 30)
- 22) Duct from test room before dilution (TC 47)
- 23) Duct from test room after dilution (TC 48)
- 24) Air to burner (TC 50)
- 25) Chamber air near test structure (TC 51).

The temperature data will require real time data acquisition and reduction (1 min. average). The output should include 1) hourly average, 2) maximum and minimum values in hour, 3) rate of average change since last hour. All data should be available for manual monitoring access. The Battelle operator will be required to maintain a process report during each shift and access to temperature data will be necessary. A copy of the manual data collection form is included in Appendix A.

In addition the following process data will be monitored.

- 1) Barometric pressure
- 2) Pressure in test chamber

- 3) Pressure in test structure (#1)*
- 4) Pressure in combustion blower outlet (#2)
- 5) Pressure drop in filter systems (DPG)
- 6) Humidity in chamber (#6)
- 7) Flow rate of air to burner (#5)
- 8) Flow rate of fuel to burner (Rotometer)
- 9) Flow rate of exhaust gas from test structure (#3)
- 10) Level of fuel in storage (Manual)

The above data can be either manually monitored or collected as part of the real time monitoring. As a minimum the above data should be collected every 4 hours. Items critical to operations will be monitored more often and may be connected to alarm signal systems if required.

4.2 Agent Monitoring

Monitoring for agent concentration will be required at 3 locations: the test chamber atmosphere, the test structure atmosphere, and the structure exhaust. The monitoring of the chamber is for safety purposes as it provides an indication of contamination of the chamber from outgassing of the test structure. The test structure monitoring using bubblers will be continuous and will be used to determine the risk hazard for entry of personnel. The exhaust stream monitoring provides an indication of the degree of volatilization of spiked agent from the structure. For purposes of concluding the test, two consecutive readings of below MDL ($.003 \text{ mg/m}^3$) will be considered as decontaminated. Note that in no event will a test be terminated without a minimum of one hour at a temperature of 300°F for thermocouples located in the test structure. A field data sheet for air monitoring is attached in Appendix B.

*Note: # designations correspond to Gauge Schedule in Agent Test Module Drawings, Sheet Number 6.

4.3 Analytical Data

Analytical data as reported by Dugway will include results of all analyses as well as assays of agent solutions used for spikes. For spikes, the concentration of HD and identification of the solvent will be required. Information and identification of each sample at each location will be recorded on analytical travelers supplied by Battelle. Example of travelers for wipe sampling, soil sampling, coupon sampling, and miscellaneous (such as paint debris, or corrosion) sampling are included in Appendix C.

4.4 Reporting

All data and analytical results will be reviewed jointly by Battelle and Dugway personnel prior to any release. Any outlier data will be identified and all calculations and records should be available in laboratory record books for review. Dugway will provide directly to the Battelle Program Manager, Dr. W. McNeill (Battelle Denver Operations), the raw data and summary reports as well as completed analytical travelers for each sample collected. The Battelle Field Manager will maintain an inventory of all samples collected for cross checking the reported results. In the event of missing data, records of sample receipt by Dugway will be checked to verify collection of samples.

All final reporting and data management will be performed at the Battelle Denver Operations office. Records of the test structure operational data will be maintained by the Battelle Field Test Manager with copies to the Battelle Denver Operations. All records of the chamber operation will be maintained by Dugway with copies to the Battelle Denver Operations. All analytical travelers provided by Battelle in conjunction with sampling efforts will be returned to Battelle Denver Operations. Copies may be retained by Dugway.

4.5 Quality Assurance

For quality assurance the following activities are identified as essential operations.

1. Records of calibrations of all flow measurement devices such as dry gas meters or rotometers will be forwarded to the Battelle Denver Operations (BDO) office for recording.
2. Copies of lab records on spike preparations will be forwarded to BDO.
3. Calibration records of air monitoring instruments will be made available if required.
4. Laboratory analyses will include internal spikes to demonstrate accuracy of analysis and split samples for precision analysis. Duplicate samples will not be taken in the field.
5. Log books will be maintained for laboratory analyses, operations, and Novel Process operations. In addition Data sheets will be used for data recording and Analytical Travelers will be provided by Battelle for sample recording. All data sheets and log books will be completed in ink only. Errors should be crossed out, initialed and an explanation should be included for each correction. At no time will correction fluid or pencil entries be used for data recording. Data sheets will be returned to BDO. Log books will be retained by original organization (Dugway or BCL) and copies of pertinent records will be forwarded to BDO.
6. Any procedural errors which result in the loss of significant data will be investigated and reported in memo form by the person responsible for each groups actions - the Battelle on-site manager or

the Dugway Project Monitor. The memo should address the data in question, the cause of the problem, action taken, and any results which may be affected. The memo should be addressed directly to the Battelle Project Manager.

5.0 Organization and Schedules

The Battelle field test team is organized under subtask 7 of Task 1, Advanced Development and Field Testing of Novel Process Technologies to Decontaminate Chemical Agent Contaminated Facilities, Contract DAAA15-86-D-001. Mr. Andrew Roach of USATHAMA, Aberdeen Proving Ground, MD is the project officer. The overall Battelle Project Manager is Dr. William McNeill of the Battelle Denver Operations Office. The Task 1 Leader is Mr. Arne Sandberg also from BDO. The following technical personnel from Battelle have been identified as personnel for this task.

Design engineer	Mr. Herb Carlton
Test engineer	Ms. Lee Anne Curtis
Design engineer	Ms. Anne Langham
Field sampling and operations	Mr. William Raytos
On site field test manager	Mr. William Piispanen
Alternate field engineer	Mr. Dale Folsom
Analytical Liaison and QA Review	Mr. Tim Hayes
Records and Documentation	Ms. Mary Bergstrom
Data Management	Mr. John Dwyer

An overall project schedule is shown in Figure 2. This schedule addresses the principle tasks required for the project. The schedule

is not fixed and is subject to revision. Any major changes in schedule will be noted to the BDO project manager by memo with copies to the DPG project manager and the USATHAMA project officer.

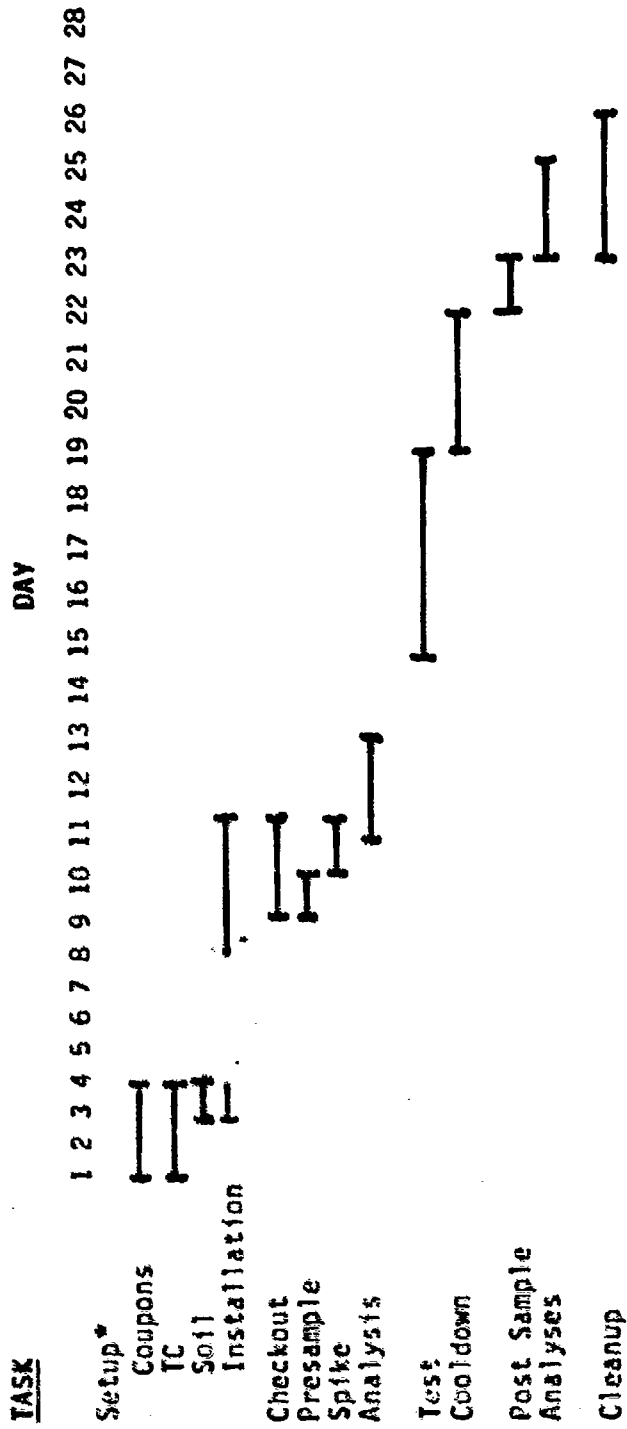


FIGURE 1. PROPOSED TEST SCHEDULE FOR NOVEL PROCESS

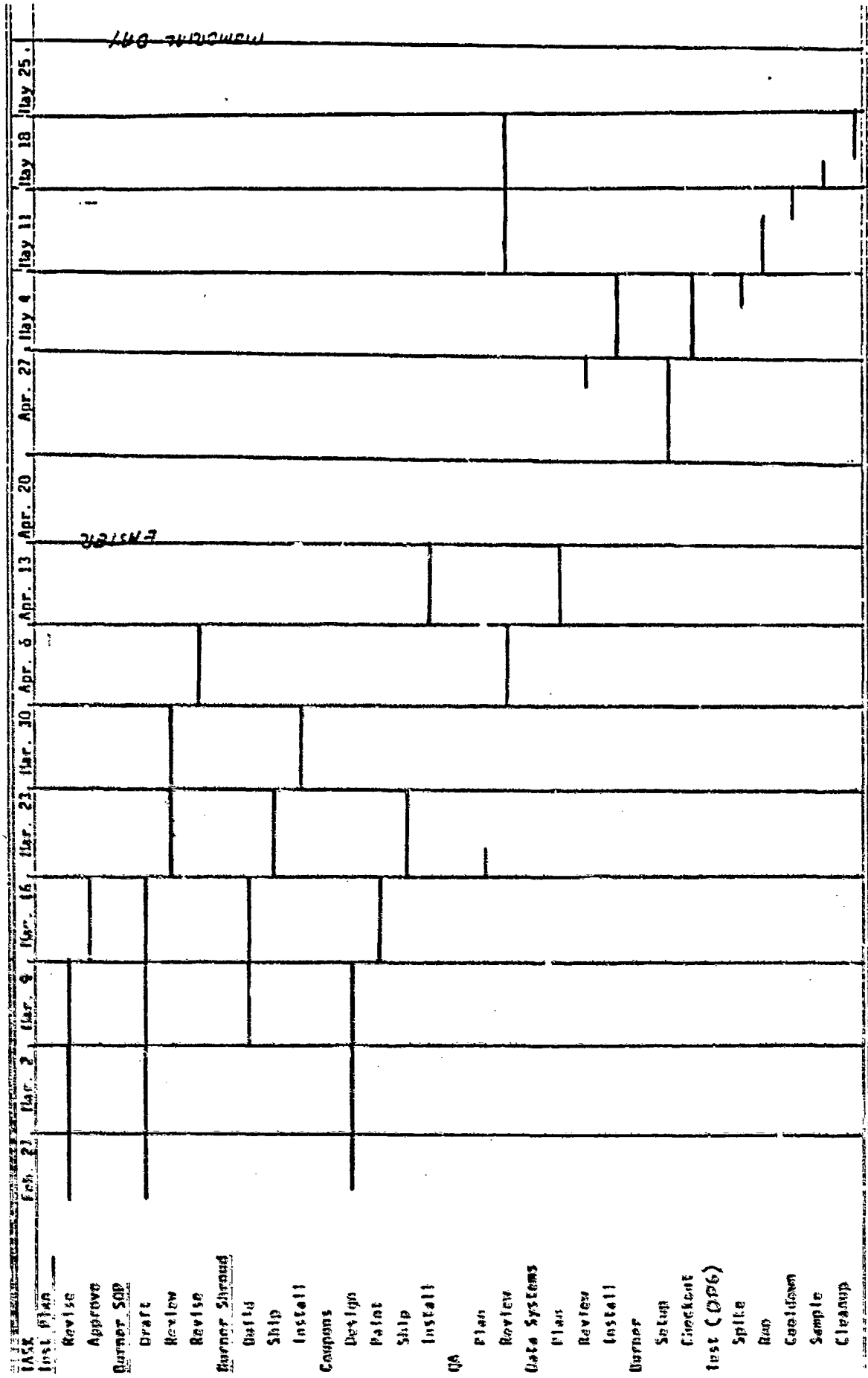


FIGURE 2. PROPOSED SCHEDULE FOR DUGWAY NOVEL TESTING

APPENDIX A
PROCESS MONITORING DATA FORM

DUGWAY PROVING GROUND -- NOVEL PROCESSES
BATTELLE PROJECT NO. G8875-0101

DATA MONITORING, TYPE _____

OPERATOR _____ DATE _____ CLOCK TIME: START _____ STOP _____

Sta NO.	STATION NAME	UNITS	H O U R L Y R E A D I N G														
			1	2	3	4	5	6	7	8	9	10	11	12			

OBSERVATIONS: _____

APPENDIX B
AIR MONITORING DATA COLLECTION FORM

NOVEL PROCESSES
BATTELLE PROJECT NO. G8875-0101

AIR SAMPLE

SITE _____ ID NO. _____

OPERATOR _____ DATE _____ START: HR _____ MIN _____

STOP: HR _____ MIN _____

TEST _____ PRE-TEST _____ POST-TEST _____ BACKGROUND _____

LOCATION OF SAMPLER _____

TYPE OF SAMPLER _____

VOLUME OF GAS (S.T.P.) _____

SAMPLES RECOVERED (1) _____

(2) _____

(3) _____

(4) _____

(5) _____

PROCEDURE REFERENCE _____

OBSERVATIONS: _____

RESULTS

DATE SAMPLE RECEIVED _____

LAB SAMPLE NO. _____

RESULT (1) _____

(2) _____

(3) _____

(4) _____

(5) _____

OBSERVATIONS: _____

ANALYST: _____

DATE REPORTED: _____

APPENDIX C

SAMPLING AND ANALYTICAL TRAVELERS FOR NON-AIR SAMPLES

C-47
NOVEL PROCESSES
BATTELLE PROJECT NO. G8875-0101

DRILL SAMPLE

SITE _____ ID NO. _____

OPERATOR _____ DATE _____ HR _____ MIN _____

PRE-TEST _____ POST-TEST _____ BACKGROUND _____

LOCATION _____

LOCATION DRAWING:

DEPTH (IN) _____

NO. OF DRILLINGS _____

CONTAINER TYPE/SOLVENT _____

PROCEDURE REFERENCE _____

OBSERVATIONS: _____

RESULTS

DATE SAMPLE RECEIVED _____ LAB SAMPLE NO. _____

RESULT _____

OBSERVATIONS: _____

ANALYST: _____ DATE REPORTED: _____

NOVEL PROCESSES
BATTELLE PROJECT NO. G8875-0101

WIPE SAMPLE

SITE _____ ID NO. _____

OPERATOR _____ DATE _____ HR _____ MIN _____

PRE-TEST _____ POST-TEST _____ BACKGROUND _____

LOCATION _____ LOCATION DRAWING: _____

AREA OF WIPE (IN²) _____

SOLVENT/VOLUME _____

CONTAINER TYPE _____

PROCEDURE REFERENCE _____

OBSERVATIONS: _____

RESULTS

DATE SAMPLE RECEIVED _____ LAB SAMPLE NO. _____

RESULT _____

OBSERVATIONS: _____

ANALYST: _____ DATE REPORTED: _____

NOVEL PROCESSES
BATTELLE PROJECT NO. G8875-0101

SOIL SAMPLE

SITE _____ ID NO. _____

OPERATOR _____ DATE _____ HR _____ MIN _____

PRE-TEST _____ POST-TEST _____ BACKGROUND _____

LOCATION _____

LOCATION DRAWING:

DEPTH _____

CORE DIAMETER _____

CONTAINER TYPE _____

PROCEDURE REFERENCE _____

OBSERVATIONS: _____

RESULTS

DATE SAMPLE RECEIVED _____ LAB SAMPLE NO. _____

RESULT _____

OBSERVATIONS: _____

ANALYST: _____ DATE REPORTED: _____

NOVEL PROCESSES
BATTELLE PROJECT NO. G8875-0101

COUPON SAMPLE

SITE _____ ID NO. _____

OPERATOR _____ DATE _____ HR _____ MIN _____

PRE-TEST _____ POST-TEST _____ BACKGROUND _____

LOCATION _____

LOCATION DRAWING:

TYPE OF MATERIAL _____

SIZE _____

PROCEDURE REFERENCE _____

OBSERVATIONS: _____

RESULTS

DATE SAMPLE
RECEIVED _____

LAB SAMPLE NO. _____

RESULT _____

OBSERVATIONS: _____

ANALYST: _____

DATE REPORTED: _____

NOVEL PROCESSES
BATTELLE PROJECT NO. G8875-0101

SAMPLE TYPE _____

SITE _____ ID NO. _____

OPERATOR _____ DATE _____ HR _____ MIN _____

TEST _____ PRE-TEST _____ POST-TEST _____ BACKGROUND _____

LOCATION _____ LOCATION DRAWING: _____

SAMPLE MEDIUM _____

CONTAINER TYPE _____

SAMPLE ADJUSTMENTS _____

PROCEDURE REFERENCE _____

OBSERVATIONS: _____

RESULTS

DATE SAMPLE RECEIVED _____ LAB SAMPLE NO. _____

RESULT _____

OBSERVATIONS: _____

ANALYST: _____ DATE REPORTED: _____

APPENDIX D
SOP FOR NOVEL PROCESS BURNER SYSTEM

Study Specific SOP NO.
G8875-01
Revised
April 24, 1987

OPERATIONS FORMAT

STANDARD OPERATING PROCEDURES FOR NOVEL PROCESS BURNER SYSTEM

A. OPERATION START-UP

Note: For pre-start-up (fuel valve sizing), contact Maxon distributor, Charles Alexander of Blair-Alexander Engineering.

Step No.	Description	Specific Instruction
1.	Purging Furnace	1.1 Start ventilation (filter) system blower. Turn cooling air damper switch to open position.
		1.2 Set temperature controller to manual and run gas valve to zero-position (min. fire).
		1.3 Set high-temperature limit controller to 800°F.
		1.4 Push start button, start combustion air blower.
		1.5 Manually reset high-temperature limit controller.
		1.6 Purge furnace thoroughly, allowing for several (5) complete air changes (at least 15 min.).
		1.7 Change cooling air damper switch to closed position.
2.	Pilot Ignition, Burner Start-up	2.1 Flip pilot/burner selector switch to "on".
		2.2 Push "start pilot" button. (Pilot will start; red "flame on" will light.)

Study Specific SOP NO.
G8875-01
Revised
April 24, 1987

2.3 Release pilot start button. (Main valve opens automatically, main burner is on.)

2.4 Go to automatic control on temperature controller, set temperature control to 200°F.

Note: In the event of flame-out, repeat steps 1 and 2.

If flame-outs persist, check condition (moisture or dirt) of UV scanner.

3. Heat-up

3.1 When the temperature of the test module has reached 200°F, raise the temperature control by 50° every hour--until module reaches 400°F.

3.2 Hold the temperature at 400°F for 6 hours.

3.3 Continue raising the control temperature by 50° every hour until the module reaches 750°F.

3.4 Hold the temperature at 750°F until the end of the test.

Note: If steaming occurs within the test structure at any point during heat-up, hold the temperature until steaming stops.

4. Shut-down

4.1 Flip pilot/burner selector switch to "off" position (keep combustion air on during cool-down).

4.2 Shut off propane feed at tank, disconnect line.

4.3 Open cooling damper.

Study Specific SOP NO.
G8875-01
Revised
April 24, 1987

Approved by:

Anna D. Barker 4/28/87
Date
Anna D. Barker, Ph.D.
Senior Vice President
Biological and Chemical Sciences

David Sticher 4/28/87
Date
David Sticher,
Safety

Ramona A. Mayer 04/28/87
Date
Ramona A. Mayer, Director
Quality Assurance Unit

James A. Gieseke 4/27/87
Date
James A. Gieseke
Associate Section Manager

William McNeill 4/28/87
Date
William McNeill
Project Leader

Afne Langham 4/24/87
Date
Afne Langham
Researcher

APPROVED
[Signature]

APPENDIX E
RESULTS OF METHOD VERIFICATION STUDIES, CPG

Dugway, Utah 84022-5000

Tim Hayes
BATTELLE
Columbus Division
505 King Ave.
Columbus, Ohio 43201

Mr. Hayes:

Attached are the results from the validation studies you requested concerning the building decontamination project. On April 14th I received a package containing two concrete (one thick, one thin), two painted concrete, one steel, and one painted steel coupon. The thin concrete coupon was pulverized for the extraction study. Because I received so few samples I divided the coupons into four 2.5" by 2.5" squares by marking each coupon with a pencil. The methods outlined in your report were executed with the following exceptions:

- 1) All extraction and coupon samples were spiked with neat HD (92% purity) to facilitate easy delivery. This spiking method also eliminated solvent effects on the painted surfaces which resulted in higher recoveries from the swab samples.
- 2) Ten milliliters of chloroform were used to extract the HD from the soil and pulverized concrete samples. The additional chloroform was necessary due to the high clay content of Dugway soil. The clay content may explain the lower than expected recoveries from the soil extractions.
- 3) Four 5ul drops were used to spike each 2.5" by 2.5" section of coupon. Thus each coupon was contaminated with a total of 80ul of HD instead of 75ul which was specified in the report.
- 4) Our surety regulations require that two people be present during any neat agent operation. Because of this requirement I was able to swab the coupon immediately after my partner spiked the surface. This procedure also contributed to the high percent recoveries from the swab samples, which is particularly noticeable in the unpainted concrete samples where recoveries were very high in comparison to your findings.

Brian Thompson
Chemical Technology Branch

TABLE 1. Results of Pulverized Concrete Extraction Samples

<u>Sample No.</u>	<u>% Recovery</u>	
1-4-16	100	$\bar{X} = 94$
2-4-16	92	SD = 3.5
3-4-16	95	%RSD = 3.7
4-4-16	90	
5-4-16	92	
Blank	BDL	
6-4-20	87	$\bar{X} = 89$
7-4-20	88	SD = 1.7
8-4-20	92	%RSD = 3.7
9-4-20	88	
10-4-20	89	
Blank	BDL	

Method Recovery = 91%

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 0.5 $\mu\text{g/g}$ Concrete

TABLE 2. Results of Soil Extraction Samples

<u>Sample No.</u>	<u>% Recovery</u>	
1-4-16	69	\bar{X} = 74
2-4-16	70	SD = 4.1
3-4-16	76	%RSD = 5.5
4-4-16	78	
5-4-16	79	
Blank	BDL	
6-4-20	78	\bar{X} = 76
7-4-20	75	SD = 1.9
8-4-20	78	%RSD = 2.5
9-4-20	77	
10-4-20	73	
Blank	BDL	

Method Recovery = 75%

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 0.7 $\mu\text{g/g}$ Soil

TABLE 3. Results of Concrete Swab Samples

<u>Sample No.</u>	<u>% Recovery</u>	
1-4-16	72	\bar{x} = 67
2-4-16	71	SD = 5.0
3-4-16	62	%RSD = 7.5
4-4-16	61	
Blank	BDL	

Method Recovery = 67%

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 1.2 $\mu\text{g}/\text{sq. in.}$ Concrete

TABLE 4. Results of Painted Concrete Swab Samples

<u>Sample No.</u>	<u>% Recovery</u>	
1-4-16	100	$\bar{X} = 100$
2-4-16	100	SD = 0.0
3-4-16	100	%RSD = 0.0
4-4-16	100	
Blank	BDL	
5-4-20	100	$\bar{X} = 100$
6-4-20	100	SD = 0.0
7-4-20	100	%RSD = 0.0
8-4-20	100	
Blank	BDL	

Method Recovery = 100%

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 0.8 $\mu\text{g}/\text{sq. in.}$ Painted Concrete

TABLE 5. Results of Steel Swab Samples

<u>Sample No.</u>	<u>% Recovery</u>	
1-4-16	100	$\bar{X} = 100$
2-4-16	100	SD = 0.0
3-4-16	100	%RSD = 0.0
4-4-16	100	
Blank	BDL	

Method Recovery = 100%

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 0.8 $\mu\text{g}/\text{sq. in. Steel}$

TABLE 6. Results of Painted Steel Swab Samples

<u>Sample No.</u>	<u>% Recovery</u>	
1-4-16	92	$\bar{X} = 96$
2-4-16	100	SD = 3.0
3-4-16	95	%RSD = 3.1
4-4-16	98	
Blank	BDL	

Method Recovery = 96%

BDL = Below Detectable Limit

Theoretical Method Detection Limit = 0.83 $\mu\text{g}/\text{sq. in.}$ Painted Steel

APPENDIX F
HOURLY TEMPERATURE AVERAGES DURING PILOT TEST, DPG

APPENDIX F. HOURLY AVERAGES OF TEMPERATURES RECORDED DURING THE PILOT TEST OPERATION, JULY, 1987, AT DUGWAY PROVING GROUND (DPG).

Attached is a table of the hourly average temperatures of selected thermocouples in the test structure recorded during the pilot test (see text for list of thermocouple locations). The data was gathered continuously by the GP-DAS data system provided by DPG. Other summarized temperature data is included in Volume 2 of this report.

The time shown in the table is in hours. All thermocouple readings are in degrees Fahrenheit. The data begins before the test structure is spiked and continues until the end of the cooldown period. The "0.00" indicates that the thermocouple connection was open during the entire hour, thus the readings are meaningless.

TIME	TC-1	TC-2	TC-3	TC-4	TC-5	TC-6	TC-7	TC-8	TC-9	TC-10	TC-11	TC-12	TC-13	TC-14	TC-15	TC-16
53	81.93	82.33	74.71	75.66	75.59	80.88	80.86	73.88	75.23	81.91	80.00	73.62	73.17	74.05	0.00	0.00
54	81.48	82.02	74.12	75.34	75.00	80.59	80.81	73.54	75.05	81.50	79.94	73.07	72.79	73.40	0.00	0.00
55	81.89	82.15	73.92	75.19	74.47	80.80	81.09	73.54	75.02	81.73	80.24	73.17	72.72	73.37	0.00	0.00
56	82.28	82.62	74.33	75.12	74.48	81.38	81.50	74.69	75.28	82.40	80.62	73.62	73.09	73.80	0.00	0.00
57	82.16	82.63	73.85	75.10	73.61	81.18	81.35	74.64	75.02	82.32	80.63	73.08	72.83	73.43	0.00	0.00
58	82.06	82.60	73.29	74.83	72.66	81.10	81.42	74.68	74.63	81.99	80.64	72.65	72.52	73.05	0.00	0.00
59	82.16	82.39	73.31	74.24	71.98	81.01	80.95	74.17	73.79	81.63	80.69	72.28	72.06	72.62	0.00	0.00
60	81.69	81.92	72.44	73.10	70.88	80.34	80.17	73.39	72.58	80.78	80.17	71.49	71.13	71.71	0.00	0.00
61	81.22	81.72	71.85	73.01	70.62	81.68	79.98	73.16	71.81	80.38	79.48	70.98	70.74	71.08	0.00	0.00
62	81.29	81.79	71.99	73.04	70.43	79.69	79.88	73.16	71.38	80.26	79.49	70.78	70.59	70.87	0.00	0.00
63	81.30	81.83	71.83	72.82	70.31	79.88	80.07	73.29	71.11	80.30	79.37	70.81	70.70	70.72	0.00	0.00
64	80.69	81.18	71.14	72.70	69.45	79.08	79.75	72.88	69.91	79.76	78.88	69.95	69.81	69.84	0.00	0.00
65	80.69	81.36	71.38	72.07	69.67	80.52	79.57	72.72	68.58	79.57	78.84	69.90	69.82	69.87	0.00	0.00
66	81.07	81.51	71.90	72.30	70.62	80.88	80.78	74.09	70.85	80.79	79.95	71.41	71.27	71.53	0.00	0.00
67	80.10	80.57	71.83	72.03	72.55	80.87	79.82	72.69	69.92	80.78	78.51	71.75	71.26	72.10	0.00	0.00
68	79.72	80.07	71.88	73.03	72.88	78.89	79.10	72.12	70.02	79.06	77.67	71.44	70.68	71.69	0.00	0.00
69	79.57	79.84	71.80	72.88	72.71	88.78	78.89	72.12	70.02	79.06	77.67	71.44	70.68	71.69	0.00	0.00
70	78.65	80.10	72.58	73.18	73.18	89.34	78.21	72.39	70.52	78.80	77.59	71.24	70.10	71.90	0.00	0.00
71	79.80	79.73	72.87	73.81	74.29	71.50	78.74	71.79	71.21	79.07	78.35	71.30	71.04	71.74	0.00	0.00
72	79.72	79.64	73.01	73.95	74.70	73.50	79.07	71.96	72.03	79.59	78.37	71.78	71.58	72.17	0.00	0.00
73	80.23	80.43	73.43	74.23	74.99	79.31	78.52	72.34	73.80	79.84	77.27	72.30	71.84	72.84	0.00	0.00
74	79.78	79.88	73.38	74.18	74.88	79.09	78.88	71.97	73.28	79.57	77.67	72.09	71.43	72.95	0.00	0.00
75	78.84	79.84	73.14	74.05	74.83	79.08	78.02	71.50	74.09	79.54	77.18	71.93	71.34	72.53	0.00	0.00
76	78.89	79.87	72.91	74.05	74.84	79.50	78.08	71.77	74.68	79.81	77.28	72.05	71.52	72.62	0.00	0.00
77	79.14	79.51	72.77	73.55	73.81	78.96	77.44	70.89	74.62	79.28	76.58	71.31	70.84	72.07	0.00	0.00
78	78.48	79.83	72.19	73.22	73.45	78.02	77.34	71.03	74.38	79.28	76.88	71.41	70.84	71.84	0.00	0.00
79	78.83	79.80	72.25	73.67	73.67	79.38	77.91	71.40	74.82	79.69	77.37	71.68	71.04	72.14	0.00	0.00
80	75.58	78.53	71.85	73.47	73.41	71.44	79.61	71.41	75.01	79.79	77.27	71.73	70.88	72.05	0.00	0.00
81	78.80	79.01	71.29	72.78	72.37	89.85	79.32	71.19	74.23	79.38	77.26	71.18	70.87	71.59	0.00	0.00
82	78.91	79.95	70.90	72.49	71.41	87.85	78.14	71.19	73.80	79.35	77.89	71.25	70.78	71.62	0.00	0.00
83	78.51	78.84	71.21	72.13	71.25	88.83	79.24	71.15	73.68	79.24	77.68	70.68	70.45	71.14	0.00	0.00
84	78.86	78.80	71.26	72.17	71.12	86.70	79.20	71.25	73.55	79.10	77.52	70.68	70.28	71.05	0.00	0.00
85	78.82	78.61	71.28	71.84	71.19	88.93	78.88	71.09	73.00	78.98	76.81	70.34	69.38	70.74	0.00	0.00
86	78.38	78.48	71.08	71.72	70.85	88.94	78.59	70.88	72.47	78.68	76.59	69.86	68.82	70.38	0.00	0.00
87	79.19	79.38	70.87	71.63	70.70	86.79	78.35	70.58	72.12	78.49	76.30	69.44	68.60	70.10	0.00	0.00
88	79.25	78.48	70.88	71.67	70.70	85.74	78.40	70.58	72.01	78.58	76.39	69.57	68.77	70.05	0.00	0.00
89	79.17	79.33	70.89	71.67	70.98	87.19	78.38	70.88	71.84	78.64	76.70	69.93	69.28	70.31	0.00	0.00
90	78.60	78.80	70.80	72.16	71.98	89.04	78.21	70.88	71.58	78.58	76.72	70.48	69.88	70.84	0.00	0.00
91	105.83	78.21	71.59	72.80	73.10	72.05	81.12	71.78	72.35	104.65	77.68	71.19	70.58	71.85	0.00	0.00
92	144.68	82.97	72.55	73.53	74.15	107.69	83.88	75.59	72.73	140.64	82.83	72.86	72.53	73.91	0.00	0.00
93	176.80	80.28	74.57	75.27	75.39	122.18	81.03	81.90	72.79	169.61	91.68	77.47	77.41	79.29	0.00	0.00
94	214.80	101.04	78.34	78.88	79.57	140.24	101.31	91.51	73.75	203.73	104.38	85.80	86.26	88.64	0.00	0.00
95	252.68	113.66	85.54	85.78	83.86	159.73	113.52	102.61	74.33	239.14	118.74	85.22	96.74	98.54	0.00	0.00
96	270.07	128.22	82.85	82.45	88.89	172.26	128.86	115.33	74.18	256.86	134.93	105.29	108.77	108.68	0.00	0.00
97	281.80	144.00	103.46	101.97	86.51	183.04	142.03	129.93	75.08	268.91	152.40	118.09	122.73	122.41	0.00	0.00
98	288.51	158.21	114.02	111.44	104.42	192.33	153.85	142.16	75.42	277.53	166.92	129.64	135.03	133.23	0.00	0.00
99	295.04	170.89	123.81	120.52	111.82	199.58	165.78	153.51	74.77	284.81	179.77	139.44	146.25	142.11	0.00	0.00
100	298.88	182.52	133.44	129.17	119.11	0.00	206.40	164.05	74.64	290.70	190.75	149.44	158.78	150.10	0.00	0.00
101	328.53	184.00	142.48	137.68	128.21	0.00	222.07	188.59	75.03	315.77	200.55	158.63	168.85	157.03	0.00	0.00
102	370.70	205.81	151.55	145.51	132.78	0.00	241.51	197.33	74.99	351.22	210.20	170.68	184.22	163.40	0.00	0.00
103	418.04	215.71	180.00	153.12	138.39	0.00	261.41	198.32	74.48	392.95	216.69	179.58	193.02	173.02	0.00	0.00
104	472.30	227.06	170.80	182.21	146.34	0.00	282.31	211.96	74.58	440.07	220.05	183.00	216.74	184.03	0.00	0.00
105	502.87	242.41	176.51	171.58	153.02	0.00	340.05	229.92	74.92	493.50	223.24	183.04	244.57	189.40	0.00	0.00

TIME	TC-1	TC-2	TC-3	TC-4	TC-5	TC-6	TC-7	TC-8	TC-9	TC-10	TC-11	TC-12	TC-13	TC-14	TC-15	TC-16
108	580.10	256.55	184.87	184.34	183.23	0.00	387.13	263.55	248.48	75.20	545.16	228.22	187.93	848.34	192.89	104.32
107	650.15	273.43	188.75	198.38	174.68	0.00	428.32	288.83	286.33	75.71	600.04	235.52	191.02	921.02	195.47	109.78
106	658.89	287.17	208.38	208.17	188.05	0.00	448.38	308.17	284.81	76.30	621.25	243.82	195.11	847.87	197.69	115.05
105	874.72	300.23	218.08	213.85	184.48	0.00	458.18	325.74	303.46	77.07	631.63	254.07	187.77	842.00	200.60	118.15
110	878.78	312.38	218.18	228.71	187.78	318.00	488.71	320.42	303.46	77.85	638.72	268.48	184.61	853.84	200.68	115.62
111	885.54	324.81	224.71	234.81	187.01	0.00	488.80	351.42	338.47	78.70	648.11	268.54	192.14	841.73	202.38	115.58
112	881.32	328.82	231.41	237.80	187.85	0.00	474.23	378.53	355.44	78.84	650.91	308.12	201.55	841.73	202.38	115.58
113	894.50	372.51	238.20	235.18	208.87	628.78	474.23	387.29	373.53	82.03	655.05	328.56	209.38	204.06	218.38	114.38
114	886.88	360.08	245.38	233.75	181.53	0.00	482.50	413.53	380.40	85.80	658.87	348.48	225.99	248.08	237.82	115.03
115	888.84	372.51	257.37	256.38	215.26	0.00	486.15	428.27	408.30	88.28	663.70	368.61	247.53	274.53	256.50	116.12
116	700.84	384.87	268.03	268.16	221.17	0.00	489.32	440.32	418.33	80.85	667.18	389.68	264.52	295.84	271.45	115.80
117	702.08	388.38	270.80	273.03	208.38	0.00	492.33	452.80	431.24	84.07	670.67	408.33	278.51	315.47	284.31	115.47
118	703.70	407.42	278.07	277.88	232.01	0.00	495.58	484.21	442.87	97.08	674.08	428.86	280.54	331.89	294.77	114.44
118	705.08	417.37	282.88	284.10	227.38	0.00	488.54	473.83	453.12	98.72	677.54	442.50	302.82	346.34	304.92	114.82
120	705.88	428.20	288.90	281.41	251.86	0.00	501.50	482.27	462.18	101.86	680.03	458.03	313.10	359.19	314.62	115.98
121	707.82	435.84	284.82	282.36	228.07	0.00	505.43	490.56	471.02	105.07	683.78	469.19	322.85	371.45	328.81	117.02
122	708.80	444.38	301.10	288.08	244.50	0.00	508.21	488.14	478.28	107.84	686.58	480.88	331.85	382.87	338.88	118.70
123	709.81	452.10	304.86	287.88	258.18	0.00	513.10	504.50	485.73	110.28	688.84	491.07	339.87	392.45	344.43	120.84
124	710.77	459.87	308.85	301.47	284.86	0.00	517.48	509.88	491.92	112.67	690.48	500.23	347.00	401.78	351.88	121.79
125	711.81	467.58	313.87	305.70	288.34	0.00	522.50	515.38	498.14	115.33	692.07	509.15	358.78	410.83	359.21	122.58
126	657.07	475.28	320.25	311.23	273.02	0.00	485.87	519.83	503.20	117.58	635.53	516.89	362.75	417.49	364.16	121.78
127	638.17	478.88	328.84	317.77	271.50	0.00	388.84	512.24	500.45	118.87	485.67	517.81	368.08	422.11	367.47	122.23
128	478.80	477.87	328.20	323.74	274.31	0.00	360.15	488.34	483.80	120.28	432.23	505.80	386.54	422.61	397.01	128.18
129	424.47	470.88	343.11	324.28	278.33	0.00	374.70	463.42	481.60	121.31	388.00	487.22	361.14	417.68	380.61	117.75
130	407.88	461.41	344.04	330.18	278.87	0.00	315.28	438.81	438.80	122.33	387.15	487.70	352.57	408.18	350.82	114.73
131	380.82	450.88	345.78	330.85	280.33	0.00	289.88	418.32	417.12	123.05	352.23	447.99	342.29	385.88	328.89	107.61
132	338.78	428.85	338.88	328.33	271.73	0.00	275.87	377.58	378.32	123.73	328.06	412.44	319.98	357.59	317.63	103.31
134	312.28	408.80	336.88	318.43	268.88	0.00	285.61	360.46	361.11	124.20	313.84	398.10	308.95	353.42	303.78	99.91
135	288.82	388.78	324.34	312.71	265.07	0.00	258.52	345.15	345.45	124.50	301.31	380.60	297.78	338.88	284.15	88.88
141	283.52	348.88	288.78	280.48	247.05	0.00	208.88	270.32	288.48	121.08	238.00	297.13	241.93	284.82	235.59	93.52
142	283.48	335.52	280.31	274.28	242.41	0.00	203.23	260.75	268.54	120.92	230.18	285.83	234.28	254.50	227.95	94.52
143	241.58	328.88	282.83	267.85	238.22	0.00	188.00	251.88	248.24	120.80	222.53	275.18	228.88	244.78	220.58	95.86
144	235.78	318.84	278.82	261.25	231.32	0.00	182.84	243.42	240.77	120.79	218.78	285.37	219.98	235.77	213.59	96.02
148	229.50	308.81	268.48	254.15	225.78	0.00	187.10	234.82	231.87	119.89	218.70	255.15	212.48	226.81	206.23	95.80
148	228.50	301.84	262.85	247.31	220.32	0.00	182.53	227.58	224.14	119.84	202.90	248.20	205.95	218.87	189.93	95.90
147	218.07	284.47	258.87	240.72	214.90	0.00	178.27	220.81	217.33	119.34	187.60	238.03	199.63	211.55	194.26	95.40
148	213.87	288.15	250.17	233.87	208.84	0.00	172.78	213.84	210.18	118.83	181.81	228.85	193.44	203.85	188.37	94.84
148	208.51	278.10	244.88	227.84	206.17	0.00	169.78	207.81	203.73	118.18	186.82	222.55	187.89	197.73	183.17	94.30
150	204.20	272.50	238.88	221.25	201.24	0.00	168.18	202.44	198.31	118.11	182.28	215.84	182.59	191.47	177.80	92.82
151	198.82	268.04	232.88	214.80	188.38	0.00	162.57	187.18	182.83	117.74	177.54	209.43	178.80	185.57	172.86	91.33
152	193.84	258.24	225.88	207.81	181.03	0.00	158.43	181.55	181.54	116.78	172.26	203.60	171.23	179.41	167.87	89.63
153	188.82	252.24	218.40	200.89	185.50	0.00	154.24	186.02	181.54	115.42	167.10	186.52	165.79	173.31	162.83	87.38
154	183.81	245.81	214.35	185.18	180.51	0.00	150.83	181.15	178.51	114.27	162.85	180.87	161.35	168.37	158.27	85.99
155	178.18	238.84	208.25	189.08	175.44	0.00	148.48	175.44	170.85	112.39	157.69	184.48	156.00	162.49	153.05	83.74
156	173.80	232.58	202.84	182.42	170.80	0.00	142.82	170.78	168.12	110.88	153.22	173.89	150.83	158.98	148.00	81.55
157	168.88	228.28	187.18	177.80	166.08	0.00	138.53	162.51	161.83	109.45	149.03	173.32	146.35	151.89	143.68	79.33
158	164.88	220.22	181.88	172.82	160.12	0.00	133.15	162.30	157.42	107.81	145.05	168.23	141.83	147.08	139.80	77.73

TIME	TC-1	TC-2	TC-3	TC-4	TC-5	TC-6	TC-7	TC-8	TC-9	TC-10	TC-11	TC-12	TC-13	TC-14	TC-15	TC-16
158	180.33	214.28	189.54	167.37	155.60	0.00	133.05	158.28	153.47	109.22	141.52	163.44	137.58	142.71	135.13	75.71
160	156.58	206.77	182.13	182.80	152.42	0.00	120.11	154.33	149.48	104.67	137.88	158.10	134.23	138.52	133.06	76.28
161	152.73	202.82	177.68	155.32	148.85	0.00	127.47	150.50	145.54	103.06	134.73	154.55	131.00	134.65	130.06	76.79
162	148.43	197.36	174.38	156.08	150.86	0.00	125.41	147.09	142.03	101.81	132.13	150.48	128.96	131.62	127.59	78.92
163	146.35	181.88	171.27	150.67	148.72	0.00	123.08	142.82	137.87	100.48	128.04	148.17	128.18	128.00	125.07	79.43
164	148.05	188.44	171.17	148.37	156.08	0.00	120.74	140.63	136.35	100.16	128.72	149.05	124.21	125.60	123.90	80.72
165	147.17	183.63	170.48	148.15	158.09	0.00	117.68	138.38	133.57	89.86	123.51	140.75	122.86	124.17	122.78	80.74

TIME	TC-31	TC-22	TC-23	TC-24	TC-25	TC-26	TC-27	TC-28	TC-29	TC-30	TC-32	TC-41	TC-42	TC-43	TC-44	TC-45
159	133.17	125.28	108.02	69.91	102.59	0.00	139.83	177.49	175.28	0.00	138.52	103.66	82.47	67.94	105.52	104.60
160	128.96	123.54	106.90	68.73	101.10	0.00	136.96	174.06	171.95	0.00	135.66	101.78	82.34	67.68	103.46	102.43
161	127.01	120.88	104.67	74.37	99.20	0.00	134.18	170.51	168.60	34.89	133.18	100.61	82.39	67.84	101.95	100.37
162	124.42	119.15	102.01	82.57	97.25	0.00	131.75	167.15	165.51	41.34	130.95	99.98	83.82	69.82	101.88	99.44
163	121.88	117.12	99.60	88.23	95.99	0.00	129.19	163.21	161.65	47.52	128.65	99.35	84.05	71.11	101.67	98.42
164	119.73	114.43	101.75	88.10	99.66	0.00	125.41	160.55	159.09	47.89	123.19	99.56	83.32	72.81	99.96	97.44
165	117.28	111.20	103.74	90.04	102.50	0.00	122.59	158.47	157.25	47.16	119.40	92.17	82.02	73.78	92.98	95.29

TIME	TC-46	TC-47	TC-48	TC-50	TC-51	TC-53	TC-54	TC-55	TC-56	TC-57
0	87.43	88.73	82.18	82.67	81.82	89.83	82.17	82.26	79.85	81.88
1	87.61	80.47	82.47	82.85	82.73	89.38	82.03	82.28	79.18	82.18
2	86.88	88.52	81.56	82.23	82.26	88.54	81.56	81.65	78.39	81.17
3	86.60	88.51	80.64	81.03	81.05	88.02	80.98	81.01	77.83	80.13
4	86.02	81.04	83.44	84.38	84.82	90.90	82.49	83.23	80.32	82.63
5	87.87	88.30	81.72	82.19	82.85	88.89	81.90	82.48	79.21	80.91
6	87.39	89.09	81.32	81.53	82.09	88.73	81.61	81.92	78.82	80.53
7	87.50	88.20	80.44	81.33	81.80	88.31	81.62	81.87	77.71	78.58
8	87.97	88.56	80.89	81.84	82.43	89.06	81.84	82.14	78.50	79.74
9	89.08	89.11	81.52	82.10	82.61	89.62	81.51	81.96	80.00	80.88
10	87.83	87.57	79.78	80.34	80.91	88.13	80.33	80.80	78.03	79.05
11	87.33	88.27	78.31	79.57	79.69	87.35	79.80	80.13	78.96	77.58
12	88.08	88.42	78.67	80.18	80.52	88.24	80.03	80.58	77.28	78.15
13	86.47	82.42	74.51	74.61	75.24	83.63	76.86	77.06	73.52	74.07
14	83.58	81.22	73.28	73.75	73.87	82.60	75.99	75.99	72.44	73.07
15	82.52	81.38	73.48	73.79	73.72	82.25	75.25	74.84	72.00	73.50
16	82.67	80.85	72.85	73.15	73.17	81.97	75.15	75.09	71.10	73.08
17	82.18	78.58	70.60	72.50	72.74	81.78	74.18	74.09	69.12	70.89
18	83.69	77.71	69.80	71.67	72.24	82.55	75.13	75.28	69.04	69.35
19	83.61	78.69	71.18	70.75	71.23	81.60	74.97	75.10	70.25	70.17
20	84.22	80.45	72.56	72.27	72.18	81.88	75.35	75.18	72.51	71.44
21	85.23	82.91	75.09	74.78	74.43	83.53	76.64	76.17	75.87	74.52
22	85.09	83.65	75.85	75.70	75.46	83.69	76.90	76.62	77.00	75.38
23	85.30	84.07	76.25	76.58	76.43	84.07	77.29	77.02	77.52	75.75
24	85.25	84.62	76.82	76.87	76.71	84.14	77.17	77.17	78.18	76.28
25	84.98	85.06	77.27	77.54	77.46	84.32	77.38	77.34	78.08	76.87
26	84.65	84.78	76.84	77.19	77.15	84.18	77.00	76.89	78.18	76.68
27	83.73	83.61	75.71	76.11	75.88	83.45	76.11	75.84	77.67	75.50
28	83.71	83.64	75.75	76.09	75.86	83.49	76.17	75.80	77.48	75.58
29	83.65	83.56	75.50	75.87	75.66	83.35	76.03	75.71	76.84	75.35
30	83.65	82.63	74.83	75.86	75.75	83.21	76.07	75.87	75.78	74.39
31	84.33	81.84	74.13	75.66	75.81	83.31	76.78	76.86	75.19	73.44
32	85.33	81.78	73.84	76.36	76.38	84.05	77.53	77.79	75.05	73.16
33	84.71	81.07	73.34	73.88	74.62	82.91	76.16	76.30	74.00	72.47
34	83.23	78.45	71.88	72.52	72.91	82.07	74.95	74.76	71.91	70.43
35	83.61	78.10	70.33	71.18	71.62	81.43	74.91	74.84	70.06	69.19
36	83.89	77.83	70.48	70.79	71.02	80.97	74.51	74.51	69.75	68.52
37	83.71	77.62	70.31	70.28	70.50	80.84	74.15	74.13	69.16	67.67
38	83.42	76.48	69.22	69.28	68.88	80.11	73.53	73.43	68.37	67.70
39	83.27	75.59	68.18	68.66	68.37	79.78	73.30	73.19	67.00	66.38
40	83.39	74.74	66.85	68.50	68.48	80.12	73.12	72.90	66.06	65.47
41	83.35	74.74	67.03	67.87	68.24	79.63	72.69	72.52	65.62	65.28
42	83.02	75.10	67.48	68.03	68.83	79.28	72.38	72.12	67.88	66.68
43	82.67	75.85	68.13	67.82	68.00	79.08	72.26	72.08	68.61	68.81
44	82.16	76.83	69.06	68.22	69.15	78.87	71.96	71.76	69.44	67.84
45	81.83	77.10	69.23	69.07	69.30	79.04	72.04	71.81	69.98	68.74
46	82.53	78.48	70.60	70.54	70.67	79.68	73.10	72.83	72.03	70.44
47	82.14	78.70	71.74	71.44	71.45	80.05	73.59	73.42	73.42	71.53
48	82.03	81.48	73.55	72.83	72.82	80.74	73.95	73.82	74.58	73.13
49	82.03	82.18	74.28	73.88	74.04	81.25	74.27	74.14	75.78	74.05
50	81.84	82.88	75.01	74.88	75.03	81.52	74.44	74.62	76.19	74.65
51	82.17	82.84	75.07	75.18	75.39	81.70	74.78	75.05	76.56	74.82
52	81.63	81.84	74.02	74.49	74.83	81.11	74.14	74.33	75.48	73.97

TIME	TC-46	TC-47	TC-48	TC-50	TC-51	TC-53	TC-54	TC-55	TC-56	TC-57
53	81.60	81.43	73.42	74.13	74.57	81.05	73.93	74.19	74.59	73.30
54	81.83	80.38	72.50	73.58	74.19	80.82	74.08	74.50	73.70	72.01
55	81.54	77.96	70.02	71.48	72.23	80.06	73.15	73.69	70.55	69.51
56	80.67	76.28	68.47	69.58	70.48	78.14	72.42	72.63	68.47	67.50
57	80.52	75.04	67.04	68.63	69.35	78.84	71.90	72.05	67.15	67.02
58	80.77	74.51	66.55	68.03	68.48	78.48	71.31	71.46	65.81	66.80
59	80.63	72.87	65.28	66.89	67.44	77.46	70.12	70.51	62.41	65.28
60	80.18	69.32	61.42	64.36	65.78	76.26	69.04	69.38	61.83	60.00
61	77.80	67.42	59.78	60.50	61.78	73.68	68.05	66.11	58.14	60.00
62	76.03	64.58	57.34	57.87	59.03	71.51	63.59	63.50	55.65	57.65
63	74.85	62.80	55.55	55.84	57.14	70.53	62.49	62.54	53.59	55.46
64	73.81	61.83	54.14	54.38	55.75	69.02	61.12	60.89	52.39	54.11
65	74.66	63.02	55.35	55.55	56.25	68.48	61.31	61.14	54.49	55.23
66	80.21	71.74	64.08	64.22	63.97	76.05	67.47	67.02	63.68	63.80
67	81.71	77.40	69.73	68.89	67.75	77.93	70.47	69.87	70.38	69.66
68	80.38	78.10	70.42	69.02	69.09	77.51	70.28	69.80	71.40	69.83
69	80.07	78.55	70.73	69.91	69.85	77.60	70.78	70.17	72.11	70.12
70	79.84	79.84	72.06	71.62	71.49	78.18	71.45	71.20	74.11	71.72
71	80.28	81.30	73.87	73.18	72.88	78.48	72.04	72.17	76.23	73.68
72	80.52	83.47	75.84	75.39	74.84	80.32	72.69	72.94	78.08	75.46
73	79.30	83.39	75.56	75.13	74.80	79.73	72.20	72.35	76.75	75.40
74	79.93	85.24	77.48	77.73	77.27	81.09	73.34	73.73	78.97	77.50
75	79.84	85.05	77.20	78.20	77.27	81.57	73.87	74.38	79.35	77.19
76	80.04	83.88	76.18	77.27	77.18	81.61	73.85	74.42	78.48	76.27
77	79.53	82.33	74.50	75.85	75.84	80.17	72.84	73.07	76.68	74.49
78	78.36	81.35	73.54	74.71	74.91	79.55	72.26	72.45	75.53	73.40
79	78.24	80.12	72.28	73.71	73.82	78.04	72.09	72.28	74.28	72.30
80	77.69	78.44	70.72	72.23	72.71	78.32	71.40	71.37	72.22	70.59
81	78.43	77.48	69.63	71.14	71.68	78.28	71.06	71.27	70.97	69.48
82	78.98	76.75	68.84	70.21	71.04	78.27	71.34	71.58	69.58	68.01
83	80.06	77.08	69.31	70.85	71.18	79.08	71.91	72.23	69.44	68.28
84	81.67	78.01	70.11	71.70	72.04	80.35	72.87	73.18	70.24	68.11
85	81.49	77.77	69.86	71.36	71.71	80.06	72.73	72.83	70.23	69.09
86	81.53	77.48	69.54	71.12	71.20	80.02	72.67	72.84	69.73	69.14
87	81.33	77.10	69.21	70.95	70.92	79.23	72.03	72.08	69.31	68.97
88	81.38	76.84	69.13	71.04	71.02	79.23	71.99	72.00	69.33	68.80
89	80.77	76.50	69.07	70.16	70.15	78.50	71.45	71.44	68.36	68.75
90	79.79	77.63	68.84	69.20	69.27	77.47	70.84	70.47	71.46	69.20
91	80.61	84.08	87.17	89.83	72.02	104.72	104.72	100.36	73.83	72.21
92	85.36	106.85	89.85	92.88	74.11	234.84	166.12	158.46	76.57	74.08
93	84.25	116.61	109.87	85.62	77.16	278.43	205.77	199.62	79.48	76.76
94	106.55	127.27	121.75	88.15	80.29	328.27	247.24	242.05	81.76	79.16
95	120.54	132.27	132.15	98.53	82.01	369.24	287.67	282.81	81.82	79.63
96	134.88	134.54	129.16	94.48	78.73	369.65	301.90	299.06	77.17	76.35
97	150.41	135.73	130.23	85.78	79.58	372.00	305.36	303.48	78.01	76.68
98	162.36	134.60	126.41	84.67	79.88	371.23	307.30	303.66	76.53	75.45
99	171.82	132.03	126.15	81.51	78.41	368.84	302.63	302.44	72.67	72.61
100	181.55	130.61	124.56	89.47	76.73	370.22	305.90	305.19	71.28	71.12
101	180.48	138.02	132.81	88.81	75.77	409.20	328.97	327.04	70.82	70.63
102	202.51	148.86	144.06	89.62	76.32	452.36	368.92	363.31	70.56	69.78
103	214.30	158.67	156.85	88.32	75.23	494.40	409.83	401.90	69.76	69.37
104	226.34	170.54	176.78	87.84	75.16	537.22	453.40	444.02	69.48	69.44
105	243.82	184.87	187.08	87.14	75.39	583.47	503.21	501.99	69.41	69.34

TK	TC-46	TC-47	TC-48	TC-50	TC-51	TC-53	TC-54	TC-55	TC-56	TC-57
105	261.22	185.21	203.70	87.37	75.95	624.56	545.03	558.18	69.67	68.40
107	280.40	217.24	229.22	88.28	76.49	699.07	567.08	598.17	70.86	69.56
108	304.51	219.90	235.27	88.74	78.52	673.85	584.44	612.60	71.30	69.72
109	328.24	218.14	231.13	88.81	79.43	673.85	592.78	614.64	71.70	69.79
110	351.37	217.64	228.78	89.46	80.60	673.33	592.87	614.84	72.71	70.19
111	371.77	216.13	226.78	89.50	80.91	673.03	591.08	615.51	71.02	70.03
112	388.25	214.45	223.77	89.03	80.47	672.38	590.46	616.31	69.79	69.37
113	406.80	214.27	223.24	89.74	80.66	671.40	591.28	615.77	72.90	69.80
114	423.08	216.12	224.48	81.26	82.41	673.38	591.17	617.47	72.77	72.32
115	438.23	218.39	227.95	84.51	85.30	673.53	592.02	620.79	75.35	75.13
116	451.12	218.80	225.73	83.17	84.37	672.51	591.16	620.59	76.21	74.22
117	462.67	213.38	223.66	82.57	83.89	671.14	591.24	621.07	77.78	73.61
118	472.67	213.28	219.61	81.41	83.52	671.63	591.22	620.48	76.27	72.03
119	481.88	215.44	224.30	82.68	83.97	671.31	592.05	619.75	77.84	73.75
120	490.83	217.25	227.24	85.24	86.27	672.04	592.10	621.69	78.36	76.31
121	497.82	217.13	227.36	85.88	87.58	671.18	592.78	622.89	80.05	76.98
122	504.68	217.91	228.06	88.06	89.06	670.61	592.17	624.63	80.01	78.80
123	511.06	220.20	229.80	101.18	92.06	671.44	594.22	626.08	82.16	82.27
124	517.11	221.57	231.56	102.51	93.72	671.80	595.23	627.39	85.61	83.93
125	521.46	221.20	230.85	102.20	93.54	670.82	593.27	626.34	89.50	83.17
126	526.70	225.08	240.35	100.83	93.22	673.86	590.45	628.03	89.81	81.66
127	521.13	247.88	258.89	96.83	89.63	695.43	430.14	494.32	80.28	78.03
128	500.83	231.02	237.25	95.38	86.72	633.50	368.28	380.21	78.58	76.19
129	476.08	223.05	227.84	93.63	83.80	285.02	334.21	336.36	78.45	74.62
130	451.88	213.25	218.52	92.02	81.06	267.13	306.95	302.24	77.13	73.05
131	429.43	200.35	202.28	91.10	78.63	248.77	283.70	277.21	76.51	72.05
132	408.85	188.34	189.27	89.31	77.34	230.68	270.23	256.27	74.84	70.87
133	389.82	178.72	177.75	87.67	75.11	218.82	254.45	238.46	72.69	69.02
134	372.26	188.88	188.88	86.71	73.70	205.55	241.07	224.44	71.40	67.65
135	355.74	183.71	181.44	86.22	72.84	186.71	230.50	213.09	70.82	67.19
136	338.61	157.59	154.45	85.83	71.83	187.77	220.05	202.00	69.87	66.79
137	324.65	152.61	150.40	85.80	71.40	180.31	209.83	191.39	70.90	67.01
138	311.28	149.76	148.83	86.74	71.70	174.06	200.10	182.47	72.39	68.00
139	288.77	147.79	144.04	85.28	73.19	168.77	192.79	176.11	74.79	70.55
140	289.73	145.03	142.10	90.86	74.89	166.15	185.93	169.41	77.20	72.70
141	277.68	143.16	140.06	92.85	76.27	162.44	180.63	163.71	78.59	74.24
142	267.90	141.41	137.46	94.84	78.33	159.20	175.93	159.67	80.19	76.45
143	258.62	140.53	136.16	97.24	80.67	156.68	171.00	154.84	81.63	78.89
144	250.42	138.56	134.08	98.42	81.68	153.67	166.02	150.44	82.59	80.04
145	241.92	136.74	132.18	98.80	81.59	151.17	161.28	148.25	83.17	80.81
146	234.74	135.44	130.39	100.55	83.47	148.76	156.81	143.57	84.72	81.51
147	228.91	133.16	127.58	99.92	83.01	145.18	151.77	140.01	84.00	81.62
148	219.88	130.62	124.89	99.44	83.43	142.02	148.02	136.45	84.02	80.28
149	213.95	128.20	123.59	99.49	83.45	139.51	144.74	133.23	83.60	79.99
150	208.01	125.74	120.42	98.73	82.83	136.78	141.63	130.71	82.23	79.68
151	203.24	123.25	117.68	97.24	82.72	133.82	137.75	127.80	81.63	77.88
152	197.03	122.39	117.19	95.27	80.86	129.41	132.61	124.73	79.98	76.03
153	191.68	119.79	114.66	93.88	79.12	126.04	127.69	121.71	78.27	75.18
154	187.47	117.94	112.72	93.02	78.53	123.65	124.28	118.82	77.47	74.20
155	182.80	115.41	109.88	91.50	77.29	121.55	120.55	115.69	75.05	72.40
156	177.88	112.48	107.01	90.59	75.71	118.03	117.08	112.71	73.50	70.75
157	172.72	109.20	103.69	89.76	73.73	114.83	113.39	108.59	71.38	68.75

TIME	TC-46	TC-47	TC-48	TC-50	TC-51	TC-53	TC-54	TC-55	TC-56	TC-57
158	189.42	106.74	101.06	87.71	72.80	112.04	110.14	104.11	89.85	67.72
159	184.02	104.90	98.56	86.90	71.51	109.53	106.88	101.58	88.10	66.84
160	189.90	103.30	97.23	86.78	70.85	107.85	104.23	99.45	87.86	66.78
161	185.98	102.25	95.12	87.42	76.58	106.30	161.82	97.89	88.27	67.38
162	182.72	102.42	95.37	88.87	72.04	108.84	101.40	97.20	72.88	69.24
163	148.81	102.86	96.43	81.58	73.81	106.72	100.45	96.81	75.08	71.29
164	148.16	97.08	90.68	80.87	74.84	100.12	100.84	98.86	74.16	73.55
165	143.88	93.82	86.74	78.23	75.22	98.30	101.68	97.19	73.83	73.88



DEPARTMENT OF THE ARMY
US ARMY ENVIRONMENTAL CENTER
5179 HOADLEY ROAD
ABERDEEN PROVING GROUND, MD 21010-5401

SFIM-AEC-IEA

15 SEP 2006

MEMORANDUM TO Mr. Larry Downing, Defense Technical Information Center, 8725
John J. Kingman Road, Ft Belvoir, VA 22060-6218

SUBJECT: Downgrading from Limited Distribution to Unlimited Distribution

1. The following documents were reviewed, and it was determined by our Technical POC, Mr. Marty Stutz, that the distribution statement should be changed from limited to unlimited distribution.

a. AD Number: ADB073052: Development of Novel Decontamination Techniques for Chemical Agents (GB, VX, HD) Contaminated Facilities. Phase I. Identification and Evaluation of Novel Decontamination Concepts. Volume 1.
From Distribution Code 03 - US GOVERNMENT ONLY; DOD CONTROLLED to Distribution UNLIMITED, APPROVED FOR PUBLIC RELEASE.

b. AD Number: ADB073034: Development of Novel Decontamination Techniques for Chemical Agents (GB, VX, HD) Contaminated Facilities. Phase 1. Identification and Evaluation of Novel Decontamination Concepts. Volume 2.
From Distribution Code 02 - US GOVERNMENT AND THEIR CONTRACTORS to Distribution UNLIMITED, APPROVED FOR PUBLIC RELEASE.

c. AD Number: ADB093506: Development of Novel Decontamination Techniques for Chemical Agents (GB, VX, HD) Contaminated Facilities. Phase II. Laboratory Evaluation of Novel Agent Decontamination Concepts.
From Distribution Code 02 - US GOVERNMENT AND THEIR CONTRACTORS to Distribution UNLIMITED APPROVED FOR PUBLIC RELEASE.

d. AD Number: ADB087418: Development of Novel Decontamination and Inerting Techniques for Explosives Contaminated Facilities. Phase 1. Identification and Evaluation of Novel Decontamination Concepts. Volume 1.
From Distribution Code 03 - US GOVERNMENT ONLY; DOD CONTROLLED to Distribution UNLIMITED APPROVED FOR PUBLIC RELEASE.

e. AD Number: ADB125304: Pilot Plant Testing of Hot Gas Building Decontamination Process.
From Distribution Code 03 - US GOVERNMENT ONLY to Distribution UNLIMITED APPROVED FOR PUBLIC RELEASE.

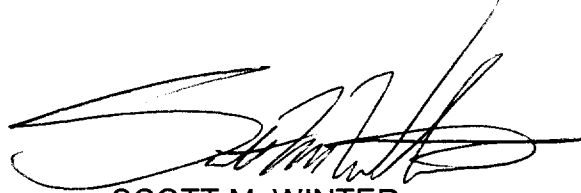
SFIM-AEC-IEA

15 SEP 2006

SUBJECT: Downgrading from Limited Distribution to Unlimited Distribution

2. The POC is Ms. Janet Wallen, USAEC Records Manager, 410-436-6317.

FOR THE COMMANDER

A handwritten signature in black ink, appearing to read 'Scott M. Winter', with a large, sweeping flourish extending to the right.

SCOTT M. WINTER

MAJ, CM

Security Officer