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DEVELOPMENT OF NOVEL DECONTAMINATION AND
INERTING TECHNIQUES FOR EXPLOSIVE CONTAMINATED
FACILITIES, LABORATORY EVALUATION OF CONCEPTS

Phase II. - Laboratory Evaluation of Novel Explosives
            Decontamination Concepts

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TASK FINAL REPORT FOR THE PERIOD AUGUST 1983 TO
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Prepared for:

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Study experimentally evaluated concepts, identified, evaluated, and recommended in a previous phase, for decontamination of explosive contaminated buildings. Five concepts were first submitted to laboratory scale screening studies. The successful operating conditions so identified were then further evaluated in bench scale studies using 5-inch x 5-inch coupons of typical building materials, both in a painted and unpainted condition. An engineering and economic analysis was then performed on the three concepts (Hot Gas, NaOH/DMSO Reduction,

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and combined Hot Gas/Chemical) so demonstrated to be effective. Remaining knowledge gaps pertaining to the implementation of these concepts were then identified and methods for resolving them delineated in a Field Test Plan. Keywords included:

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EXECUTIVE SUMMARY

The past manufacture or testing of explosives as well as the loading and/or unloading of munitions has resulted in buildings and their contents being contaminated with RDX, HMX, tetryl, TNT, 2,4-DNT and 2,6-DNT, the explosives commonly used in munitions. Reuse or even safe destruction of these buildings requires that they first be processed to remove this contamination. Consequently, the Army initiated this multi-phased program with the objective of identifying, evaluating, and developing novel processes to decontaminate such buildings and materials.

In the first phase of this program (Task 2), decontamination ideas were systematically developed into concepts which were then evaluated and rank ordered according to selected technical and economic factors. Five concepts were recommended for further evaluation in the second phase effort.

In this, the second phase of the Novel Processes Program, these five concepts were evaluated experimentally. Solubility of the explosives in various solvents as well as pertinent chemical reaction rates were determined in laboratory scale investigations. The applicability of the concepts for decontamination of building macerials (concrete, stainless steel, and mild steel) in both painted and unpainted conditions was then evaluated in bench scale experiments utilizing 5 x 5-inch coupons of these materials. Three concepts (Hot Gas, Chemical, and combined Chemical/Hot Gas were found to be capable of removing/destroying all six explosives contained within or one these building materials. These experimental results indicated that the combined concept (Chemical/Hot Gas) offers the best potential for reliable and highly effective decontamination. A detailed engineering and economic analysis indicated that the chemical concept would be the least expensive to use. However, since each concept offers discreet advantages and disadvantages and has a significant number of technical uncertainties that remain to be resolved, it was not possible to evaluate the trade-offs and select a single concept as the best. It is therefore recommended that all three concepts be carried on to Phase 3 pilot studies. A field test plan outlining such studies is included in this report.

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FINAL REPORT

for

TASK 4

DEVELOPMENT OF NOVEL

DECONTAMINATION AND INERTING TECHNIQUES

FOR

EXPLOSIVE CONTAMINATED FACILITIES

June 10, 1985

1.0 INTRODUCTION

Certain facilities located in Army installations were in the past used for the manufacture or testing of explosives and/or the loading of munitions. The explosives used in these operations include TNT, RDX, Tetryl, HMX, 2,4-DNT, and 2,6-DNT. As a result, these facilities are believed to be contaminated with these explosives, all of which are suspected carcinogens. Before these facilities can safely be restored and released for either restricted or public use, they must first be decontaminated. The development of novel concepts for the decontamination of explosive contaminated buildings is being carried out by Battelle Columbus Laboratories (BCL) for the United States Army Toxic and Hazardous Materials Agency (USATHAMA) under contract DAAK11-81-C-0101.

2.0 BACKGROUND

During the initial phase of this effort (Task 2), decontamination ideas were systematically developed into concepts for decontaminating buildings and equipment, evaluated, and rank ordered with respect to selected

1

technical and economic factors.[1] The five concepts so identified to be the most promising were then selected for further evaluation, and knowledge gaps pertaining to their implementation were identified.

The second phase (Task 4) of this effort was initiated to further develop these concepts. The Design Plan for this task, containing the USATHAMA Tasking Document, is attached to this report as Appendix I. As can be seen in this document, four experimental subtasks were planned to permit laboratory resolution of the knowledge gaps previously identified. This experimental effort has been completed and is summarized by subtask below. The subtask reports can be found in Appendix II.

2.1 Subtask 3 - Enhanced Aqueous Solubilization Studies

Various cosolvents, surfactants and complexing agents were evaluated for their ability to enhance the aqueous solubility of the six explosives. Surfactants were excluded from further consideration primarily because of the need to use unpractically high concentrations to achieve meaningful solubility enhancements. Complexing agents gave the highest observed solubility enhancements for TNT but were not considered further because of their lack of general applicability to the other five explosives as well as other potential chemical reactivity problems. The aqueous cosolvents DMSO and DMF, when used at concentrations of thirty percent or higher, were judged most suited for use as decontamination or extraction solvent systems and were recommended for further evaluation.

2.2 Subtask 4 - Prescreening of Chemical Decontamination

The cosolvent systems of water and dimethyl sulfoxide (DMSO) or dimethylformamide (DMF) judged most suitable in Subtask 3 were used for the evaluation of three chemical decontamination reactions. The decontamination effectiveness of solutions of sodium hydroxide, sodium sulfide, sodium disulfide and Fenton's Reagent were determined. The variables studied with 2,4-DNT, TNT and RDX were water to solvent ratio, temperature, and reagent concentration. The nature of the reaction products were also determined. As a

result of Subtask 4, the most acceptable chemical decontamination concept for explosives was judged to be a solution of sodium hydroxide in aqueous DMSO (30 or 75 percent DMSO). While a solution of sodium sulfide is even more effective as a decontaminant, it was rejected because of toxic characteristics of the reaction products and the sodium hydroxide system was recommended for further study.

2.3 Subtask 5 - Stainless Steel Surface Decontamination Screening

The concept of using hot gases to decontaminate stainless steel surfaces was evaluated for its effectiveness in decomposing/removing explosives contamination from stainless steel surfaces. Tests were conducted with 5-inch square stainless steel coupons spiked with 400 mg of one of the six explosives. These tests indicated that the removal of explosives from stainless steel by this concept occurs through a complex combination of vaporization and decomposition. While the less volatile explosives (HMX and RDX) are the most difficult to remove, sufficiently mild conditions (500 F for 1 hour) were shown to effect 99.97 percent removal of any of the six explosives. Consequently, hot gas treatment by these conditions was recommended for further evaluation on the more porous building material matrices.

2.4 Subtask 6 - Evaluation of Candidate Decontamination Concepts on Building Materials

The concepts recommended by the other subtasks were evaluated on more of the pourous building matrices, namely concrete and painted surfaces. Aspects of surface as well as subsurface contamination were addressed. While difficulties were encountered with analytically obtaining quantitative recovery of explosives from concrete, three concepts (Hot Gas, Chemical, and Combined Chemical/Hot Gas) were found with in this analytical limitation to be capable of removing/destroying explosives from the building materials of concern. Consequently these concepts were recommended for more detailed engineering and economic evaluations.

3.0 PURPOSE AND OBJECTIVES

The purpose of this engineering and economic analysis was to incorporate the results of the experimental subtasks into a coherent description of the processes that would be used to decontaminate actual buildings. The objectives pursued in performing this analysis were to:

- Produce P&I drawings for the processes
- Describe the equipment required in prototype configurations of the laboratory concepts
- Estimate the costs associated with decor;aminating model buildings considered representative of those exi~ting on Army installations
- Define all assumptions that were necessary to produce the design and cost estimation
- Identify any remaining knowledge gaps
- Recommend a best concept for subsequent field/pilot testing.

4.0 APPROACH

In performing this engineering and economic analysis it was necessary to expand the existing definition of the decontamination process as it would be applied to actual buildings. It was first necessary to determine the buildings that were considered to be in need of processing to remove explosives contamination and to define model structures which could serve as models for the design of the processes. The starting point was to review the information compiled in the Task 2 studies. The process descriptions in the Task 2 report and the previous Task 4 subtasks were then consulted and a piping and instrumentation (P&I) drawings were produced for each concept. These drawings were then used to define the major hardware needs for each of the three candidate concepts.

Next the equipment identified while producing the P&I drawing was sized to process the model structures. In this sizing of equipment, it was assumed that the important operating parameters were the steady state condi-

tions utilized during the experimental tests (e.g., the solvent application rate or temperature) and the time for which they were applied. In addition, engineering judgment was used to determine any additional limitations that should be placed on these conditions. The start-up time or the time required to achieve the steady state conditions was not considered an important parameter. Accordingly this parameter was not considered in sizing equipment except that care was taken to assure that the capability of the equipment and physical parameters such as heat transfer coefficients would produce startup times longer than those tested experimentally.

After defining operating scenarios and equipments lists, equipment manufacturers and standard sources of engineering information were consulted to determine the purchased equipment capital (PEC) costs for each concept. The manpower and materials needs were then estimated and the associated operating costs determined.

While this analysis is based on bench scale experimental results, it is important to remember that significant differences exist between these test conditions and the actual buildings that are contaminated and that all three concepts will require a significant pilot scale development program before they can be implemented. A field test plan detailing these testing needs has been written and can be found in Appendix I. However, it is anticipated that the costs associated with pilot studies will be similar for all three concepts and will therefore not change the relative economics of the concepts. Since the purpose of this analysis is to only make comparisons of the concepts, no detailed estimate was made of these similar development costs.

These cost estimates were based on the assumption that the hardware purchased would be used for a single building. In actual practice, multiple buildings of the same type could be processed with the same capital equipment. In addition, some overlap of equipment for different size buildings and types could also be realized.

5.0 RESULTS

The results of the economic engineering analysis are presented below starting with the selection of the model structures. Each of the three

concepts are then presented individually. The engineering analysis is presented by describing the process, the necessary calculations, the necessary building modifications, the hardware requirement, and finally the post processing building restoration needs. The process economics associated with this engineering analysis follows immediately th eafter.

5.1 Model Structures

Review of the trip reports from the Task 2 field survey as well as the final report of that task indicated that the buildings considered contaminated with explosives range in size from 400 to 40,000 square feet. The materials of construction varied from wood frame to brick and mortar. Performing an economic analysis for decontaminating every building type in this range would have exhausted the resources of this program. It was therefore decided to restrict this analysis to only two model structures. The Task 2 trip reports indicated that most of the wood frame structures surveyed were beyond hope of restoration. In addition, neither wood nor brick was included in the matrix of building materials studied in the experimental subtasks. Therefore, in selecting the two model structures for this analysis, the following assumptions were made:

- All wood and similar materials (including roofs and window frames) will be removed prior to the decontamination process
- Bricks, clay blocks, and other masonry materials will behave similarly to the concrete coupons studied
- All vaporized explosives and any noxious decomposition products can be destroyed by exposure to a 1600 F combustion environment for 1 second
- Decontamination in depth of the walls can be achieved by applying the conditions tested in Subtask 6.

Based on these assumptions and the information gleaned from the Task 2 trip reports it was determined that the range of building types that must eventually be decontaminated can be adequately reflected by performing the engineering and economic analysis on the following two model structures:

- Building 1 - A two-story 1000 square foot wood frame building that was constructed on a 1-foot thick concrete pad.
- Building 2 - A four-story 15,000 square foot building with 1-foot thick concrete walls and a wood and shingle roof.

5.2 Hot Gas Concept

5.2.1 Process Description

In the hot gas concept, hot gases produced by the combustion of an organic fuel are allowed to flow through the building heating the building and its contents to 500 F. These hot gases will either destroy or volatilize any explosive contamination and carry the volatile products away from the building. The time required to bring the entire structure to this temperature must exceed 2 hours and the steady-state temperature must be maintained for an additional hour. Since the explosives and possibly the products of decomposition are considered toxic, the gaseous effluents from the building must be treated to prevent the release of these volatile materials to the environment.

This concept will be applied to a building by performing minor modifications of the structures and installing the equipment illustrated in the P&I drawing shown in Figure 1. This equipment consists of four main pieces of hardware; a hot gas supply burner, an afterburner, an induced draft fan, and a heat exchanger.

5.2.2 Heat Balances

In order to estimate the effects and interactions of the major process variables during application of this concept to the model structures, a series of heat balances were performed for each building type. These calculations were based on an iterative unsteady state treatment of the heat transfer equations. An example of these calculations is presented in Appendix IV. The results are summarized in Table 1 (Building 1) and Table 2 (Building 2).
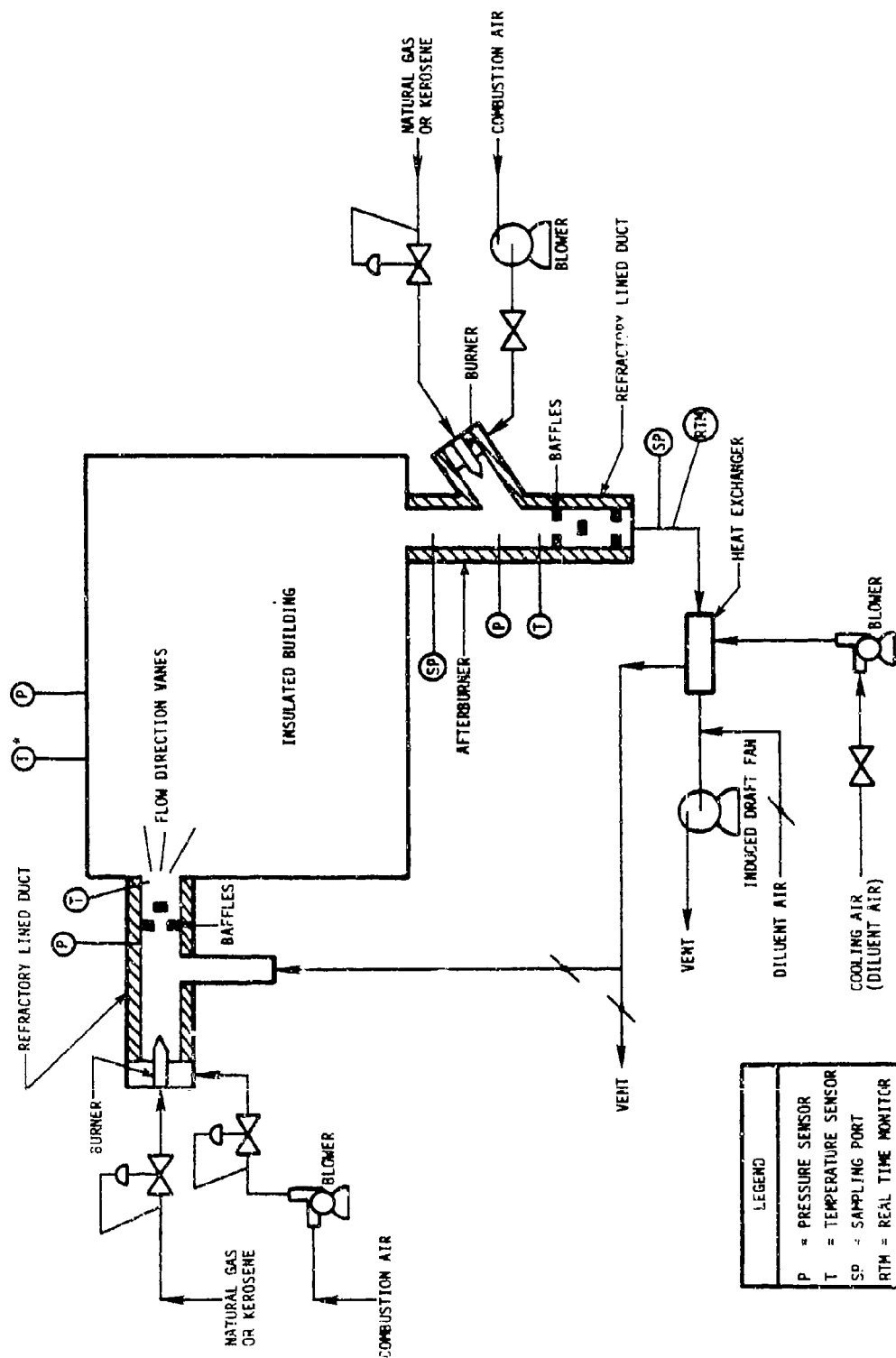
FIGURE 1. HOT GAS CONCEPT P&I DRAWING

* MORE THAN ONE TEMPERATURE PROBE WILL BE INSTALLED IN THE BUILDING.

LEGEND

P = PRESSURE SENSOR
T = TEMPERATURE SENSOR
SP = SAMPLING PORT
RTM = REAL TIME MONITOR

TABLE 1. RESULTS OF HEAT BALANCES FOR BUILDING 1

(Hot Gas Concept)

Hot Gas Flow Rate, SCFM	Inlet Gas Temperature, F	Insulation Thickness, Inches	Time to Reach(a) Temperature, Hours	Burner Heat Duty, MM BTU/hr	CH4 Required, (b) 1000 SCF	Wall(c) Temperature F
1000	1500	2	37	2.43	90	884
1000	1500	4	34	2.43	83	936
1000	1500	12	33	2.43	80	977
1000	2000	2	25	3.07	77	1118
1000	2000	4	24	3.07	74	1183
1000	2000	12	23	3.07	71	1221
2000	1500	2	25	4.37	109	1066
2000	1500	4	24	4.37	105	1111
2000	1500	12	24	4.37	105	1114
2000	2000	2	18	5.66	102	1266
2000	2000	4	18	5.66	102	1449
2000	2000	12	18	5.66	102	1490
4000	1500	2	20	8.26	165	1143
4000	1500	4	20	8.26	165	1259
4000	1500	12	19	8.26	157	1278
4000	2000	2	15	10.84	163	1622
4000	2000	4	15	10.84	163	1666
4000	2000	12	15	10.84	163	1695
8000	1500	2	18	16.04	289	1332
8000	1500	4	17	16.04	273	1352
8000	1500	12	17	16.04	273	1369
8000	2000	2	13	21.20	276	1777
8000	2000	4	13	21.20	276	1805

(a) Time required for the outside wall temperature to reach 500 F.

(b) Natural gas was assumed to be the fuel source.

(c) Inside wall temperature at end of heat up.

TABLE 2. RESULTS OF HEAT BALANCES FOR BUILDING 2

(Hot Gas Concept)

Hot Gas Flow Rate, SCFM	Inlet Gas Temperature, F	Insulation Thickness, Inches	Time to reach Temperature, (a) Hours	Burner heat Duty, MM BTU/hr	CH₄ Required, (b) 1000 SCF	Wall Temperature at End, (c) F
2000	2000	2	61	52.3	3190	766
2000	2000	4	51	52.3	2667	835
2000	2000	12	47	52.3	2458	886
3000	2000	2	42	78.2	3284	883
3000	2000	4	38	78.2	2972	953
3000	2000	12	36	78.2	2815	1000
5000	2000	2	29	130.0	3770	1051
5000	2000	4	27	130.0	3510	1113
5000	2000	12	27	130.0	3510	1168

(a) Time required for the outside wall temperature to reach 500 F.

(b) Natural gas was assumed to be the fuel source.

(c) Inside wall temperature at end of heat up.

To prevent excessive damage to the structures during processing, engineering judgment suggested limiting the temperature of the inside wall to a maximum of 750 F. Inclusion of this limit to the heat balances already presented, indicated that the most desirable combination of process variables for application of the hot gas concept to Buildings 1 and 2 are those presented in Table 3. These conditions represent the minimum conditions the process equipment must be capable of producing. As can be seen in this table, the limit on the wall temperature forces the selection of conditions that produces long heatup times.

5.2.3 Building Modifications

In order to process buildings with this concept, it is necessary to remove all wood and similar materials from the structure. As a consequence, Building 1, the two-story wood frame 1000 square foot structure will be reduced to the 40-foot long, 25 foot wide, by 1-foot thick pad on which it was constructed. Therefore in order to contain the heated gas atmosphere with which it is being decontaminated, a enclosure of steel plates must be constructed over the bare pad. This enclosure will be 2 feet high with the same rectangular dimensions as the pad itself. To minimize the heat losses, the plates are covered with an insulating material.

Building 2, the four-story 15,000 square foot building of concrete construction also contains wood construction which must be removed prior to being treated by the hot gas concept. This includes windows and frames as well as a wood and shingle roof. The roof as well as the windows are then replaced by steel plates, and the entire structure insulated. Due to the size and multiple floor construction of this building, additional modifications should be considered to enhance the distribution of the hot gasses and assure uniform heating of all surfaces. Such modifications might include construction of walls to serve as baffles and forming additional openings between floors to supplement existing openings such as stair wells. It is anticipated that the results of the recommended field studies will aid in this consideration.

TABLE 3. SELECTED OPERATION CONDITIONS (HOT GAS CONCEPT)

Building	Hot Gas Flow Rate, SCFM	Inlet Gas Temperature, F	Insulation Thickness, Inches	Burner Duty, MM BTU/hr	Heat Up Time, Hours	CH_4 Required, 1000 SCF
1	1000	1500	2	2.43	37	90
2	20,000	2000	2	52.3	61	3190

5.2.4 Process Equipment Systems

The major process equipment systems required by this concept are summarized in Table 4. A brief description of these systems is presented below.

5.2.4.1 Hot Gas Supply Burner.
This system supplies the correct volume of hot gas to the building at the desired temperature. As earlier shown in Table 1., Building 1 requires a 2.43 MM BTU/hr burner supplying 1000 SCFM of gas at 1500 F. Building 2 requires a 52.3 MM BTU/hr burner supplying 20,000 SCFM of gas at 2000 F.

For the purposes of versatility, portability and simplicity, this system will be capable of burning either fuel oil or gas, be skid mounted, and, in addition to the actual burner, contain all the necessary control systems. Consequently this system contains the following items:

- 1 - burner
- 1 - combustion air blower
- 1 - gas control valve
- 1 - control valve with a motor operator
- 1 - light-oil valve train
- 1 - gas pilot valve train with assembly
- 1 - combustion chamber
- 1 - flame control panel

Such combinations of hardware are available as skid mounted units from a large number of vendors. Information on a natural gas fired burner system manufactured by Eclipse was obtained from Steltner and Brink(2) and used for this analysis.

5.2.4.2 Afterburner.
The afterburner must be capable of exposing the entire gaseous effluents from the building being processed to a 1600 F combustion atmosphere for 1 second. For Building 1 this volume of gas is 1000 SCFM and for building 2 it is 20,000 SCFM. Since at start up these gases will be at or near ambient temperature, start-up will place the greatest demand on the afterburner. However as the processing interval draws to a close, the

TABLE 4.   HOT GAS CONCEPT EQUIPMENT REQUIREMENTS

Equipment System	Building 1	Building 2
Hot Gas Supply Burner Package	2.43 MMBTU/hr	52.43 MMBTU/hr
Afterburner Package	0.27MMBTU/hr	7.00 MMBTU/hr
Induced Draft Fan		
Impeller Diameter	15 inches	60 inches
Motor Size	60 hp	500 hp
Heat Exchanger	532 sq ft	10650 sq ft
Heat Exchanger Fan		
Impeller Size	21 inches	96 inches
Motor Size	4 hp	90 hp
Steel Plate	1260 sq ft	17240 sq ft
Ductwork		
1/8 inch stainless steel	40 ft 30 in ID	40 ft 148 in ID
1/8 inch carbon steel	40 ft 27 in ID	40 ft 120 inch ID
Refractory lining	1 in thick	1 in thick
1/2 inch carbon steel	10 ft 63 in ID	10 ft 90 in ID
Insulation -- 2 inches thick	1260 sq ft	"29000 sq ft
R value = 40		

temperature of the gases exiting the building will be approaching the after-burner operating temperature, this unit must also have a wide turn-down ratio. In addition this system must exhibit all the characteristics of and contain all the hardware common to the hot gas supply burner. Stelter and Brink provided information for this unit as well.

5.2.4.3 Induced Draft Fan. To provide the driving force necessary to move the required volumes of hot gases through the structure, the process equipment will include an induced draft fan. This fan will also keep the building and duct-work at 2 inches of water negative pressure to assure that no outward leaks of hot gases occur before they can pass through the after-burner. As the costs of fans capable of withstanding 1600 F are prohibitive, it is assumed that the gas will be cooled to a temperature lower than 600 F before entering the fan. Using a 50% over-design factor indicates that Buildings 1 and 2 will respectively require 10 and 180 horsepower fans.

5.2.4.4 Heat Exchanger. This is an air to air heat exchanger complete with its own cooling air fan. The main purpose of this heat exchanger is to reduce the temperature of the afterburner exhaust gases and thereby protect the induced draft fan. However, this heat exchanger can be used to preheat either the combustion or dilution air entering the hot gas supply burner. Utilizing this recovered waste heat to preheat the inlet air to the hot gas control burner will effect a fuel savings. This savings is slight and of itself would not justify the purchase costs of the heat exchanger.

The following assumptions were made in designing and costing this heat exchanger:

- There are no heat losses to the surroundings
- The overall heat transfer coefficient is 5 BTU/hr/sq ft
- The heat capacity of the afterburner effluents and the air supplying the cooling is 7.11 BTU/lb mole/F

Calculations based on these assumptions indicate that Building 1 requires a maximum heat exchanger area of 534 square feet. Building 2 requires a maximum area of 10,690 square feet.

5.2.5 Building Restoration

After completion of the decontamination process by the hot gas concept, the buildings will be partially restored by removing the steel plating and disconnecting the process equipment and preparing it for transportation to another location. The concrete surfaces will be sprayed with water by a garden hose in an attempt to overcome any deleterious dehydration effects produced by the thermal cycle. While it is likely, particularly in the case of Building 2, that a much more extensive restoration effort will be required, it was not possible to estimate the magnitude of these requirements with the existing data.

5.2.6 Economics

To determine the costs associated with implementation of this concept to decontamination of actual buildings, the purchased capital equipment costs for the equipment described above was first estimated. Estimates were then made of the materials and labor required to install and operate the process.

· 5.2.6.1 Purchased Equipment Capital Costs. The purchased equipment capital (PEC) costs include the process equipment systems described previously. The PEC costs were also assumed to include the steel plate and insulation needed in the building modification as well as the ductwork needed to interconnect the process equipment systems.

Initially an order of magnitude estimate was made of the costs of the individual hardware items. Then, whenever possible, actual equipment vendors were contacted to provide more precise estimates of these costs. The remaining items were costed in more detail using standard published sources[3,4,5,6]. The estimated PEC costs for this concept, summarized in Table 5, compare favorably with the initial order of magnitude estimate.

5.2.6.2 Operating Costs. Operating costs typically include labor, materials, and utilities required by a process. The estimated operating costs

TABLE 5.   HOT GAS CONCEPT CAPITAL EQUIPMENT COSTS

Equipment System	Building 1	Building 2
Hot Gas Supply Burner Package	$30,297	$48,927
Afterburner Package	$27,927	$27,927
Induced Draft Fan	$2,500	$29,600
Heat Exchanger	• $39,900	$245,000
Steel Plate	$3,528	$48,720
Ductwork		
1/8 inch stainless steel	$6,862	$67,730
1/8 inch carbon steel	$8,242	$41,060
Refractory lining	$4,625	$8,732
1/2 inch carbon steel	$1,042	$9,271
Insulation	$960	$132,200
Total Equipment	$125,883	$659,167
Design Costs – 25% of equipment $^{(a)}$	$16,915	$145,578
Vendors Profit – 15% of equipment $^{(a)}$	$10,149	$87,347
Total Purchased Equipment Costs	$152,947	$892,092

(a)   burner packages not included

associated with the Hot Gas Concept are summarized in Table 6. The labor estimates include labor required to moḍify the structure, install the process hardware, and restore the treated building as well as the labor required to operate the process. Labor associated with installation of refactory lining was included in the PEC cost of that item. All labor costs were estimated at a rate of $25/hr.

The materials needs associated with this concept are minor and include only items such as gaskets, caulking, and replacement insulation. Using the rule of thumb typical in the literature[4], these materials were estimated at 5% of the PEC costs.

The utility requirements of this concept are mainly fuel with minor electricity and water needs. Fuel requirements are kept high due to the long heatup time needed. Fuel was estimated to cost $10/Million BTUs. Electricity was estimated from the fan requirements at $0.05/kilowatt hour. No estimate of the minor water usage was made.

A significant effort will be required to sample and analyze the gaseous effluents during processing as well as the decontaminated building itself. These analyses, which are required to assure safe and complete decontamination will be better defined during the pilot scale studies.

5.2.6.3 Total Costs. The estimated total costs associated with decontaminating both model structures by the Hot Gas Concept are summarized in Table 7. Due to the high capital equipment costs, the total costs are relatively high, particularly for Building 2. If this building could be processed in segments permitting use of the equipment sizes utilized in Building 1, the costs could be reduced appreciably.

5.3 Chemical Destruction Concept

5.3.1 Process Description

Based on the experimental results reported in the Task 4, Subtask 6 report, the chemical destruction concept is defined as the process of spraying an aqueous based reagent on the internal surfaces of the contaminated

TABLE 6. HOT GAS CONCEPT OPERATING COSTS

Category	Building 1	Building 2
Labor ($25/hr)		
Modifications	100	300
Installation	100	100
Operation	40	65
Restoration	100	200
Total hrs	340	665
Costs @ $25/hr	$8,500	$16,625
Materials (5% of PEC)	$7,647	$44,605
Utilities		
Fuel @ $10/MMBTU	$972	$11,267
Electricity @ $.05/KWH	$21	$654
Total	$993	$11,921
Analytical Costs		
Sampling	$1,000	$3,000
Analysis	$2,000	$6,000
Total	$3,000	$9,000
Total Operating Costs	$20,140	$82,151

TABLE 7. HOT GAS CONCEPT COST SUMMARY

Cost	Building 1	Building 2
Capital Equipment	$152,947	$892,092
Operating	$20,140	$82,151
Total Concept Costs	$173,087	$974,243

structure for a period of four hours. It is anticipated the reagent selected was a 30% DMSO solution containing enough dissolved sodium hydroxide to produce 0.1 normality. In this experimental subtask, the reagent was applied as a finely divided spray to a 4-inch diameter circular area at a rate of 50 ml/min. This is equivalent to a spraying rate on a flat wall of 0.15 gal/min/sq. ft. Spray was applied at this rate for 5 seconds of every 5 minute interval. The 5 second/5 minute interval was selected in the Task 3 Diffusion Studies[7] as a procedure that minimized spray application and still provided sufficient reagent to keep the surface from becoming dry either by the reagent penetrating the wall or evaporating from the surface. This delivery rate and interval therefore represent minimum conditions at which reagent must be applied to the surfaces being decontaminated. If applied continuously the above rate equates to .15 gallons/hour/square foot or 0.0025 gallons/minute/square foot.

In addition to the type of reagent and the rate at which it must be applied, the previous experimental studies identified certain hazards associated with the use of this concept. Potentially toxic materials including the explosives themselves were found to be dissolved in the used reagent. Since direct incineration of the used reagent would be costly and produce excessive sulfur oxide emissions, all used reagent must be collected and cleaned up. Also, care must be taken to avoid exposing unprotected personnel to the used reagent due to the ability of DMSO solutions to carry dissolved materials through human skin.

The P&I drawing of the process developed for this concept is presented in Figure 2. As can be seen in this drawing, the process hardware consists of a reagent mixing and supply system, a spray delivery system, a used reagent collection system, and a reagent cleanup system. To provide mobility these systems will all be skid mounted.

5.3.2 Building Modifications

As in the hot gas concept, all wood and similar materials must be removed from the structures before processing. Similar enclosures must then be installed to contain the reagent. For this study it was assumed that the
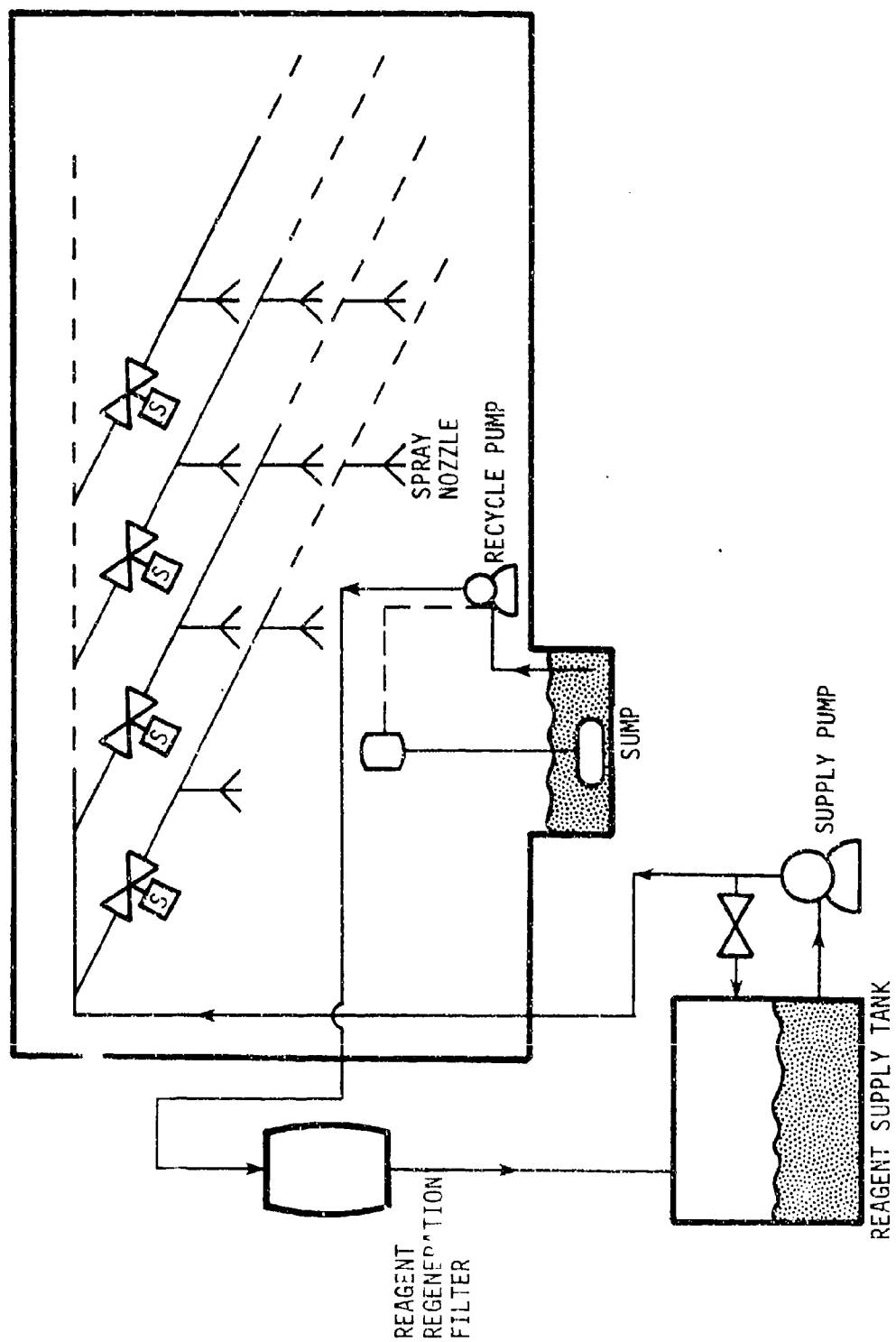
FIGURE 2. CHEMICAL DESTRUCTION P&I DRAWING

metal sheet used in the Hot Gas Concept would also be used here as well. However a plastic sheet made of a material such a Teflon® is capable of containing DMSO and might be cheaper to install.

Since most buildings of this type contain a sump, the used reagent will tend to collect there and the sump can most likely serve as the collection system. It will therefore be necessary to adequately clean the sump and any associated plumbing of debris to permit recovery of all reagent. If no adequate sump exists, one must be constructed.

5.3.3 Process Equipment Systems

In designing the process equipment systems for this process, it was assumed that the entire building would be processed simultaneously. For Building 2 this necessitates an extremely large and somewhat complex system. Options not selected include processing the building in segments such as one floor at a time and applying the spray by hand-held applicators. Future pilot studies should investigate the validity of this selection.

The equipment needs for this concept are summarized in Table 8. A brief description of the components is given below.

5.3.3.1 Reagent Mixing and Supply System. The main purpose of this system is to act as a reservoir for the the reagent to be sprayed into the building. A system of one or more stainless steel tanks can serve this purpose. This system also doubles as a mixing vessel for preparation of reagents. To prepare new reagent or regenerate used reagents, water, DMSO, and sodium hydroxide can be added to this tank in the proper proportions. As these materials easily go into solution, recirculation of reagent by the supply pump should provide adequate mixing. This system must be skid mounted limiting the individual tank size to 3300 gallons. The total tank volumes required to compensate for reagent retention by the buildings are 3000 gallons for Building 1 and 9000 gallons for Building 2.

The system must be capable of withstanding caustic corrosion. Either stainless steel or lined mild steel may be used. As the costs are similar and an uncertainty associated with the ability of DMSO to penetrate the liner, stainless steel was selected.

TABLE 8. CHEMICAL CONCEPT EQUIPMENT REQUIREMENTS

Equipment System	Building 1	Building 2
Supply Pump (316 ss)	50 GPM @ 20 psig	50 GPM @ 20 psig
Recycle Pump	50 GPM @ 20 psig	50 GPM @ 20 psig
Interval Timer	none	30 intervals
Reagent Supply Tanks - Stainless	1 - 3300 gal	3 - 3300 gal
Control Valves	1	48
Nozzles & Ball Fittings	4	600
Piping (one inch ss)	1000 feet	10000
Scaffolding & Safety	na	na

5.3.3.2 Spray Delivery System. This system is responsible for delivering the reagent to the building surfaces at the proper rate in a relatively uniform manner. It includes a pump to move the reagent, a timer to control spraying intervals, nozzles to control rate and distribution, and a myriad of piping to interconnect the pump and nozzles.

The heart of the spray delivery system is the spray nozzles. The nozzles must provide a finely divided spray at a minimum rate of 0.0025 ml/minute/square foot. A Spraying System 1/2 HH45WSQ nozzle will deliver 7 gallons/minute(GPM) to a square area of 400 square feet when operating at 20 psig. This equates to 0.0175 GPM/sq ft. While this is considerably in excess of the minimum required rate, it was selected because nozzles delivering lower rates tend to plug easily and the excess spray will be collected and returned to the process. Each nozzle will be attached to the required plumbing with a ball joint to permit aiming each nozzle at particular surface areas.

To estimate the number of nozzles required for each structure, it was assumed that a single nozzle could cover a 20x20 foot square area. The building configuration was then examined to provide nozzle locations that would assure coverage of all walls, ceilings and floors. For Building 1, this simply means directing four nozzles directly downward on the 25x40 foot concrete pad. Building 2 is much more complex containing 4 stories each with diminsions of 50 feet wide, 300 feet long, and 20 feet high, encompasses 44,000 sq. ft. of surface area per story. This would require a minimum of 110 nozzles/story. To provide for overspray and internal walls, a need of 150 nozzles/story was estimated.

The nozzles are assummed to be grouped in combinations of at least two and no more than eight. Each combination of nozzles is interconnected with 1 inch schedule 40 stainless steel pipe and will include a normally closed solenoid valve. The entire nozzle system will be connected to the supply pump by additional 1-inch pipe.

The supply pump must be capable of delivering 50 gpm. The pump must not only deliver 20 psig to the nozzles, but over come a head of up to 80 feet as well. Pumps that deliver the rather low capacity required by this operation and are capable of overcoming this total net positive head are not easily obtainable. For these reasons a 1 hp Model SC-100-100V centrifugal pump was

selected for both buildings. This pump will deliver in excess of 50 gpm at 30 psig. It is also made of 316 stainless steel and is equipped with an explosion proof motor.

Building 1 can be sprayed continuously. However, to avoid the need for an excessively large pump, it is envisioned that each grouping of nozzles in Building 2 will be activated for 15 second periods over intervals not to exceed 5 minutes. This interval will be controlled by a timer operating the solenoid valves on each group of nozzles. Timers are available capable of switching 30 such systems. To avoid damaging the distribution pump one solenoid must be open whenever the pump is operating.

5.3.3.3 Reagent Collection System.

This system serves to collect the excess reagent that does not penetrate the surfaces to which it is applied. It is assumed that the building will contain a sump that can serve as the collector. If a sump does not exist in the building, one can easily and economically be constructed.

To move reagent collected in this sump back to the supply tank, a pump identical to the distribution pump will be installed, external of the sump. A level float in the sump will activate this pump when the sump is full. A manual control system should also be available to activate this pump when ever desired.

5.3.3.4 Reagent Cleanup System.

Before being returned to the supply system, used reagent must be stripped of explosives and decomposition products that are dissolved in it. It is also anticipated that an appreciable amount of dirt and other mpurities will collect in this reagent. It is assumed that this stripping function can be safely performed by a charcoal filter installed in the return line between the sump and the supply reservoir. If pilot studies indicate that a different filter material such an ion-exchange resin is preferred, the expected economic impact is minor.

In this estimate the filter is sized to collect all such impurities that are anticipated to accumulate in one days operation. Carbon is assumed to absorb 0.2 lbs of organic/lb of carbon. The maximum organic concentration on the walls is assumed to be less than 0.001 lb/square foot.

5.3.4 Building Restoration

As with the hot gas concept, this concept also requires removal of the process equipment and the temporary roofing and window barriers that were installed. The remaining cleanup needs are not well understood. It is assumed that spraying the treated walls with water will remove the unwanted residual reagent. As only a 4 hour processing period is specified, it is further assumed that switching from reagent to water for the remainder of the work day will suffice as a cleanup measure.

Decontaminating thick concrete walks by this procedure may take even longer application and cleanup periods.

5.3.5 Economics

The costs associated with use of this concept presently defined were estimated in a manner similar to that described previously for the hot gas concept.

5.3.5.1 Purchased Equipment Capital Costs.

These costs were estimated by consulting either the literature cited earlier[3,4] or vendors catalogs. As with the Hot Gas Concept, the PEC costs included the steel plate for building modification as well as all the required hardware. As can be seen in the estimated PEC costs summarized in Table 9, the simple hardware associated with this concept results in very low capital cost requirements.

5.3.5.2 Operating Costs.

As with the Hot Gas Concept, $25/hr was used to cost the labor estimate. As can be seen in Table 10, the major materials cost are the costs of the reagent retained by the walls. This cost was estimated by using the quantity of reagent absorbed during the diffusion studies[5] to estimate the volume of reagent used and the current market prices of the DMSO and sodium hydroxide to replace it. The second major materials cost item was replacement charcoal for the filter. It was assumed that spent charcoal could be disposed in some safe manner and would be replaced as used. Additional costs were included for sampling and analysis of

TABLE 9.   CHEMICAL CONCEPT CAPITAL EQUIPMENT COSTS

Equipment System	Building 1	Building 2
Supply Pump	$2,000	$2,000
Recycle Pump	$2,000	$2,000
Interval Timer	$0	$1,000
Reagent Supply Tanks – Stainless	$10,700	$32,100
Control Valves	$800	$38,400
Nozzles & Ball Fittings ($25 ea)	$100	$15,000
Piping ($7.5/ft)	$7,500	$75,000
Scaffolding & Safety	$7,000	$36,000
Total Equipment	$30,100	$201,500
Fabrication (50% of equipment)	$15,050	$100,750
Total Purchased Equipment Costs	$45,150	$302,250

TABLE 10.   CHEMICAL CONCEPT. OPERATING COSTS

Category	Building 1	Building 2
Labor		
Modifications	50	150
Installation	200	500
Operation	12	12
Restoration	50	125
Total hrs	312	787
Costs @ $25/hr	$7,800	$19,675
Materials		
Reagents	$1,000	$3,000
Charcoal	$1,000	$3,000
Total	$2,000	$6,000
Utilities		
Water @ 1/1000 gal	$36	$144
Electricity @ $.05/KWH	$1	$1
Total	$37	$145
Analytical Costs		
Sampling	$5,000	$15,000
Analysis	$10,000	$25,000
Total	$15,000	$40,000
Total Operating Costs	$24,837	$65,820

the rea'ent, charcoal, and the structure surfaces. It was assumed that extensive sampling would be required by this concept. This estimate must be refined after the results of pilot studies are available.

5.3.5.3 Total Costs. The estimated costs for decontaminating the model structures by the Chemical Concept are summarized in Table 11.

5.4 Combined Chemical/Hot Gas Concept

5.4.1 Process Description

This combined process is defined as a brief application (1 hr) of the previously described chemical concept followed by hot gas treatment of 300 F for one hour. It embodies all the hardware systems previously described for both systems. It is anticipated that this combination concept can do a more complete job of removing explosive contamination with less damage to the building. Restoration operations should be appreciably less than either of the individual concepts.

The testing previously performed on this concept, while sufficient to demonstrate effectiveness, was limited. A single set of conditions was tested and these conditions, as well as the order in which they were applied may not be the optimum. The analysis described below was based on the assumption that the conditions tested would be those put into practice. Further study of this concept particularly as applied to thicker concrete surfaces is both needed and warranted.

5.4.2 Building Modifications

The required building modifications are identical to those previously described for the individual concepts. Due to the thermal cycle, use of steel plates rather than plastic sheets is required.

5.4.3 Heat Balances

Since the operating temperature of the hot gas portion of this concept is operated at only 300 F, it was necessary to generate a new set of

TABLE 11. CHEMICAL CONCEPT COST SUMMARY

Cost	Building	Building 2
Capital Equipment	$45,150	$302,250
Operating	$24,837	$65,820
Total Concept Costs	$69,987	$368,070

heat balances for both buildings. These balances, which were generated by the technique used in the hot gas concept and previously described in the discussion of that concept, are presented in Tables 12 and 13. The set of conditions selected from these heat balances to be the most desirable are presented in Table 14. Thicker insulation became more attractive as the lower operating temperature lessened the danger of overheating the inner wall.

5.4.4 Process Equipment Systems

This concept incorporates all the equipment systems common to both of the individual concepts. While the treatment conditions are less severe for the combined concept, the equipment size requirements remain the same.

5.4.5 Building Restoration

The experimental studies indicated that the thin film of sodium hydroxide remained on the coupons. In addition, a sulfurous odor, probably resulting from the decomposition of DMSO was observed. Rinsing the chamber with water removed both of these residues. It was then assumed that spraying water through the nozzle system for a minimum of 1 hour after the thermal cycle would accomplish this clean up. Removal of the process hardware and the remaining restoration operations are identical to the concepts.

5.4.6 Economics

Since this concept requires all of the capital equipment needed for both of the individual concepts, the PEC costs are simply the sum of those for the two concepts. Since the operating conditions for this concept are less severe than those of the individual concepts, some operating cost savings over the individual concept operating costs are realized. As shown in Table 15, these have been realized primarily through reduced fuel and reagent usage. It is likely that appreciably more savings will be observed when a better determination is made of the building restoration requirements of all the concepts. The total costs estimated to be associated with the use of this combined concept are summarized in Table 16.

TABLE 12. RESULTS OF HEAT BALANCES FOR BUILDING 1

Hot Gas Flow Rate, SCFM	Inlet Gas Temperature, F	Insulation Thickness, Inches	Time to Reach Temperature,(a) Hours	Burner Heat Duty, MM BTU/hr	CH₂ Required,(b) 1000 SCF	Wall Temperature at End,(c) F
1000	1500	2	20	2.43	49	784
		4	19	2.43	46	825
		12	19	2.43	46	855
	2000	2	15	3.07	46	1003
		4	15	3.07	46	1062
		12	15	3.07	46	1095
2000	1500	2	15	4.37	66	990
		4	14	4.37	61	1022
		12	14	4.37	61	1047
	2000	2	12	5.66	68	1306
		4	12	5.66	68	1359
		12	12	5.66	68	1389
3000	1500	2	13	6.32	82	1098
		4	13	6.32	82	1136
		12	13	6.32	82	1159
	2000	2	10	8.25	83	1452
		4	10	8.25	83	1495
		12	10	8.25	83	1521
4000	1500	2	12	8.26	99	1167
		12	12	8.26	99	1199
		12	12	8.26	99	1220
	2000	2	10	10.84	108	1562
		4	10	10.84	108	1602
		12	10	10.84	108	1626

(a) Time required for the outside wall temperature to reach 300 F.

(b) Natural gas was assumed to be the fuel source.

(c) Inside wall temperature at end of heat up.

TABLE 13. RESULTS OF HEAT BALANCES FOR BUILDING 2 (COMBINED CHEMICAL/HOT GAS CONCEPT)

Hot Gas Flow Rate, SCFM	Inlet Gas Temperature, F	Insulation Thickness, Inches	Time to Reach Temperature,(a) Hours	Burner Heat Duty, MM BTU/hr	CH_2 Required,(b) 1000 SCF	Wall Temperature at End,(c) F
4000	1500	2	+300	8.26	--	253
4000	1500	4	271	8.26	2238	352
4000	1500	12	140	8.26	1156	387
4000	2000	2	+300	10.84	--	325
4000	2000	4	124	10.84	1344	386
4000	2000	12	96	10.84	1041	421
6000	1500	2	+300	12.15	--	328
6000	1500	4	122	12.15	1482	387
6000	1500	12	94	12.15	1142	420
6000	2000	2	107	16.02	1714	383
6000	2000	4	76	16.02	1218	431
6000	2000	12	66	16.02	1057	463
8000	1500	2	133	16.04	2133	372
8000	1500	4	86	16.04	1379	417
8000	1500	12	73	16.04	1171	449
8000	2000	2	70	21.20	1484	429
8000	2000	4	57	21.20	1208	472
8000	2000	12	52	21.20	1102	503
10000	1500	2	90	19.93	1794	402
10000	1500	4	68	19.93	1355	443
10000	1500	12	60	19.93	1196	473
10000	2000	2	54	26.38	1425	466
10000	2000	4	47	26.38	1240	509
10000	2000	12	44	26.38	1161	541

TABLE 13. (Continued)

Hot Gas Flow Rate, SCFM	Inlet Gas Temperature, F	Insulation Thickness, Inches	Time to Reach Temperature,(a) Hours	Burner Heat Duty, MM BTU/hr	CH2 Required,(b) 1000 SCF	Wall Temperature at End,(c) F
12000	1500	2	70	23.81	1667	426
12000	1500	4	57	23.81	1357	467
12000	1500	12	52	23.81	1238	497
12000	2000	2	45	31.56	1420	499
12000	2000	4	40	31.56	1262	541
12000	2000	12	38	31.56	1199	571
15000	1500	2	55	29.65	1631	460
15000	1500	4	47	29.65	1394	500
15000	1500	12	44	29.65	1305	530
15000	2000	2	37	39.34	1456	546
15000	2000	4	34	39.34	1338	590
15000	2000	12	32	39.34	1259	614
20000	1500	2	42	39.37	1654	509
20000	1500	4	35	39.37	1496	551
20000	1500	12	36	39.37	1417	578
20000	2000	2	30	52.29	1569	618
20000	2000	4	28	52.29	1464	662
20000	2000	12	27	52.29	1412	690

(a) Time required for the outside wall temperature to reach 300 F.
(b) Natural gas was assumed to be the fuel source.
(c) Inside wall temperature at end of startup.

TABLE 14. SELECTED OPERATING CONDITIONS
(Combined Chemical/Hot Gas Concept)

Building	Hot Gas Flow Rate, SCFM	Inlet Gas Temperature	Insulation Thickness, Inches	Burner Duty, MM BTU/hr	CH_4 Required, 1000 SCF
1	1000	1500	2	2.43	49
2	20,000	2000	12	52.3	1412

TABLE 15. COMBINED CHEMICAL/HOT GAS OPERATING COSTS

Category	Building 1	Building 2
Labor		
Modifications	100	300
Installation	250	550
Operation	25	30
Restoration	50	125
Total hrs	425	1005
Costs @ $25/hr	$10,625	$25,125
Materials		
Reagents	$300	$1,000
Charcoal	$300	$1,000
Misc	$2,000	$10,000
Total	$2,600	$12,000
Utilities		
Fuel @ $10/MMBTU	$510	$14,644
Electricity @ $.05/KWH	$5	$5
Total	$515	$14,649
Analytical Costs		
Sampling	$4,000	$12,000
Analysis	$8,000	$20,000
Total	$12,000	$32,000
Total Operating Costs	$25,740	$83,774

TABLE 16.   COMBINED CHEMICAL/HOT GAS COST SUMMARY

Cost	Building	Building 2
Capital Equipment	$198,097	$1,194,342
Operating	$25,740	$83,774
Total Concept Costs	$223,837	$1,278,116

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 Economic Comparisons

Comparisons of the costs estimated to be associated with decontaminating the model buildings by the three concepts are presented in Table 17. As can be seen, this cost estimation indicates that the Chemical Concept is markedly the least expensive to apply. The high capital costs associated with Hot Gas Concept make that concept as well as the Hot Gas more costly.

6.2 Decontamination Effectiveness

As delineated in the experimental subtask reports, the decontamination effectiveness is dependant upon the uncertainties associated with the analytical recovery of explosives from the building material matrices. It must further be pointed out that the number of tests conducted with the Combined Chemical/Hot Gas Concept was limited. However based on these results, the Combined Concept holds the highest potential for achieving a high degree of removal or destruction of the explosives that may be deposited on the building material matrices studied. Further, the Hot Gas Concept and the Chemical Decontamination Concept rank second and third in removing or destroying all six explosives. The Chemical Concept's relative position would slip further when depth of treatment is considered.

6.3 Building Restoration

The experimental work performed to date does not permit a quantitative judgement of the relative positions of this parameter. The less severe operating conditions of the Combined Chemical/Hot Gas Concept should minimize structural degradation and the need to clean up reagent and degradation products. The Chemical Concept most likely ranks second in this category.

TABLE 17.   CONCEPT TOTAL COST COMPARISONS

Concept	Building	Building 2
Hot Gas		
Capital	$152,947	$892,092
Operating	$20,140	$82,151
Total	$173,087	$974,243
Chemical		
Capital	$45,150	$302,250
Operating	$24,837	$65,820
Total	$69,987	$368,070
Combined		
Capital	$198,097	$1,194,342
Operating	$25,740	$83,774
Total	$223,837	$1,278,116

6.4 Recommendations

The engineering and economic analysis summarized in this r port has indicated that each concept has discreet advantages and disadvantages. For any single category it is possible to rank order the concepts but, with the present data base it is very difficult to evaluate the trade-offs and produce an overall ranking. Resolution of the knowledge gaps described in the Field Test Plan included in Appendix C is necessary prior to selecting a single best concept. It is therefore recommended that all three concepts be carried on to the pilot scale studies and selection of a best concept from these three be deferred until those studies are complete. If these knowledge gaps can be resolved successfully, with no major impact on the economics detailed in this report the chemical concept would be the best of the concepts evaluated.

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APPENDIX I

FIELD TEST PLAN

TEST PLAN

for

PHASE III PILOT STUDIES

for

DEVELOPMENT OF NOVEL DECONTAMINATION AND INERTING
TECHNIQUES FOR EXPLOSIVES CONTAMINATED FACILITIES

Contract DAAK11-81-C-0101

January 4, 1985

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1.0 INTRODUCTION

Certain facilities located in Army installations have in the past been used for the manufacture or testing of explosives and/or the loading of munitions. The explosives used these such operations include TNT, RDX, Tetryl, HMX, 2,4-DNT and 2,6-DNT. As a result, these facilities are believed to be contaminated with these explosives which are suspected carcinogens. Therefore, before these facilities can safely be restored and released for either restricted or public use, they must be decontaminated. The development of novel concepts for the decontamination of explosive contaminated buildings is being carried out by Battelle-Columbus Laboratories (BCL) for the United States Army Toxic and and Hazardous Materials Agency (USATHAMA) under contract DAAK11-81-C-0101.

2.0 BACKGROUND

In the initial phase of this effort (Task 2), decontamination ideas were systematically developed into concepts for decontaminating and inerting buildings and equipment[1]. These concepts were then evaluated and ranked with respect to selected technical and economic factors. The five concepts determined to be the most promising were then selected for further evaluation. Technical knowledge gaps pertaining to the implementation of these concepts were identified and the second phase (Task 4) was initiated to permit laboratory resolution of these knowledge gaps in the laboratory[2].

As a result of the laboratory and bench scale experiments conducted in Task 4[3,4,5,6] the Hot Gas, Chemical Destruction, and Combined Hot Gas/Chemical concepts have been found to be capable of removing/destroying explosive from common building materials[6]. In addition, a detailed engineering/economic analysis is being performed on these three concepts as they are defined by the experimental results.[7] While the above studies have demonstrated the viability of these three concepts, they have also identified additional knowledge gaps that still

exist. These knowledge gaps as well as any assumptions made i
engineering analysis need to be resolved in pilot scale studies prior to
implementation of these approaches to the decontamination of actual
buildings.

3.0 PURPOSE AND OBJECTIVES

The purpose of this test plan is to better define the
knowledge gaps that still exist as well as the assumptions that were
necessary to complete the engineering analysis and give a descriptive
outline of the type of tests necessary to resolve them. The objectives
of these tests are to:

- Determine if the contamination effectiveness demonstrated
  on the laboratory scale can be achieved on an actual
  structure.
- Evaluate the effects of application of these processes on
  the integrity of actual structures.
- Provide sufficient information to finalize the process
  designs and select a best concept for specific structures.

4.0 TECHNICAL APPROACH

While each concept has knowledge gaps specific to its applica-
tion, there are also several general knowledge gaps remaining that apply
to the application of any concept. In the following discussion those
general knowledge gaps are addressed first before the discussion of the
individual concepts.

4.1 Analytical Methodology

4.1.1 Analytical Method

During the Task 4 studies, the quantity of explosives con-
tained in coupons of building materials was determined by first

extracting the explosives from the coupons with a solvent and t
analyzing the extract by HPLC[5]. When applied to concrete, the
recovery of explosives by this method was shown to be variable and a
strong function of the concentration of explosives applied to the
coupon. Consequently, this method as it is presently in use is not
considered adequate to certify decontamination of a building.

In other studies[8] a method based on UV fluorescences has
been used successfully to estimate the extent of surface contamination.
This method may have application to this analysis as well. However,
before proceeding with any additional studies it will first be necessary
to demonstrate that one of these analytical methods is reliable for
determining both the level of contamination that exists within walls of
the structures, as well as the degree of decontamination that is
achieved by application of any of the three concepts.

4.1.2 Sampling Method

In the previous studies, the entire coupon spike with a known
amount of explosive was submitted for the analysis after treatment. In
order to determine the level of contamination that exists within the
walls of either an actual building or a structure used for pilot
evaluation, eit. · before or after treatment, a method of obtaining a
sample of the building matrix must be developed. This is particularly
true when addressing the issue of the depth to which of a porous matrix,
such as conc·ete, is contaminated.

Water cooled saws are commercially available that are capable
of removing a cylindrical core sample from solid concrete. Such hard-
ware appears to have the potential of producing the samples required for
this study. However, testing is required to show that explosives con-
tained in the concrete are not destroyed by such a process.

4.1.3 Statistical Sampling

During the development of both the analytical and sampling
methods, a determination must be made by the Army as to the statistical

significance that will be placed on the data. This significanctly will determine the number of tests that must be conducted to certify both the analytical methods as well as the effectiveness of the decontamination treatment themselves.

4.2 Field Survey

In Task 2, a field survey was conducted of the Army facilities for structures that would be considered for decontamination. This survey, was subsequently used to select the two building types used in the engineering/economic analysis.[7] Before initiating the pilot studies, this survey needs to be expanded. While conducting this survey it is recommended that consideration be given to how well the processes and equipment configurations detailed in the engineering and economic analysis matches the actual structures surveyed. However the main purpose of this expanded survey would be to characterize the type, location, and degree of contamination that exist. Samples of the structure (walls, floors, etc.) should be taken and analyzed not only for the amount of explosive they contain, but also for the distribution of the contamination with the wall matrix. It is anticipated that this can be accomplished by dividing the samples into axial segments for separate analysis. Particular care should be given to determining if explosive contamination is located underneath a layer of paint. This information will be of great importance in determining the need for further investigation of the depth of decontamination that can be obtained by the three concepts.

4.3 Additional Common Knowledge Gaps

Besides the concerns of analytical methodology and the levels of contamination that must be treated, the three concepts share other common knowledge gaps. Since the test plan and facilities must address all of these knowledge gaps, they are listed and discussed briefly below. In some cases, more specific details are included the discussion of each of the individual concept.

4.3.1 Type and Age of Concrete

The effects of the composition as well as the age and history of the concrete structures on the decontamination process is unknown. The previous laboratory studies used cured but still only freshly poured concrete. The pilot studies should therefore compare several types of concrete walls including perhaps cinder block and brick. It is anticipated that these differences will effect heat and mass transfer within the walls, and their structural integrity after treatment.

4.3.2 Decontamination in Depth

In the laboratory tests, the procedures used in an attempt to study decontamination below the surface of concrete did not work. In addition, while adequate removal of the contamination was shown for all three concepts on 1/4 in. thick coupons, migration of explosives within the concrete was known to occur during processing. Since decontamination in depth was not proven, the concern that explosives contamination is being driven deeper into a wall rather than removed remains and must be further tested on the pilot scale. In the procedures and facilities used for the testing it must therefore be possible to place explosives below the surface or within a wall and either demonstrate they have been removed or, by a material balance, account for all explosives contamination either as products or intact explosives in an effluent. Accomplishing a closure of a material balance that is adequate to demonstrate the latter would appear to be highly unlikely based on experimental results to date.

4.3.3 Fate of the Products

During the previous studies, the final disposition of the products of decontamination was not determined and they could have remained within the concrete. However the product identification completed to date did indicate that some of the products themselves might be harmful. For example, in the case of Chemical Decontamination,

the products might include complexes of the explosives. The tests conducted and facilities used should be capable of addressing this issue as well. To be performed in detail, such a study would require a significant development of a method to determine the reaction products within the concrete matrix.

4.3.4 Structural Integrity

The concepts, particularly those involving application of heat, have the potential to significantly degrade the structure. To evaluate this, the tests and facilities must therefore adequately reflect the type of construction encountered in the structures that will eventually be decontaminated. Specifically there is concern for thermal stresses that might have occurred and how they may be controlled or relieved.

4.3.5 Process Optimization

In the Phase II studies, no attempt was made to actually optimize any of the concepts. Rather, a brief parametric study was made and then conditions that engineering judgement indicated to be preferred were further tested to demonstrate universal effectiveness. Selection of optimum conditions requires a more detailed parametric study.

4.4 Hot Gas Concept

4.4.1 Heat Up Schedule

It has previously been shown that heating 1/4 in. thick concrete coupon to 500 F and maintaining that temperature for 1 hour would adequately remove all six of the explosives. Further, it was possible to achieve that temperature in less than 2 hours. The heat up schedule predicted in the engineering and economic analysis is much longer and is based on empirical correlations to select both the thermal conductivity of the wall as well as the heat transfer coefficients to and from it.

It is anticipated that these values, particularly the thermal conductivity, is highly material dependent. It is necessary to determine experimentally the actual heat up rates that can be achieved with a matrix of building materials and wall thickness. These parameters must be selected to accurately reflect the buildings that are in need of decontamination. During these test particular attention should be given to the heat gradients that occur through the wall and whether uniform heating of the surfaces is achieved.

4.4.2 Stability of the Concrete

The available literature indicates that poured concrete should be able to withstand heating to 500 F. However, during the laboratory tests the concrete coupons became brittle after such heat treating. Whether such degradation will affect the integrity of walls and whether the initial strength can be restored by some process such as washing the walls with water remains to be determined.

An additional concern about the integrity of the walls that arises with the hot gas concept is the effect of the thermal stresses that will occur within a structure during decontamination by this process. The rectangular nature of a room will produce many areas of stress concentration, particularly in the sharp corners. In addition window and door frames as well as concrete reinforcing materials are commonly metallic and would exhibit thermal expansion markedly different than concrete. The likelihood of these factors significantly degrading a concrete structure during thermal processing must be evaluated.

4.4.3 Migration of Explosives

This knowledge gap is closely related to the general knowledge gap of decontamination in depth. It has been shown that while sufficient removal of explosives from 1/8 in. thick coupons can be achieved by hot gas treatment, some of the explosives migrated completely through the coupon. Tests must be designed to determine whether a similar inward migration would occur within a wall and if so, operating conditions would be required to overcome this problem.

4.4.4 Afterburner Design

For the engineering and economic analysis it was assumed that
a typical fume incinerator would suffice to destroy any explosives or
noxious products volatilized during hot gas treatment. It remains to be
demonstrated whether incineration conditions typical typical of these
units (1 second residence time at 1600-1800 F) produces sufficient
destruction.

4.4.6 Process Optimization

Several combinations of temperature and residence times were
shown to have potential, particularly with the more volatile explosives.
In selecting the preferred conditions, it was assumed that operating for
shorter periods at slightly higher temperatures would be more economi-
cal. While this is a reasonable assumption, it does not guarantee that
the conditions selected and proved to be effective is the optimum. This
is particularly true when explosives are considered individually or when
other factors, such as the potential for thermal degradation of the
structure, are better understood.

4.5 Chemical Decontamination Concept

4.5.1 Spray Nozzle Configuration

To permit approximation of the economics of this concept, a
spray nozzle configuration that appears reasonable was selected. The
type and orientation of the spray nozzles that will provide complete
coverage of the surfaces of a room while minimizing over spray must be
determined experimentally.

4.5.2 Reagent Penetration

The Phase II experimental work indicates that achieving
adequate penetration of concrete by the DMSO/NaOH/water reagent was

difficult. This result has a major impact on the decontamination depth general knowledge gap addressed earlier and could limit the effectiveness of the concept. However, if only surface contamination is encountered, this concern is abated. In any case, a more detailed evaluation of the reagent penetration is needed not only to better determine decontamination effectiveness, but also evaluate reagent usage. It is conceivable that different formulations, including perhaps addition of surfactants, will produce better penetration.

4.5.3 Reagent Regeneration

To clean up the recovered reagent for reuse and thereby minimize solvent consumption, it was assumed that the explosives and or their degradation products could be removed from the liquids by a charcoal or ion exchange resin filter. The validity of this assumption needs to be demonstrated. Also the breakthrough concentration as well as safety factors concerning explosivity and toxicity need to be given further consideration.

4.5.4 Process Optimization

As with the hot gas concept, a sufficient rather than an optimum process was selected. Factors such as the reagent composition have not been adequately evaluated, particularly with regards to the quantity and rate of reagent applied. Cleanup methods and effectiveness should also be factored into the optimization process.

4.6 Combined Chemical/Hot Gas Concept

This concept, which appears to hold the most promise, has the detriment of combining all of the knowledge gaps of both of the individual concepts that are discussed above. In addition, the testing of the concept was limited. Only one set of conditions with each of the two most difficult explosives was tested. Obviously, a more detailed matrix of tests is required to not only optimize the process but also prove

that all of the explosives can be removed by its application. The following additional knowledge gaps also remain.

4.6.1 Products

No attempt was made to determine the products of this process or their disposition. While no obvious problems appear likely, a residual odor, most likely from DMSO degradation was observed in the laboratory tests.

4.6.2 Clean Up

Closely related to the question of products is clean up. In addition to the previously mentioned odor, a white film assumed to be sodium hydroxide was left on the treated surfaces. A water rinse appeared to remove this film and decrease the odor. A more in depth study is required in this area.

5.0 FACILITIES

5.1 Test Structure

The test structure to be used to evaluate these concepts should closely model the structures that will eventually be decontaminated. While a better definition of these needs should be produced by the field survey, at this time it is assumed that these are represented by Building 1 and 2 described in the engineering analysis. The test facility should also permit the following:

- Instrumentation of the walls for determining temperature gradients.
- Spiking of test levels of contamination in a controlled manner reflecting types and levels determined by the field survey.

- Collection and containment of effluents that permeate the walls in order to both evaluate such permeation and prevent the spread of contamination.

The obvious options are to adapt an existing facility or to construct a new facility specifically designed for those tests on an Army installation. None of the building described in the previous field survey[1] appears adequate for these tests. However, a large quantity and variety of sack sumps, cess pools, french drains, and leaching pits do exist on Army installations[9]. Of these the sack sumps, with concrete walls and floors appears the most attractive and in fact might closely model the concrete basement that constitutes Building 1. The prior use of these sumps raises concerns about their applicability for these tests. Continued exposure to water and or silt would most likely produce physical characteristics in the concrete that is much different than the characteristics common to a concrete wall or basement. In addition there is a high probability that these sumps are already contaminated with unknown levels and types of explosives making controlled experiments difficult. A further concern is that the geometry of these sumps would not adequately reflect the thermal stress that would occur when a rectangular building containing windows and doors as well as metal frames and reinforcing is heated to 500 F. A more in-depth evaluation of all of these concerns is required.

If a new facility was constructed, the ideal case would be to build a 10 ft cubical within a room of an existing facility. Besides more closely modeling Building 2, this room within a room would permit ease of instrumentation as well as collection and containment of the effluents. Ideally such a room would contain a door in one wall, a window in another, and possibly reinforcing rods in a third wall. Additionally, all or part of such facilities could be constructed of cement blocks or bricks as deemed appropriate by the field survey. A deficit of such constructed facilities is that they would be costly to build several to permit multiple testing. Further, either freshly poured concrete or new cement blocks, bricks and mortar, might not adequately reflect the older buildings.

The best option appears to be the use of both types of facilities. One or two new rooms could be constructed to permit initial testing of concepts in a well controlled and instrumented manner. Several sumps could then be utilized to produce the quantity of data that is most likely necessary to prove sampling and analysis procedures, demonstrate decontamination effectiveness in a statistically significant manner.

5.2 Hardware

The hardware required to test these concepts very closely resembles that described in detail in the engineering and economic analysis. Like that hardware, it should be skid mounted and capable of being transported to a number of sites. A creative selection of the test structure should eventually permit the direct application of the test hardware to facilities reclamation.

6.0 REFERENCES

(1) "Development of Novel Decontaminating and Inerting Techniques for Explosives Contaminated Facilities, Phase I Identification and Evaluation of Concepts", Contract DAAK-11-81-C-0101 to USATHAMA, July 1983, by H. P. Benecke, et al.

(2) "Design Plan for Task 4 - Development of Novel Decontaminating and Inerting Techniques for Explosives Contaminated Facilities", Contract DAAK-11-81-C-0101 to USATHAMA, August 29, 1983, by H. P. Benecke and D. R. Hopper.

(3) "Task 4, Subtask 3 - Enhanced Aqueous Solubilization of Explosives", Contract DAAK1-81-C-0101 to USATHAMA May 17, 1984, by H. P. Benecke et al.

(4) "Test Report for Task 4, Subtask 4 - Prescreening of Chemical Decontamination Concepts", Contract DAAK1-81-C-0101 to USATHAMA, July 6, 1984, by H. P. Benecke et al.

(5) "Test Report for Task 4, Subtask 5 - Stainless Steel Surface Decontamination by Hot Gases", Contract DAAK11-81-C-0101, to USATHAMA, August 3, 1984, by D. R. Hopper et al.

(6) "Test Report for Task 4, Subtask 6 - Evaluation of Candidate Decontamination Concepts on Building Materials", Contract DAAK11-81-C-0101, to USATHAMA, October 20, 1984, by D. R. Hopper and S. Lugibihl.

(7) "Final Report for Task 4, Phase II - Development of Novel Decontamination and Inerting Techniques for Explosive Contaminated Facilities", Contract DAAK11-81-C-0101, to USATHAMA, estimated for January 31, 1986, by D. R. Hopper et al.

(8) "Surface Sampling Techniques Using Ultra Violet Illumination", Report to USATHAMA - AMXTH-TE-CR-84308 (Nov. 1984).

(9) "Task 7 - Economic Evaluation of Alternative Incineration Options for Explosives Contaminated Soils at the Cornhusker Army Ammunition Plant", Grant Island, Report to USATHAMA, by Roy F. Weston Inc., September 1984.

APPENDIX II

DESIGN PLAN AND TASKING DOCUMENT

DESIGN PLAN

for

TASK 4

DEVELOPMENT OF NOVEL DECONTAMINATION AND
INERTING TECHNIQUES FOR EXPLOSIVES
CONTAMINATED FACILITIES

Contract DAAK11-81-C-0101

August 29, 1983

by

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TASK 4

DEVELOPMENT OF NOVEL DECONTAMINATION AND
INERTING TECHNIQUES FOR EXPLOSIVES
CONTAMINATED FACILITIES

SUMMARY

The development of novel concepts for the decontamination
and inerting of explosives-contaminated buildings is being carried out
by Battelle Columbus Laboratories (BCL) for USATHAMA under Contract
No. DAAK11-81-C-0101. In the previous phase (Task 2), ideas were
systematically developed into concepts for decontaminating and inert-
ing buildings and equipment. These concepts were evaluated and ranked
with respect to selected technical and economic factors and five were
selected for laboratory evaluation. The overall objective of Task 4
is to determine and compare the technical economic/feasibility of
decontaminating buildings by the most promising concepts selected in
Task 2.

Task 4 is divided into eight subtasks: a planning subtask,
a test preparation subtask, four experimental subtasks, an engineering
and economic analysis subtask, and a reporting subtask.

BACKGROUND

Decontamination or inerting of facilities previously used by
the Department of the Army for explosive manufacture or testing is
necessary to permit restoration of such facilities for alternate use
or to excess them safely. Included are the structures themselves plus
storage tanks, processing equipment, sumps, transfer systems, munition
loading/packing equipment, etc.

Materials of concern for which decontamination methods are
to be developed are:

- Stainless steel -- painted and unpainted.
- Mild steel - painted and unpainted
- Concrete - painted and unpainted.

The previous study identified and evaluated potential explosive decontamination concepts from both technical and economic factors. The concepts were then rank ordered, and BCL, in conjunction with USATHAMA, recommended five for laboratory evaluation in Task 4. It is anticipated that this effort will result in one to three explosive decontamination concepts being sufficiently evaluated such that recommendations can be made for field testing of the selected concept(s).

OBJECTIVES

The objectives of this task are to (1) produce sufficient data to technically validate the concepts selected from Phase I, (2) perform an engineering/economic analysis on the most promising of these, and (3) identify remaining knowledge gaps associated with the most viable concept(s) and prepare test plans to resolve them and/or evaluate the concept(s) in the field.

TECHNICAL APPROACH

The Task 4 effort is divided into eight technical subtasks as shown in the work breakdown structure (WBS) illustrated in Figure 1. The logic diagram of this task describing the sequence and interactions of the various subtasks is provided in Figure 2. The objectives of these subtasks and technical approaches to be used are described below.

Subtask 4.1 Task Planning

This subtask has dual objectives: (1) produce the overall work/resource plan for the task, and (2) review regulatory and safety

requirements concerning explosives contamination. As given in the
WBS, the following work elements comprise this subtask:

- Prepare design plan (this document)
- Prepare a regulatory/safety agency discussion plan
- Visit appropriate agencies.

In order to properly evaluate new concepts, knowledge of the
most recent regulatory and safety developments is required. For
example, the "5X dilemma" and the nature and definition of allowable
decontamination levels will be discussed with agencies such as the
Department of Defense Explosion Safety Board, the DARCOM Field Safety
Office, and the Department of Transportation. The plan for conducting
these visits will consist of a letter listing the recommended agencies
for visitation as well as those topics which will be discussed. This
plan will be pepared in cooperation with the COTR and then submitted
for USATHAMA approval by September 9, 1983. After receipt of this
plan, it is anticipated that USATHAMA will identify the staff to be
visited at the agreed-upon agencies and schedule the visits. Visits
will be made by a team consisting of at least one USATHAMA and two BCL
staff members. A report summarizing the visits and the findings of
the regulatory/safety review will be incorporated in a Subtask Summary
Report to be submitted by November 28, 1983.

<div align="center">

Subtask 4.2 Preparation for
Concept Evaluations

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The design and construction of all required experimental
equipment will be performed in this subtask. A test chamber will be
designed and constructed which will permit the controlled application
and evaluation of decontamination concepts on spiked coupons. A
design for a chamber in which to safely conduct the tests will be sub-
mitted to USATHAMA in a letter test plan format by September 9, 1983.
This chamber will be sized to fit within a laboratory hood and will be
fitted with entry/exit ports to permit the introduction of samples and
the removal of decontamination effluents.

TASK 4

LABORATORY EVALUATION OF

EXPLOSIVES DECONTAMINATION CONCEPTS

1.0 Task 4 Plan

- 1.1 Task 4 Design Plan
- 1.1.1 Approval by USATHAMA
- 1.2 Regulatory/Safety Agency Discussion Plan
- 1.2.1 Approval by USATHAMA
- 1.3 Agency Visits
- 1.4 Summary Report

2.0 Test Preparations

- 2.1 Chamber Design
- 2.2 Chamber Operation Test Plan
- 2.2.1 Approval by USATHAMA
- 2.3 Chamber Construction and Testing
- 2.4 Coupon Preparation
- 2.5 Analytical QA/QC Plan
- 2.5.1 Approval by USATHAMA

3.0 Enhanced Aqueous Solubilization Studies

- 3.1 Letter Test Plan
- 3.1.1 Approval by USATHAMA
- 3.2 Surfactants
- 3.3 Complexing Agents
- 3.4 Cosolvents
- 3.5 Letter Test Report
- 3.6 Approval by USATHAMA

4.0 Prescreening of Chemical Decontamination Concepts

- 4.1 Letter Test Plan
- 4.1.1 Approval by USATHAMA
- 4.2 Sulfur Reductants
- 4.3 Bases (DS-2)
- 4.4 Radicals
- 4.5 Letter Test Report
- 4.5.1 Summary of Results
- 4.5.2 Recommendations (Three Concepts)
- 4.6 Approval by USATHAMA

FIGURE 1. WORK BREAKDOWN STRUCTURE TASK 4

TASK 4, CONTINUED

5.0 Stainless Steel Surface Decontamination
- 5.1 Letter Test Plan
- 5.1.1 Approval by USATHAMA
- 5.2 Hot Gases
- 5.3 Vapor Circulation
- 5.4 Solvent Removal
- 5.5 Best Three Chemical Decomposition Concepts
- 5.6 Waste Product Determination
- 5.7 Letter Test Report
- 5.7.1 Summary of Results
- 5.7.2 Recommendations (Three Concepts)
- 5.8 USATHAMA Approval

6.0 Decontamination of Steel and Concrete
- 6.1 Letter Test Plan
- 6.1.1 Approval by USATHAMA
- 6.2 Evaluation of Three Highest-Ranked Concepts
- 6.3 Waste Product Verification (Concrete and Painted Steel)
- 6.4 Test Report
- 6.4.1 Summary of Results
- 6.4.2 Analytical Performance
- 6.4.3 Recommendations
- 6.4.4 Select Best One to Three Concepts
- 6.5 Approval by USATHAMA

7.0 Engineering/Economic Analyses
- 7.1 Engineering Analyses
- 7.1.1 Waste Disposal Alternatives
- 7.2 Economic Analysis
- 7.3 Knowledge Gap Identification
- 7.4 Test Report (Select or recommend the minimum number of concepts which will be applicable to all six explosives)
- 7.5 Approval by USATHAMA

8.0 Task 4 Report/Field Test Plan
- 8.1 Draft Final Report
- 8.1.1 Approval by USATHAMA
- 8.2 Final Task 4 Report
- 8.3 Field Test Plan
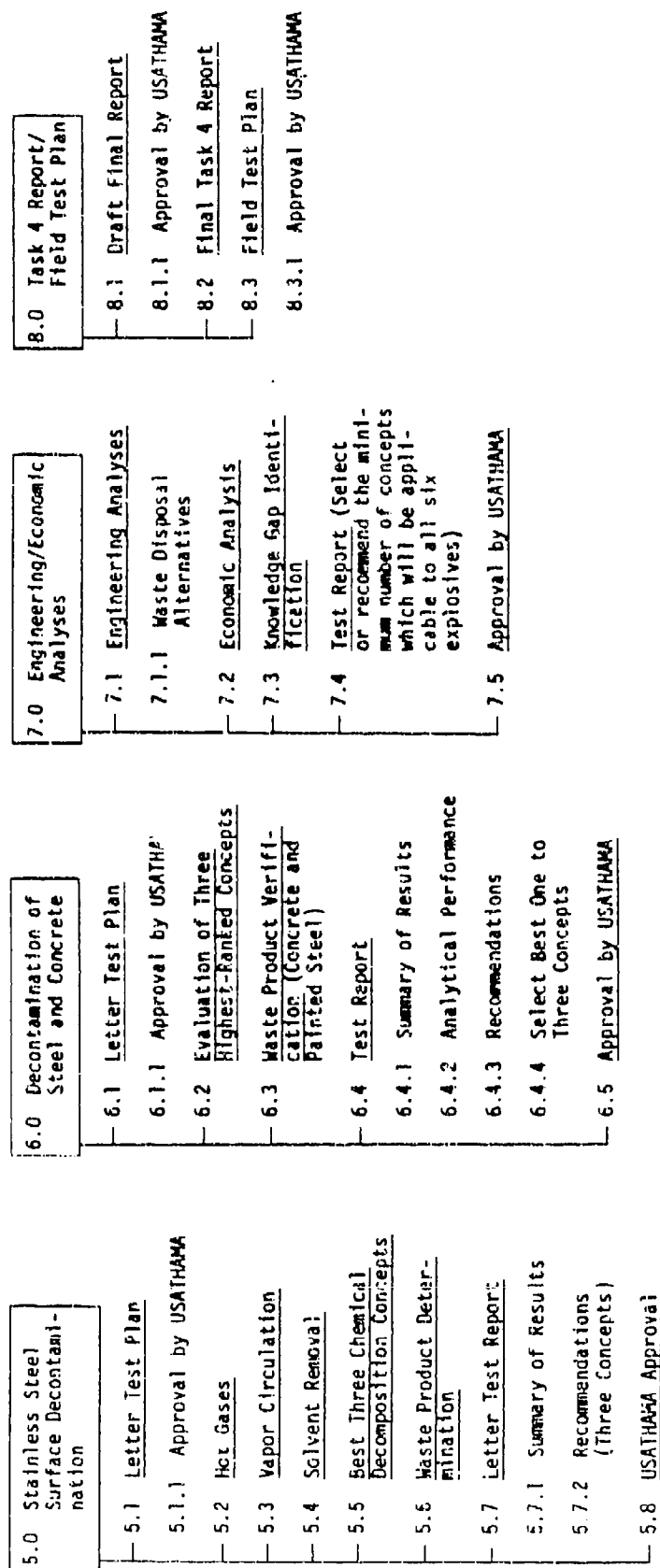- 8.3.1 Approval by USATHAMA

FIGURE 1. (Continued)

FIGURE 2.   TASK 4 LOGIC DIAGRAM

In the test program, coupons of the six relevant substrates will be contaminated with explosives and then subjected to various decontamination procedures. To provide a uniform surface area and permit direct comparison of the results, coupons of identical dimensions will be prepared and contaminated with explosives at levels which can be measured by existing analytical methods. These coupons will be prepared in this subtask.

The final work element of this subtask will be production of a quality assurance and quality control plan for the analytical methods. After receiving guidance from USATHAMA this plan will be prepared by September 19, 1983.

Subtask 4.3 Enhanced Aqueous Solubilization Studies

Solubilization of explosives is required for the effective chemical decontamination or the extractive removal of explosives. The best potential solvent for the solubilization of explosives in terms of minimum cost and safety hazards is water. However, those explosives which will be investigated in these Task 4 studies have low solubilities (about 0.01 percent) in water. The objective of this subtask is to evaluate those additives which may significantly increase the aqueous solubilities of various explosives. If it is found that these additives are required in major proportions for effective solubilization of explosives, then an alternative objective of this subtask will be to determine the optimum amount of water which may be incorporated in such a manner.

Those additives considered for investigation for increasing the aqueous solubilities of explosives include:

- Surfactants
- Complexing Agents
- Cosolvents

A Letter Test Plan will be submitted by September 2, 1983, which will identify those additives which will be examined and will

also detail the approach to be used in the experimental evaluation. The experimental results will be summarized in a subtask report to be submitted by October 4, 1983.

Subtask 4.4 Prescreening of Chemical
Decontamination Concepts

The decomposition effectiveness of the three highest-ranked chemical concepts identified in the previous Task 2 studies will be evaluated in this subtask. These evaluations will be performed in laboratory glassware under closely controlled conditions. Each concept will be evaluated under a variety of conditions in an attempt to determine the optimum operating conditions required to decompose each of the six explosives. The relative effectiveness of the three concepts under various conditions will be determined quantitatively by determining the decontamination rate constants. If conditions cannot be found for the effective decomposition of explosives by any one chemical concept, an alternate replacement concept will be chosen with USATHAMA approval and its effectiveness determined in the same manner.

The three concepts which will be initially evaluated are:

- Sulfur-based Reductants
- Based-initiated Decomposition
- Radical-initiated Decomposition.

Analytical methodology to determine explosives concentrations will also be evaluated for their applicability in the presence of the decomposition solutions and additives.

A Letter Test Plan will be submitted by October 21, 1983, and the results of this subtask will be discussed in a test report to be submitted by January 3, 1984.

Subtask 4.5 Stainless S   Surface
Decontamination Screening

The candidate decontamination processes will be further screened in this subtask. The three chemical concepts selected in

Subtask 4.4 along with the hot gas and vapor/solvent circulation concepts identified in Task 2 will be evaluated for their ability to decompose or remove explosives placed on the surface of stainless steel coupons. Stainless steel was selected because it is the substrate from which the analytical techniques achieve the highest recoveries and therefore yield the most accurate measure of the percentage of decontamination/removal achieved. Waste product determination will also be made.

A letter test plan detailing the test to be conducted will be submitted to USATHAMA for approval by October 28, 1983. The results of these tests will be incorporated in a letter test report to be submitted by February 10, 1984. This report will recommend three concepts to be further evaluated in subsequent subtasks. These recommendations will be based on both technical and economic factors.

Subtask 4.6 Evaluation of Decontamination Candidate Concepts on Steel and Concrete

This subtask, which represents the final test series in this task, will address aspects of sub-surface decontamination of concrete as well as surface decontamination of mild steel. This subtask will be oc ined in detail in a letter test plan to be submitted by January 20, 1 . The three concepts agreed upon after evaluation of the Subtask 4.5 data will then be tested for effectiveness on concrete and mild steel, simulating as closely as possible field decontamination scenarios. Decontamination versus depth into the material shall be verified on spiked concrete coupons. The reaction off-gases and other reaction products will be analyzed in an attempt to verify that the products formed are the same as formed on steel surfaces. A test report, to be submitted by April 6, 1984, will summarize the test results and select the best 1 to 3 concepts for analysis in Subtask 4.7. Potentially, this subtask could be moved up in the schedule and precede Subtask 4.5.

Subtask 4.7 Engineering and Economic Analysis

A detailed engineering analysis will be performed on the three best concepts selected in Subtask 4.6. Experimental data concerning reaction rate constants as well as reagent (solvent) and heat requirements generated in the previous subtasks will serve as the basis of this engineering analysis. This analysis will address the following factors: reagents and solvents, process equipment, support facilities, decontamination scheduling, waste disposal alternatives, and safety requirements.

After this analysis is completed and the necessary equipment identified and sized to decontaminate the model structure defined in Task 2, a detailed economic analysis will be performed. This analysis will include: building repair costs, development costs, utility and fuel costs, equipment costs, material cost, and manpower costs.

The results of this subtask will be summarized in a final subtask report which will recommend decontamination procedures to be used for decontamination of all six explosives. USATHAMA shall participate in the selection of the final concept(s). The report will be submitted to USATHAMA for review by June 15, 1984. Knowledge gaps that remain after ther above evaluations are completed will be identified and outlined as part of the report.

Subtask 4.8 Field Test Plan/Task 4 Report

A test plan will be developed for applying the selected treatment concept(s) in the field to decontaminate concrete and steel structures at Army installations. A sampling plan will be devised which will describe the types of samples and methods by which these samples be taken. This subtask will also develop a plan designed to experimentally resolve the technical uncertainties and knowledge gaps identified in Subtask 4.7.

The final work element will be the preparation of a final task report. It is anticipated that this report will consist of a

narrative and technical summary with the letter test plans and reports from the other subtasks included as appendi. material. This report will be submitted for review in draft form by July 27, 1984.

DEVIATIONS/ADDITIONS

This design plan generally follows the government tasking document and all 31 work units have been incorporated into the eight subtasks. In Subtask 4.5, Stainless Steel Surface Decontamination, the solvent extraction concept is to be evaluated in addition to the vapor circulation concept.

INTERFACING

The planning of Task 4 and the projected test plans will draw upon the results of the previous Task 2 results. In addition, some of explosives decontamination concept studies planned for Task 4 will parallel agent decontamination studies planned for Task 3. These parallel approaches will provide economies in the design and construction of the test chambers and coupons as well as permit direct comparison of the concepts being evaluated. For example, the solvent diffusion study of Task 3 can have significant impact on the performance of Task 4.

PERSONNEL

The Task Leader for Task 4 will be Herman P. Benecke. Engineering support will be provided by David R. Hopper and analytical support by Benjamin C. Garrett.

GOVERNMENT-FURNISHED PROPERTY/ASSISTANCE

The government will furnish 10 grams each of the following explosives for these studies: TNT, Tetryl, 2,4-DNT, 2,6-DNT, RDX, and HMX. They will first be needed for Subtask 4.3 which is to start about September 12, 1983. The government will make arrangements for meetings with regulatory/safety government agencies (Subtask 4.1).

SCHEDULE

The subtask and reporting schedule is shown in Figure 3. Open triangles identify BCL Milestones while closed triangles identify USATHAMA Approval Milestones. The overall period of performance is 60 weeks.

WEEK

	August			September			October			November			December			January		
	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
	1 8	15	22	29	5	12	19	26	3	10	17	24	31	7	14	21	28	5	12	19	26	2	9	16	23	30

TASK 4   NOVEL PROCESSING

1.0 Work/Resource Plan

  1.1 Design Plan

    1.1.1 USATHAMA Approval

  1.2 Regulatory/Safety Agency Review Plan

    1.2.1 USATHAMA Approval

  1.3 Agency Visits

  1.4 Regulatory/Safety Summary Report

2.0 Test Preparations

  2.1 Chamber Design

  2.2 Chamber Operation Test Plan

    2.2.1 USATHAMA Approval

  2.3 Chamber Construction and Testing

  2.4 Coupon Preparation
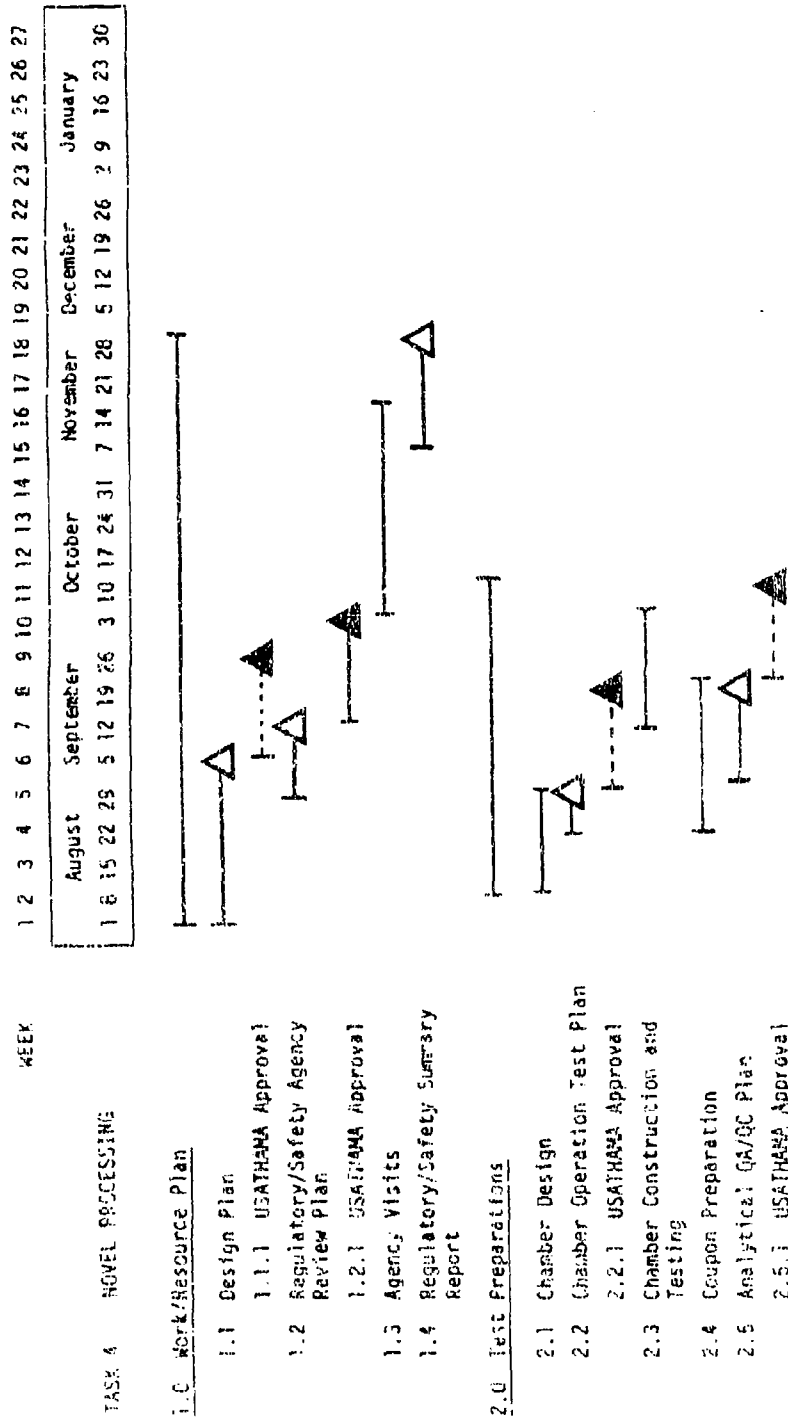
  2.5 Analytical QA/QC Plan

    2.5.1 USATHAMA Approval

FIGURE 3. SCHEDULE FOR TASK 4
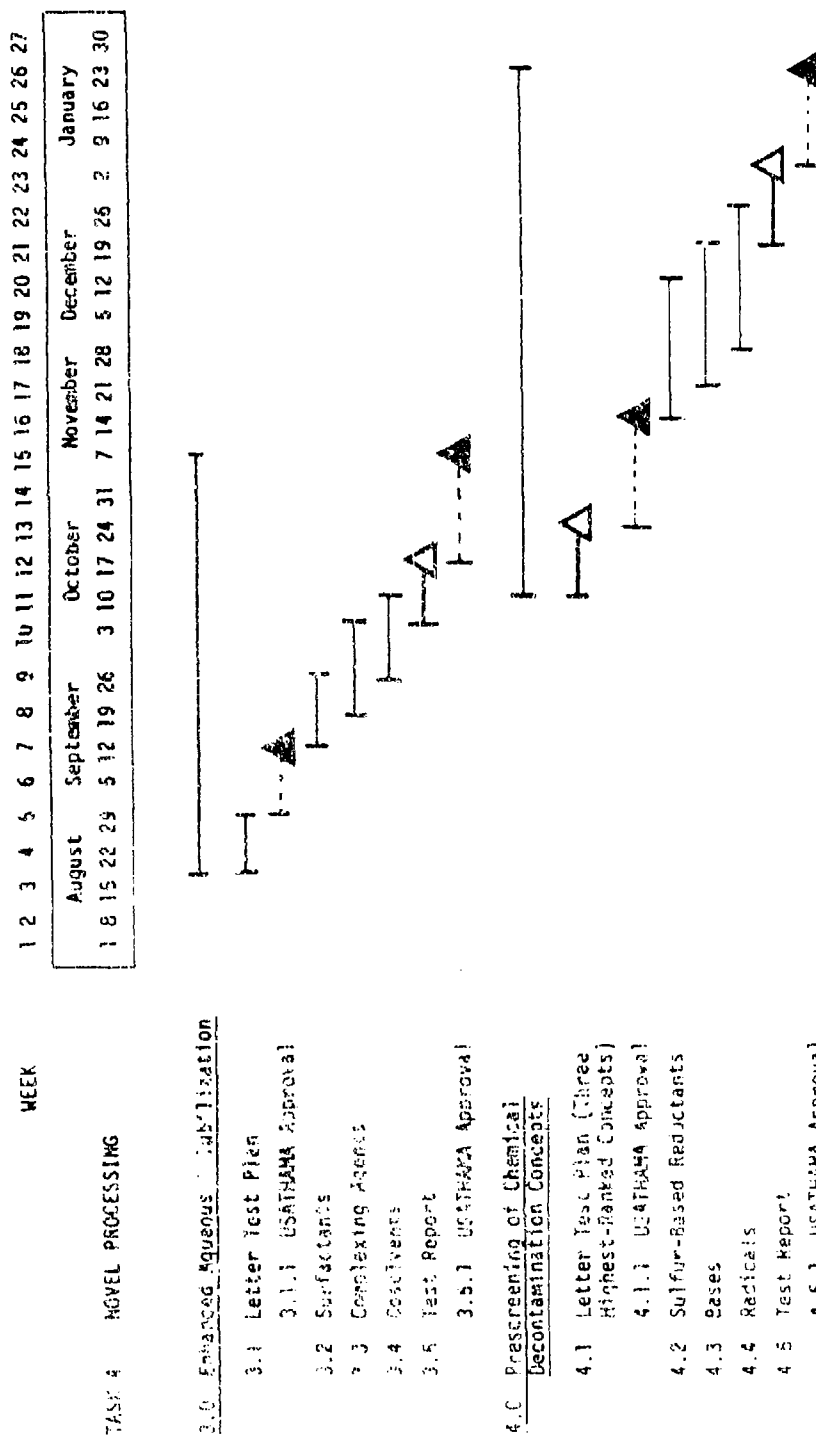
FIGURE 3. (Continued)

FIGURE 3. (Continued)

WEEK

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

April	May	June	July	August	September
2 9 16 23 30	7 14 21 28	4 11 18 25	2 9 16 23 30	6 13 20 27	3 10 17 24

TASK 4   NOVEL PROCESSING

7.0 Engineering/Economic
Analysis

7.1 Engineering Analysis

7.2 Economic Analysis

7.3 Knowledge Gap Identification

7.4 Report with Recommendations
for all Six Explosives

7.5 USATHAMA Approval

8.0 Task 4 Report/Field Test Plan

8.1 Draft Final Report

8.1.1 USATHAMA Approval

8.2 Final Task 4 Report

8.3 Field Test Plan

8.3.1 USATHAMA Approval
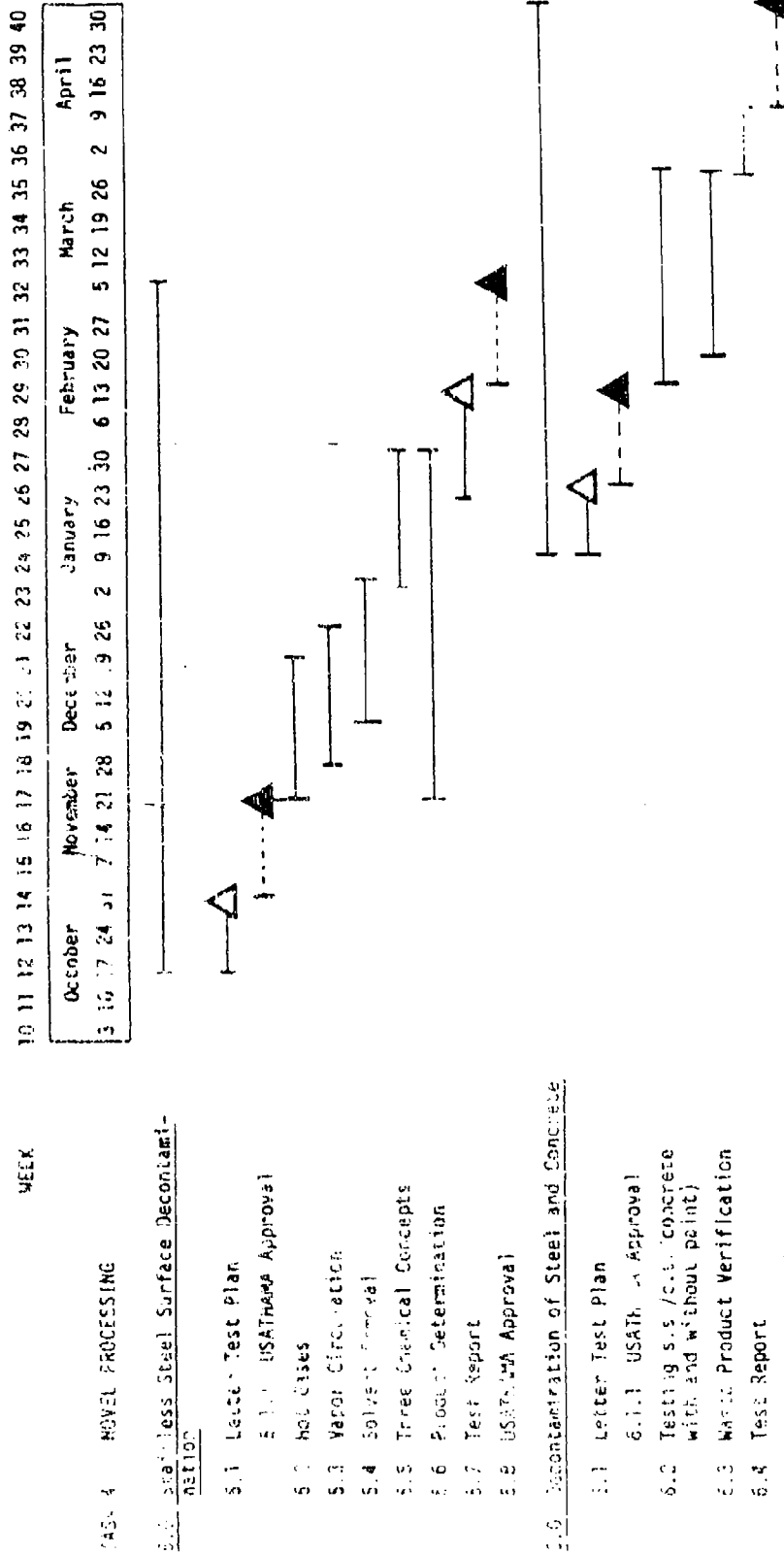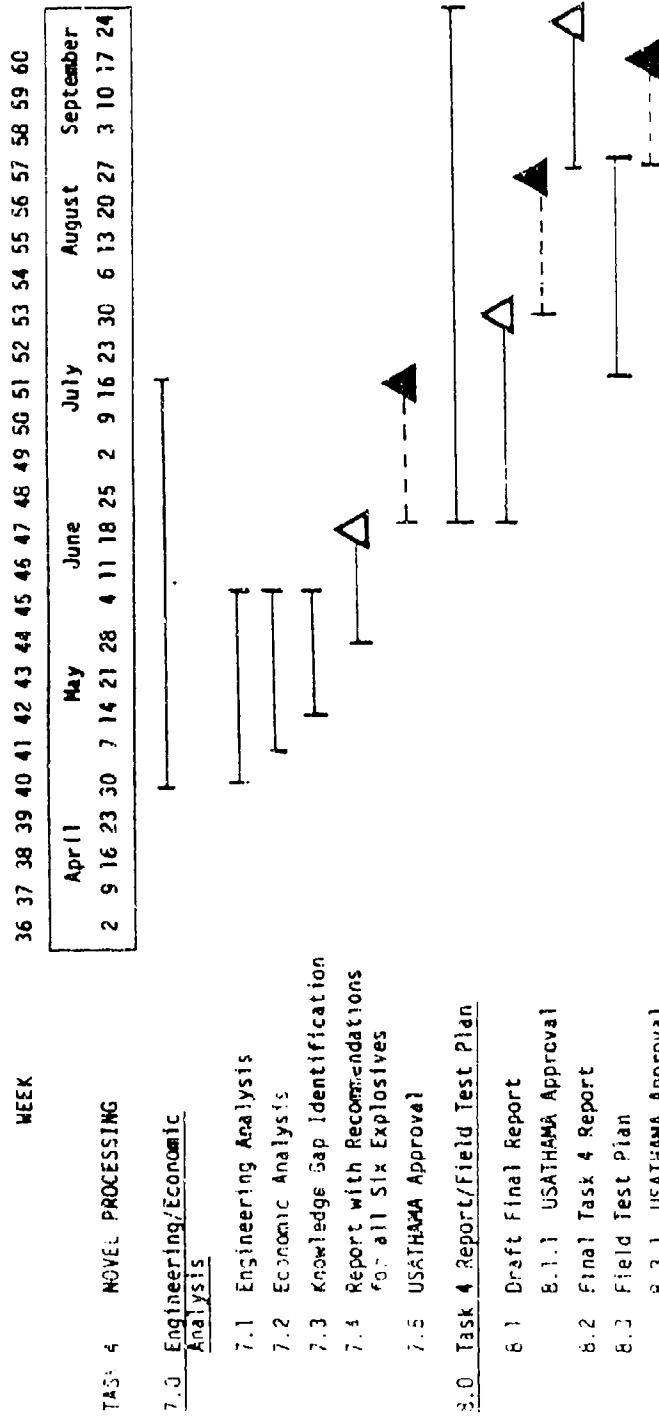
FIGURE 3.   (Continued)

APPENDIX II-A

GOVERNMENT TASKING DOCUMENT

Novel Processing Technology

A. Task Order Number: 4

B. Task Order Title: Development of Novel Decontamination and Inerting
Techniques for Explosive Contaminated Facilities - Phase II

C. Contract Number: DAAK11-81-C-0101

D. Background:

1. Decontamination or inerting of facilities previously utilized by the
Department of the Army for explosive manufacture or testing is necessary to
permit restoration of such facilities for alternate use or to dispose of them
safely. Included are the structures themselves plus storage tanks, processing
equipment, sumps, transfer systems and munition loading/packing equipment.
Items requiring decontamination include exposed surfaces as well as materials
into which explosives have penetrated, plus cracks, or other openings.

2. Materials of concern for which decontamination methods shall be
developed are:

    a. Stainless steel - painted and unpainted.

    b. Mild steel - painted and unpainted

    c. Concrete - painted and unpainted.

3. This task order is a follow-on to Task Order 2 of this contract. The
previous study identified and evaluated potential explosive decontamination
concepts from both technical and economic aspects. The concepts were then rank
ordered, and the contractor, in conjunction with USATHAMA, recommended those
deemed worthy of laboratory test and evaluation in Phase II (this task). It is
anticipated that this task will result in one to three explosive decontamination
concepts being sufficiently evaluated such that recommendations can be made for
pilot testing the selected concept(s).

E. Phase II Purpose and Objectives: The purpose of this task order is to
validate, by lab scale testing and technical/economic analyses, new cost
effective, technically sound decontamination concepts selected in Phase I for
use on explosive contaminated facilities. The objective of this task is to
develop a sufficient information/data base so that final selected concept(s) can
be recommended for subsequent pilot testing.

F. Statement of Work:

1. Figure 1 outlines the various work units for this task and the schedule
for their implementation. This statement-of-work is keyed to the work units as
shown on Figure I. The work to be performed on this task is based on the Task II
effort and generally follows the recommendations of the preliminary Phase II
test plan submitted under Item 5.7 of that task order.

2. The first work unit to be performed after task initiation shall be preparation of the overall work/resource plan. This plan shall include a detailed schedule, work breakdown structure (WBS) and a resource utilization plan keyed to the WBS.

3. Work unit 3 entails a review of regulatory and safety requirements and shall be performed to determine the most recent regulatory developments in these areas to better define decontamination levels. Pertinent organizations such as EPA, Department of Defense Explosives Safety Board, Department of Transportation and the DARCOM Field Safety Office shall be included. A report summarizing the findings of this review shall be prepared and submitted.

4. Work Unit 4 includes design and construction of special lab test equipment necessary to conduct the experimental work. Included is a chamber for conducting tests on explosive compounds. This chamber shall be sized to fit into a conventional fume hood, fitted with entry/exit ports for introduction and removal of samples and exhaust ports to permit sampling of off-gas products.

5. Work Units 5-9 are for the preliminary screening tests of chemical concepts selected on Task 1b. These concepts include: radical initiated decomposition, base initiated decomposition, sulfur base reduction, sodium borohydride reduction, and reductive cleavage. A letter test plan shall be submitted detailing test results when concluded. Chamber tests of each concept shall be conducted and waste products and analytical performance shall be evaluated. Three concepts, based on these tests, shall be recommended for further evaluation.

6. Work Units 10-14 delineate tests of methods for enhancing solubilization of explosives. Concepts to be tested include; surfactants, complexing agents, and cosolvents. These tests shall involve pure explosives and those which have been allowed to penetrate into concrete. Tests shall be conducted with the various concepts to evaluate solubility properties of the explosives and extraction efficiency from the concrete substrate. Feasible methods determined from these studies shall be used in conjunction with decontamination concepts to improve their effectiveness. Results of these efforts shall be provided in a letter test report.

7. Work Units 15-19 are for a series of tests utilizing stainless steel as the substrate to further screen candidate decontamination concepts. The three chemical concepts resulting from paragraph 5 testing (above) shall be tested along with hot gas and vapor circulation concepts. Analytical performance and waste product verification shall be incorporated into this test series. A letter test plan and a letter test report shall again be required. Included in the test report shall be recommendations for the three selected concepts to be carried forward to the final phase of lab testing.

8. Work Units 20-24 are for the final test series. The three best concepts from all of the previous testing shall be tested on concrete and steel, simulating as closely as possible, actual field decontamination scenarios.

Decontamination versus depth into the material shall be verified as well as identification of reaction by-products and off-gases. The test report for this test series shall recommend the final best 1-3 concepts.

9. Work Units 25-26 include technical and economic analysis of the final 1-3 concepts and a rank ordering of the concepts (if more than one is recommended). In addition to technical performance criteria, also to be considered are factors such as: building repair cost, development cost, utility and fuel costs, equipment costs, material and labor costs. These analyses shall result in selection of the final single best concept to be recommended for field/pilot testing. USATHAMA shall participate in and approve the final selection in writing.

10. Work Units 27-29 are for the preparation of a comprehensive final report. All pertinent information developed during efforts on this task order shall be compiled into a draft final report and submitted for USATHAMA review and approval, after which the final task report shall be submitted. This report shall be prepared in accordance with MIL Std 847-A.

11. Work Unit 30 requires the preparation and submission of a recommended test plan for a field evaluation of the final selected decontamination concept.

12. Analytical method development/refinement/certification shall be carried out as required in conjunction with this task.

13. Technical review shall be held as delineated in Work Unit 31. Four of these reviews shall be held at the Contractor's facility and four shall be held at USATHAMA. Additional meetings shall be scheduled on an as required basis.

G. Testing Requirements: Analytical methods certified in accordance with the USATHAMA QC Plan shall be utilized where applicable. However, the actual QA/QC level shall be specified by the CO,R.

H. Items/Data to be Delivered:

1. Letter test plans and letter test reports shall be submitted for each of the four test sequences outlined in Figure 1. (Work Units 3,5, 8,10,13, 15, 18, 20, 23). Ten copies of each shall be submitted.

2. A draft technical report (10 copies) documenting the findings and recommendations of this study shall be provided to the Government 52 weeks after task initiation. The final technical report (Sequence A002, CDRL, DD Form 1423) shall be provided to the Government within 30 days after approval of the draft.

I. Government Furnished Property and/or Assistance: The Government shall make arrangements for meetings with regulatory/safety government agencies for the purpose of conducting regulatory/safety review (Work Unit 3).

J. Hazards Information: Information regarding surety materials used on this task order are contained in the basic contract.

K. __Desired Period of Performance:__ Refer to figure 1. The overall period of
performance shall be 60 weeks.

L. __Security:__ Pertinent information regarding security is contained in the
basic contract.

M. __Estimated Level of Effort:__

Labor Category	Man-Hours
Program Manager	1,000
Organic Chemist	840
Analytical Chemist	840
Chemical Engineer	1,700
Analytical Technician	1,440
Lab Technician	1,400

APPENDIX III

SUBTASK REPORTS

APPENDIX III-1

SUBTASK 3 - ENHANCED AQUEOUS SOLUBILIZATION OF EXPLOSIVES - TEST REPORT

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TEST REPORT

for

TASK 4, SUBTASK 3

ENHANCED AQUEOUS SOLUBILIZATION
OF EXPLOSIVES

CONTRACT NO. DAAK11-81-C-0101

to

UNITED STATES ARMY
TOXIC AND HAZARDOUS MATERIALS AGENCY

February 15, 1985

by

H.P. Benecke
S.S. Harsh
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BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

MANAGEMENT SUMMARY

Various cosolvents, surfactants and complexing agents were evaluated for their ability to enhance the aqueous solubility of six explosives (TNT, 2,4-DNT, 2,6-DNT, tetryl, RDX and HMX). Surfactants were excluded from further consideration primarily because of the need to use unpractically high concentrations to achieve meaningful solubility enhancements. Complexing agents gave the highest observed solubility enhancements for TNT but were not considered further because of their lack of general applicability to all five explosives and other potential chemical reactivity problems. The aqueous cosolvents DMSO and DMF, when used at concentrations of thirty percent or higher, were judged most suited for use as decontamination or extraction solvent systems and are recommended for further evaluation in chemical prescreening experiments or explosive extraction studies.

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TASK 4, SUBTASK 3
TEST REPORT FOR
ENHANCED AQUEOUS SOLUBILIZATION
OF EXPLOSIVES
Contract DAAK11-81-C-0101
to
UNITED STATES ARMY
TOXIC AND HAZARDOUS MATERIALS AGENCY
February 15, 1985

1.0 INTRODUCTION

The development of novel concepts for the decontamination of chemical agent or explosive contaminated facilities is being carried out by Battelle Columbus Laboratories (Battelle) for the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) under Contract No. DAAK11-81-C-0101. In the previous phase (Task 1 (agents) and Task 2 (explosives)), ideas were systematically developed into concepts for decontaminating buildings and equipment. These concepts were evaluated and ranked with respect to technical and economic factors.

As described in the test plan for this subtask (Appendix A), solubilization of explosives is required for the effective chemical decontamination in solution or the extractive removal of explosives. Water is the best potential solvent for the solubilization of explosives in terms of its low cost and the low safety hazards associated with its use. Each of the three chemical concepts which are to be tested in Subtask 4 are compatible with and typically performed in aqueous based solvent systems. Furthermore, solvent extraction of explosives (to be studied in modified Subtask 6) by aqueous-based solvents would benefit from appreciable solubility in the solvents systems studied in this subtask. However, each of the six target explosives, which are to be investigated in these later studies, has very low solubility (about 0.01 percent or lower) in water.

2.0 OBJECTIVE

The objective of this subtask was to evaluate additives which were predicted in Phase I studies to increase the aqueous solubilities of the six target explosives (TNT, 2,4-DNT, 2,6-DNT, RDX, HMX and tetryl).

Those additives screened in this subtask for their potential enhancement of the aqueous solubilities of explosives included the following materials:

- **Cosolvents.** The cosolvents dimethylsulfoxide (DMSO), dimethylformanide (DMF) and acetone are soluble in all proportions in water and some of the target explosives had high solubilities in these solvents (see Appendix B).
- **Surfactants.** A variety of nonionic, cationic, anionic as well as some specialty surfactants.
- **Complexing agents.** The potential complexing agents such as diethanolamine (DEA) and 4-hydroxyethylpiperazine (HEP) possess both nitro group coordinating sites and hydroxyl functions which would increase the aqueous solibility of coordinated explosives.

3.0 TEST EQUIPMENT AND MATERIALS

A Lauda Model B-1 circulating constant temperature bath was connected through external connections to three jacketed 250 ml beakers. These beakers were placed on top of magnetic stirrer motors and were filled with approximately one inch of water. The temperatures of this contained water and the circulating bath water was measured with mercury and alcohol thermometers.

Solubility measurements were performed with High Performance Liquid Chromatography (HPLC). The following HPLC equipment and operational parameters were employed:

Equipment

- Altex 110 A pump
- LDC Spectromonitor III-UV detector
- Micrometics 725 Autoinjector
- Hewlett Packard 1000 Computer with Computer Inquiry System (CALS) Chromatographic Software.

Parameters

- Column: Spherisorb ODS 5μ, 25cm x 4.6 mm I.D.
- Mobile Phase: methanol/water 50/50, isocratic
- Flow Rate: 1.0 ml/min
- Detector: UV @ 254 nm
- Attenuation: unattenuated output to computer
- Injection volume: 10 μl

The 2,4-DNT was used as received from Aldrich Chemical Company after grinding to uniform particle size by rolling the material between a wooden dowel and a rubber pad covered by a plastic sheet. The 2,6-DNT was used as received from Aldrich Chemical Company without grinding.

The TNT and tetryl used in these studies were munition grade explosives and were obtained through internal transfer from other projects at Battelle. No attempts were made to modify the particle size or recrystallize these materials.

A small portion of recrystallized RDX was also transferred from another project within Battelle. This material was shown to have the identical HPLC retention time as the RDX which was shipped to Battelle from Holston AAP. (Batch No. 4RC 14-71). This material and HMX (Batch No. 6ABCH 14-1) were shipped wetted with isopropyl alcohol. Prior to use, small quantities of both RDX and HMX were dried under high vacuum for one day to remove this alcohol.

The classification and chemical composition of surfactants are described below: Triton X-100 is a non-ionic surfactant and is composed of octylphenyl polyoxyethylenealcohol. Tween 21 is a non-ionic surfactant and is composed of polyoxyethylene sorbitan fatty acid esters. Alkamox LO is an ylid surfactant of the amine oxide type. Stephanate CS is an anionic surfactant and is composed of cumene sulfate. Cetyl pyridinium bromide is a cationic surfactant.

4.0 GENERAL TEST CONDITIONS AND ANALYTICAL APPROACHES

The solubilities of explosives in general were measured by stirring a slurry of 200 mg of the explosive in 2-4 ml of the solubilization system being evaluated in one or two dram vials equipped with 10 x 3 mm polyethylene covered stir bars and teflon lined screw caps. Temperatures were maintained either at room temperature (23-25 C) or in thermostatted baths (either 23-24 C or 24-25 C). Efforts were made to maintain approximately the same stirrer speed but the stirring speeds were not calibrated.

The first analytical method which was used to determine the amount of solubilized explosives was the "filter, dry and weigh" method. This method involved accurately weighing the starting quantity of explosive (200-300 mg), stirring the explosive in 2.00 ml of the solubilization solution being tested for a set period of time, and filtering, drying and weighing the undissolved explosive. In the evaluation of cosolvents and surfactants, the undissolved explosives were collected in fritted glass funnels and dried under high vacuum to a constant weight. Water blanks were employed to correct for any losses through mechanical means (The amounts of explosives solubilized by water was assumed to be negligible.) This method was employed in the initial studies in which 2,4-DNT and TNT were studied. This method was discontinued because it was found to give non-reproducible solubility values that ranged up to one order of magnitude higher than the solubilities measured by the HPLC method described below. These erratic results were presumed to be due to solvent specific "milling" effects which may have reduced the particle size below the fritted-glass filter pore size after stirring for prolonged periods of time.

The analytical method of choice to determine the solubility of explosives in the presence of aqueous cosolvents and surfactants was high performance liquid chromatography (HPLC). This HPLC method directly measures the concentrations of explosives in solution and therefore, does not require the initial accurate weighing of explosives. Approximately 200 mg of explosives were dispersed into vials for each determination. HPLC was presumed not to be applicable for determining the solubility of explosives in the presence of complexing agents because new species other than dissolved explosives could potentially be generated.

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The initial HPLC determinations of 2,4-DNT in various cosolvents employed centrifugation of the solubilization samples before aliquots were withdrawn for HPLC analysis. However, this sequence led to relatively low levels of precision. All subsequent HPLC determinations employed the centrifugation of the test sample but the supernatant was withdrawn and passed either through a 5 mm. I.D. pipet which had been packed with approximately 15 mm of surgical cotton or through Millipore filters. In the evaluation of surfactants, Millipore filters were used almost exclusively to generate clear solutions prior to HPLC analyses.

Aliquots of the filtrate (20 μl) were then removed by Eppendorf pipets and diluted with methanol (1 to 100) to provide samples for HPLC testing. The excess filtrate was then returned to the test mixture. This procedure was repeated periodically to give solubility, time profiles as long as undissolved explosive was present. Linear regression analysis from plots of standard concentrations of explosives (which bracketed the test concentrations) versus detector area response were used to determine explosive concentrations in solubility samples.

Solubilization by complexing agents was determined by stirring weighed amounts of explosive (approximately 200 mg) in 4.00 ml of solubilization solution followed by filtration through Whatman No. 2 filter paper. The emptied vials were flushed with filtrate but were not flushed with fresh water because, in the case of TNT, the solid materials remaining after treatment with these complexing agents were found to be soluble in the fresh water unlike neat TNT. (See particulars in Sections 5.2 and 5.3). The filtered solid materials were dried under high vacuum until constant weights were achieved.

After the HPLC monitored solubility studies were in progress, it was discovered that the type of Millipore filters which were initially used (Millex GS) were removing part of the dissolved explosives prior to the HPLC analyses. These filters were 22 micron filters and made from cellulose esters and PVC. This problem was studied and rectified by subsequently using Millex SR 50 micron filters which were shown not to remove dissolved explosives in control studies. Millex SR filters are made from polytetrafluoroethylene and polyethylene. In the tables which present the results of these solubilization tests, the types of filters which were employed are specified.

5.0 PRELIMINARY SOLUBILITY DETERMINATIONS ON 2,4-DNT, TNT AND RDX

The initial solubilization tests were performed with the explosives 2,4-DNT, TNT and RDX since these explosives were expected to respond similarily to solubilizing agents as would 2,6-DNT, tetryl and HMX respectively. The solubilization additives which were judged to be the most effective or appropriate for further chemical concept screening in Subtask 4 were then validated with the explosives 2,6-DNT, tetryl and HMX. Therefore a significantly greater number of solubilization experiments were performed with 2,4-DNT, TNT and RDX than performed with 2,6-DNT, tetryl and HMX.

The initial explosive studied was 2,4-DNT since it was relatively non-explosive and judged to be insensitive to spark discharge. TNT was studied next and RDX was studied last because it was judged by explosives experts to be spark sensitive and there was only a limited supply at Battelle until the Army shipment was received towards the end of this study.

5.1 Solubilization of 2,4-DNT

5.1.1 Aqueous Cosolvents

The results of the solubilization of 2,4-DNT by aqueous cosolvents are shown in Table 1. Most of the data shown was obtained by only centrifuging the analytical samples (i.e., without subsequent filtration) before HPLC analyses were performed. Data points were obtained within relatively short time intervals because initial solubilization results indicated that the magnitude of solubilizations were highly time dependent and were highest at early sampling times and tended to decrease with increasing time intervals. This effect, which was generally observed in each cosolvent studied at various concentrations, may result from the initial supersaturation of the solution which is followed by a decrease in concentration to equilibrium values. When duplicate or triplicate samples were determined, the data were averaged and listed in terms of the mean values with the standard deviations. Relatively large variations were observed in sixty percent acetone so that caution must be used in interpreting the values in Table 1. Some solubilization values are also included which were obtained by filtering the analytical samples through

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TABLE 1. SOLUBILIZATION OF 2,4-DNT BY AQUEOUS COSOLVENTS
(GRAMS/100 ML SOLUTION)a,b HPLC ANALYTICAL METHOD

Cosolvent	Percent	1 Hr	2 Hr	4 Hr	12 Hr	17 Hr	27 Hr	52 Hr	76 Hr
						Time			
Acetone	10		0.031	0.036	0.034		0.082		
	20		0.11	0.15	0.071		0.067		
	30		0.23	0.15	0.16		0.16		
	45		0.70	0.72	0.66	0.13	0.61		
	60	5.3±.42	4.9±.61	2.5±.87	1.6	.46±.08 1.6±.40	1.6		
DMF	10			0.10	0.036		0.033		
	20		0.067	0.079	0.070		0.078		
	30		.27±.14, .14f	0.13	0.13	.12±.01	.18±.04, .13f	.11±.02, .15e,f	.21±.00
	45		.54±.18, .30f,g	0.33	0.80	.30±.02	.37±.10, .24f	.19±.10, .25e,f	0.30
	60		1.1	1.2	1.2	1.0±.07	1.3, 1.4f		
DMSO	10		0.025	0.044	0.027		0.026		
	20		0.087	0.088	0.042		0.053		
	30		.075, .069f	0.100	0.070	0.073	.20, .062f	0.072e,f	
	45		.47±.11, .11f,g	0.34	0.12	.11±.001	.20±.03, .11f,g	.25±.06, .11e,f	.24±.06
	60		.71±.18	0.55	0.42		.35±.04, .45f	.40±.04	.55±.23
Water	100						0.007c,d	0.013c,e	
	100							0.021e,f	

(a) Temperatures were either maintained at 23 to 25 C in ambient air or at 24 to 25 C in thermostatted and jacketed beakers.

(b) Analytical solutions were removed by pipette after centrifugation.

(c) These analytical samples were also filtered through Millex GS 22 micron filters.

(d) Determined after 24 hours.

(e) Determined after 48 hours.

(f) Filtered through cotton after centrifugation.

(g) 40 percent cosolvent was employed.

cotton after initial centrifugation. It can be seen that these solubilization values are generally lower than the values obtained without employing filtration.

For 2,4-DNT, the order of effectiveness of the three cosolvents at comparable concentrations is: acetone > DMF > DMSO. The solubilities of 2,4-DNT in each of these ten-percent cosolvents concentrations are similar and are only 2 to 3 times higher than the measured solubility of 2,4-DNT in pure water (0.021 percent). The order of the rate of increased solubility of 2,4-DNT as the cosolvent concentrations are increased is: acetone > DMF > DMSO. It appears that the tendency for supersaturation at 60 percent cosolvent concentrations may be greatest for acetone compared to the other cosolvents at this concentration.

5.1.2 Surfactants

The results of the solubilization of 2,4-DNT with a range of different type surfactants is shown in Table 2. The surfactant concentrations employed were five percent except in the case of cetyl pyridinium bromide where 0.1 percent was used due to its low solubility. Millex GS filters were used initially in this work but Millex SR filters were subsequently used in the two cases where solubilization appeared to be occurring. By comparison of solubility values derived using these two types of filters, it appears that some 2,4-DNT was removed from the analytical samples by the Millex GS filters. Nevertheless, it can be seen that the only surfactants which appreciably solubilized 2,4-DNT were Triton X-100 and Alkamox LO. These enhanced solubilities were only approximately one order of magnitude higher than the aqueous solubility of 2,4-DNT. It should be noted that the analytical approach used measured the quantity of truly dissolved explosive versus that quantity of explosive which may have been solubilized as colloidal particles which would be large enough to be filtered by the Millex GS filter.

5.1.3 Complexing Agents

The potential complexing agents diethanolamine (DEA) and 4-hydroxy-ethylpiperazine (HEP) were evaluated by the filter, dry and weigh method as

TABLE 2. SOLUBILIZATION OF 2,4-DNT BY AQUEOUS
SURFACTANTS (GRAMS/100 ML. SOLUTION)[a,b]
HPLC ANALYTICAL METHOD

Surfactant	Percent	Time	
		24 Hr	48 Hr
Triton X-100	5	0.011, 0.16[c]	0.091, 0.087, 0.16[c], 0.18[c]
Tween 21	5	0.012	0.016, 0.014
Alkamox LO	5	0.071	0.092, 0.088, 0.10[c]
Stephanate CS	5	0.010	0.018, 0.018
Cetyl Pyridinium Bromide	0.1		0.009, 0.009
Water	100	0.007	0.013

(a) Temperatures were maintained at 23 to 24 C in thermostatted and jacketed beakers.

(b) Analytical solutions were removed after initial centrifugation of samples followed by filtration of supernatant through Millex GS 22 micron filters.

(c) After centrifugation, the supernatant was filtered through Millex SR (solvent resistant) 50 micron filters.

previously described. The degree of solubilizations (Table 3) varied between 1.2-1.4 percent when 10-30 percent complexing agents were employed. These solubilizations are significantly greater than the solubilities (.03-.2 percent) achieved with the same concentrations of aqueous cosolvents. However, these complexing agents immediately formed intensely colored brownish black solutions which in practice might present secondary decontamination problems.

5.2   Solubilization of TNT

5.2.1   Aqueous Cosolvents

After examination of data concerning the solubilization of 2,4-DNT with aqueous cosolvents, the 10%-acetone, -DMF and -DMSO solutions were eliminated from further testing. Results of the solubilization of TNT by aqueous cosolvents are shown in Table 4. The data indicates that with the 20 and 30 percent acetone and DMF mixtures, higher than equilibrium solubilization initially occurred which was followed by some decreases with time. TNT solubilities in DMSO appear to reach equilibrium values and change little with time. By comparison, it can also be seen that TNT has significantly lower solubilities than does 2,4-DNT in the same cosolvent compositions. It appears that the order of solubility for TNT (after 27 hours and between 20 to 30 percent cosolvent) is that acetone is slightly better than DMF which is only slightly better than DMSO. DMF and DMSO were evaluated in the 40 and 60 percent range also. (Acetone was not evaluated because it had been eliminated from further consideration as a consequence of flash point determinations which were performed after the 10-30 percent tests were performed. See Section 6.) These extended tests showed that almost 20 times more TNT dissolved in DMF when the concentration was increased from 30 to 60 percent whereas approximatley 10 times more TNT dissolved when DMSO concentration was increased by the same amounts.

It is noteworthy that the same solubilizations were achieved, in the cases of 60 percent DMF and DMSO after 27 hours, when the solutions were

TABLE 3. SOLUBILIZATION OF 2,4-DNT BY AQUEOUS COMPLEXING
AGENTS[a,b] FILTER, DRY, AND WEIGH METHOD

Complexing Agents	Percent	Time	Grams/100 ml Solvent
Diethanolamine	10	48 hr	1.3
(DEA)	30	48 hr	1.3
4-Hydroxyethyl-	10	48 hr	1.2
Piperazine	30	48 hr	1.4
(HEP)			

[a] Temperatures were maintained at 25 C in thermostatted beakers.

[b] Sediments were removed by filtration through Whatman No. 2
filter paper. The vials were rinsed out with filtrate but
were not rinsed with water.

TABLE 4. SOLUBILIZATION OF TNT BY AQUEOUS COSOLVENTS
(GRAMS/100 ML SOLUTION)a,b HPLC ANALYTICAL METHOD

Cosolvents	Percent	Time				
		2 Hr	7 Hr	24 Hr	27 Hr	48 Hr
Acetone	20	0.029, 0.033	0.033, 0.029		0.013, 0.020	
	30	0.062, 0.082	0.062, 0.056		0.029, 0.042	0.062
DMF	20	0.036, 0.033	0.036, 0.036		0.026, 0.016	
	30	0.062, 0.065	0.059, 0.056		0.036, 0.029	
	40				0.10	0.067[c], 0.067[c], 0.11
	60				0.61, 0.66[d]	0.64
DMSO	20	0.026, 0.026	0.026, 0.026		0.023, 0.023	
	30	0.039, 0.039	0.039, 0.042		0.033, 0.033	
	40			0.024, 0.024[c]	0.057	0.035[c], 0.034[c], 0.059
	60				0.28, 0.30[d]	0.29
Water	100			0.006[c]		0.012[c], 0.021

(a) Temperatures were maintained at 24 to 25 C in thermostatted and jacketed beakers.

(b) Analytical solutions were removed after initial centrifugation of samples followed by filtration of supernatant through cotton filters.

(c) Filtered through 22 micron Millex GS filters.

(d) No stirring was performed.

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either vigorously stirred or not stirred at all. These results imply that contact of these solvent systems with TNT will approach saturation without stirring or other mechanical actions given sufficient time. Therefore, TNT which may have diffused into concrete walls would be expected to approach saturation in contact with these solvent systems without the benefit of any agitation.

Figure 1 shows the chromatogram of munition grade TNT completely dissolved in methanol which has a retention time of 8.95 minutes. Figure 2 shows the chromatogram of TNT which has been stirred with 30 percent DMSO for 27 hours. It can be seen that a significant peak appears at 13.45 minutes which was close to the retention time of 2,4-DNT under these conditions. Some speculation was initially advanced that 2,4-DNT may be resulting from denitration of TNT in these aqueous cosolvents. However, a more plausible explanation is that the more soluble 2,4-DNT is being selectively leached from the TNT which contains small amounts of 2,4-DNT. That this is the case was shown in later chemical decontamination tests which involved totally dissolving measured quantities of the same grade TNT in neat DMSO and then diluting this solution to 30 percent DMSO by the addition of water. These solutions did not generate any significantly larger HPLC peak in the 2,4-DNT retention time region when compared to the results from the analysis of TNT itself.

5.2.2 Surfactants

The results of the solubilization of TNT with the same surfactants which were used to study 2,4-DNT are shown in Table 5. As in the study of 2,4-DNT, Millex SR filters were used to retest the more promising surfactants which were initially filtered through Millex GS filters. By comparison of the two sets of data, it appears that a significant amount of TNT had been depleted from the analytical solutions by employing the Millex GS filters. The highest solubilities were observed with Triton X-100, Tween 21 and Alkamox LO and ranged between .04 and .08 percent. These solubilities were lower than those observed in the solubilization of 2,4-DNT by surfactants and are only several fold higher than the aqueous solubility of TNT (.012 percent) determined in this study. (The previously reported aqueous solubility of TNT is 0.013 percent).

File: GC290L
FE = 0

TNT 10 STD
Inst: 8 Ch:0 Tray # 0

10:17:42 11/10/1983
Pol +   Page 1

TNT
8.95

RT 8.95
A 671.359
H 25.022

Area Analysis

File: GC290   TNT 10 STD   10:17:42 11/10/1983
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1340   TNT 10 STD   14:36:36 11/14/1983

Name	Wt %	Pk	RRT	RRF	Area	Height
TNT	100.000	BCB	8.95	1.0000	671.359	25.022
	100.000					

2.0   4.0   6.0   8.0   10.0   12.0   14.0   16.0   18.0   20.0

M I N U T E S

Max: 54.917 mv
Min: 29.063 mv

% Smx: 100.000
% Smn: 0.000

FIGURE 1. HPLC OF MUNITION GRADE TNT (10 μg/ml)
Dissolved Completely in Methanol

File:GC482L
FF = 0

52-23B   30% DMSO 27 hours
Inst: 8   Ch:0   Tray # 0

00:40:54   11/22/1983
Pol +   Page 1

TNT #
8.4+

Area Analysis

File.GC482   52-23B
Mthd: 1310   Inst 8

Chnl 0   Vial #   0

00:40:54   11/22/1983
Stnd/Smpl %   100.000

Mthd: 1340   52-23B

07:33:42   11/22/1983

Name	WT %	Pk	RRT	RRF	Area	Height
001	4.974	BCB	6.30	1.0000	20.817	1.189
TNT	56.838	BCV	8.97	1.0000	237.903	10.058
002	9.289	VCB	10.06	1.0000	38.880	1.366
003	28.900	BCB	13.45	1.0000	120.963	4.576
	100.000					

M I N U T E S

Max: 23.301 m
Min: 19.925 m

1 Sm.   100.000
1 Scn   0.000
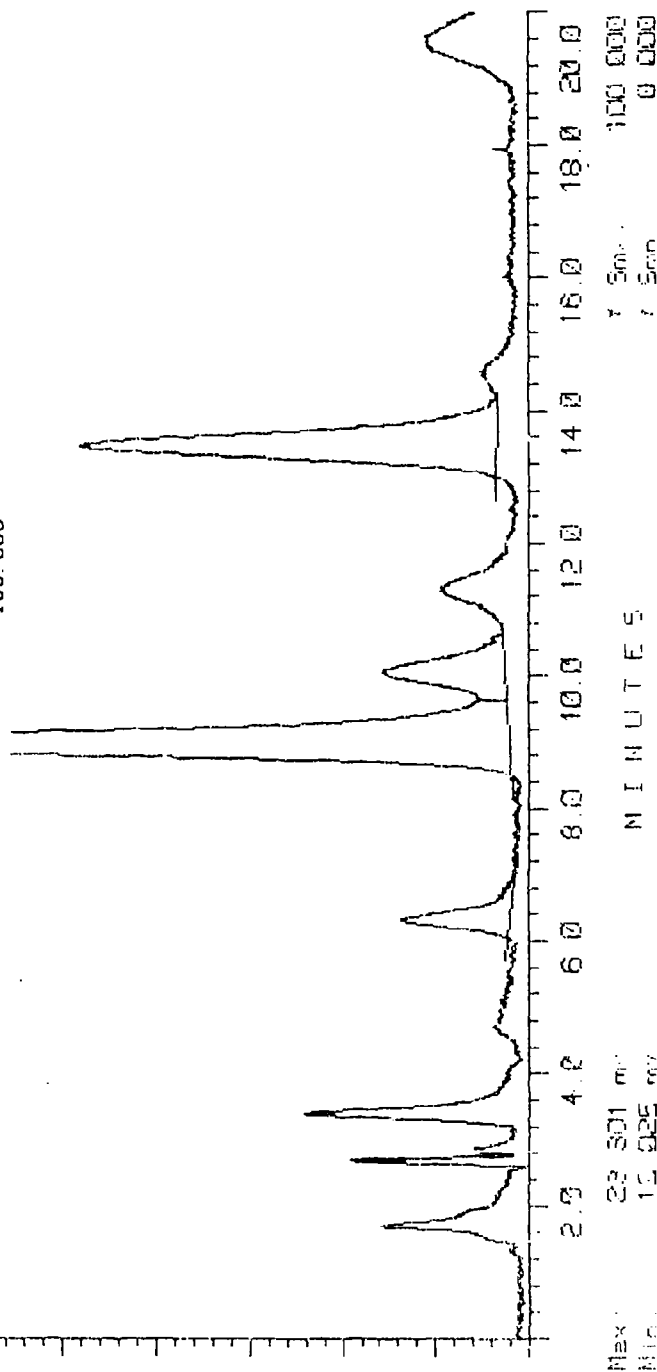
FIGURE 2.   HPLC OF TNT IN THIRTY PERCENT DMSO FOR 27 HOURS

TABLE 5. SOLUBILIZATION OF TNT BY AQUEOUS SURFACTANTS
(GRAMS/100 ML SOLUTION)[a,b] HPLC ANALYTICAL METHOD

Surfactant	Percent	Time 24 Hr	Time 48 Hr
Triton X-100	5	0.067[e]	0.014, 0.011, 0.065[e], 0.019[e]
Tween 21*	5	0.050	0.057, 0.055[c], 0.077[e]
Alkamox LO*	5		0.026, 0.025[d], 0.041[e]
Stephanate CS	5	0.007	0.012, 0.012
Cetyl Pyridinium Bromide	0.1	0.011	0.011, 0.011
Water	5	0.006	0.012

[a] Temperatures were maintained at 23 to 24 C in thermo-statted and jacketed beakers.

[b] Analytical solutions were removed after initial centri-fugation of samples followed by filtration of super-natants through Millex GS 22 micron filters.

[c] 0.019, 0.022 (DNT)

[d] 0.024, 0.025 (DNT)

[e] Analytical solutions were filtered after initial centri-fugation through Millex SR 50 micron filters.

* TNT solubilization in Tween 21 and Alkamox LO showed second compound, presumed to be 2,4-DNT, which eluted at 12.5 minutes.

5.2.3 Complexing Agents

The solubilizing effects of the complexing agents DEA and HEP towards TNT are shown in Table 6. The data shows that the solubility of TNT is markedly enhanced by both complexing agents in comparison to the aqueous cosolvents evaluated at the same concentrations. HEP was found to be more effective than DEA at the same concentrations and was found to solubilize TNT to an extent greater than 11.7 percent when 30 percent HEP was employed. This solubilization of TNT by HEP represents a minimum of an approximately 350 fold solubility enhancement in comparison to the solubilizations observed   the aqueous cosolvents of the same concentration. As was the case with 2,4-DNT, the complexing solutions were colored dark reddish-black and the recovered sediments were also darkly colored. These sediments were found to be water soluble.

These very large enhanced solubilizations of TNT by HEP and DEA, compared to that of the cosolvents tested, is compatible with the formation of sigma bonded complexes between the amine nitrogen atoms and the aromatic nitro groups. This interpretation is reinforced by the fact that TNT was solubilized to a significantly greater extent by HEP and DEA than was 2,4-DNT. It would be expected that the nitro groups in TNT would be more electron deficient than the nitro groups in 2,4-DNT and thus be attacked more readily by the nucleophilic amine nitrogen atoms. That a chemically altered TNT species is formed is indicated by the HPLC chromatogram (Figure 3) of the solution which resulted when TNT was solubilized by 30 percent HEP. The fact that no HPLC peak is evident at the characteristic retention time of TNT (approximately nine minutes) indicates that the large percentage of TNT which was solubilized (greater than 11.7 percent) must have been converted to some other species. This explanation is reinforced by the fact that when the TNT was not totally dissolved, brown red sediment remained which could be solubilized by adding water. Thus the material could not be unmodified TNT and logically represents some modified form of TNT.

5.3 Solubilization of RDX

5.3.1 Aqueous Cosolvents

The solubilities of RDX were determined in 20 and 30 percent acetone, DMF and DMSO as well as higher concentrations of DMF and DMSO as

TABLE 6.   SOLUBILIZATION OF TNT BY AQUEOUS COMPLEXING
AGENTS[a,b] FILTER, DRY, AND WEIGH METHOD

Complexing Agents	Percent	Time	Grams/100 ml Solvent
Diethanol-Amine (DEA)	10	48 hr	1.1, 1.6
	30	48 hr	3.6, 4.3
4-Hydroxyethyl-Piperazine (HEP)	10	48 hr	1.9, 2.2
	30	53 hr	>11.7[c]

(a) Temperatures were maintained at 23 to 24 C in thermostatted and jacketed beakers.

(b) Sediments (brown red) were removed by filtration through Whatman No. 2 filter paper without rinsing (sediments were soluble in water).

(c) No sediment was evident after centrifugation and filtration.

FIGURE 3. HPLC OF TNT IN THIRTY PERCENT HYDROXYETHYLPIPERAZINE (HEP) FOR 48 HOURS

shown in Table 7. Higher percentages of acetone were not tested because it had been eliminated from further consideration as a consequence of flash point determination which indicated that aqueous acetone concentrations containing as low as 20 percent acetone would be prohibitively flammable. RDX solubilities in all cosolvents tested appear to reach equilibrium values early and change little with time.

By comparison to tests on TNT it can be seen that RDX has slightly lower solubilities than does TNT in the same solvent compositions. Comparisons between variable concentrations of DMF indicated that approximately 20 times more RDX was dissolved when the concentration was increased from 30 to 60 percent, whereas approximately 10 times more RDX was dissolved when DMSO was increased by the same amount. These increases were similar in magnitude to those observed with TNT in the same two cosolvent systems.

5.3.2 Surfactants

Since surfactants were shown to be marginally effective in previous solubilization studies with 2,4-DNT and TNT, Triton X-100 was the only surfactant examined for RDX. The data are shown in Table 8 and indicate RDX solubilities (at five percent Triton X-100 concentration) are equal to or higher than was realized in each of the aqueous cosolvents studies at the thirty percent level after 24 hours.

5.3.3 Complexing Agents

The solubilization effects of the complexing agents DEA and HEP towards RDX are shown in Table 9. Ambient air was used to establish the solubilization temperatures. It can be seen when the temperature ranged from 24-25 C, that the measured RDX solubilizations by DEA and HEP complexing agents are slightly above or below the blank value effected by water alone respectively. The significant water blank value (0.30 percent) indicates that mechanical losses or other sources of error of RDX are inherent in the experimental method employed since the aqueous solubility quoted in the literature is 0.005 percent. The results in Table 9 suggest that the complexing agents have marginal solubilizing effects on RDX at the concentration levels which were studied.

TABLE 7. SOLUBILIZATION OF RDX BY AQUEOUS COSOLVENTS
(GRAMS/100 ML SOLUTION)[a],[b] HPLC ANALYTICAL METHOD

Cosolvents	Percent	Time				
		2 Hr	7 Hr	17 Hr	27 Hr	48 Hr
Acetone	20	0.015, 0.017	0.013, 0.013	0.013, 0.012	0.013, 0.013	
	30	0.033, 0.032	0.027, 0.031	0.031, 0.032	0.028, 0.027	
DMF	20	0.015, 0.015	0.015, 0.017	0.018, 0.015	0.013, 0.013	0.029[c]
	30	0.028, 0.029	0.028, 0.027	0.027, 0.027	0.027, 0.027	0.061[c]
	40				0.56[c]	
	60					0.49[c]
DMSO	20	0.012, 0.012	0.010, 0.010	0.011, 0.011	0.011, 0.011	0.021[c]
	30	0.019, 0.019	0.018, 0.018	0.019, 0.019	0.018, 0.018	0.034[c]
	40				0.31[c]	
	60					0.26[c]
Water	100	0.00	0.00	0.00	0.00	

(a) Temperatures were maintained at 23 to 24 C in thermostatted and jacketed beakers.

(b) Analytical solutions were removed after initial centrifugation of samples followed by filtration of supernatant through cotton filters.

(c) These solubility determinations were performed at a later time compared to the other solubilization data presented.

TABLE 8. SOLUBILIZATION OF RDX BY AQUEOUS TRITON X-100
(GRAMS/100 ML SOLUTION)[a],[b] HPLC ANALYTICAL METHOD

Percent	Time	
	27 Hr	48 Hr
1	0.016, 0.008	0.022, 0.024
5	0.024, 0.021, 0.026[c],[d]	0.052, 0.064, 0.027[d]

(a) Temperatures were maintained at 24 to 25 C
in thermostatted and jacketed beakers.

(b) Analytical solutions were removed after
initial centrifugation of samples followed
by filtration of supernatants through
cotton filters.

(c) Solubilization time was 24 hours.

(d) Filtered through Millex SR 50 micron
filters.

TABLE 9.   SOLUBILIZATION OF RDX BY AQUEOUS COMPLEXING
AGENTS[a,b] FILTER, DRY AND WEIGH METHOD

Complexing Agents	Percent	Time	Grams/100 ml Solvent
Diethanolamine (DEA)	10	48 hr	0.19[c], 0.58[d]
	30	48 hr	0.20[c], 0.59[d]
4-Hydroxyethyl-Piperazine (HEP)	10	48 hr	0.36[c], 0.44[d]
	30	48 hr	0.42[c], 0.40[d]
Water	100	48 hr	0.30[c]

(a) Ambient air temperatures were recorded and employed to establish complexation temperatures.

(b) Sediments were removed by filtration through Whatman No. 2 filter paper. The vials were rinsed with filtrate but were not rinsed with water.

(c) Ambient air temperatures of 24 to 25 C were recorded.

(d) Ambient air temperature of 27 C was recorded.

6.0 COSOLVENT FLASH POINT DETERMINATIONS

The flash points of 20 and 30 percent acetone, DMF and DMSO were determined with a TAG closed cup tester (ASTM D56). The test solutions were placed in a closed cup which was heated slowly by immersion in an attached water bath, while periodically exposing the collected vapors in the cup to an open flame. When a "pop" was heard it indicated the vapors had flashed and the corresponding water bath temperatures were recorded. The data (Table 10) indicate that 20-100 percent aqueous acetone compositions flash at unacceptably low temperatures which indicated that acetone should be dropped from consideration in the context of building decontamination. These flash point tests were performed after the solubilization effects of all aqueous cosolvents in the 10-30 percent range had been determined on 2,4-DNT (higher percentages were evaluated with 2,4-DNT), TNT and RDX. No further solubilization tests with acetone were performed after these flash point tests were completed.

The flash temperature of 100 percent DMSO (198 F) was found to be significantly greater than the flash temperature of 100 percent DMF (138, 139 F). The flash temperatures of 20 and 30 percent DMSO and DMF were found to be greater than 212-215 F (which is the temperature limit of this apparatus). The flash temperatures of 30 to 100 percent DMSO and DMF aqueous compositions are therefore expected to be greater than 198 F and 138 F respectively.

7.0 SELECTION OF SOLUBILIZATION METHODS
FOR FURTHER VERIFICATION

The results of the solubilization tests on 2,4-DNT, TNT and RDX were used to eliminate certain aqueous solvent systems from further consideration. After these systems were eliminated, verification tests were performed on with the most promising aqueous solvent systems on 2,6-DNT, tetryl and HMX. In certain cases, the decision to either omit or favor aqueous solvent systems for further testing were based partly on considering the impact that these systems would have on the predicted behavior of the chemical concepts to be evaluated in the Task 4 screening efforts.

TABLE 10. FLASH POINT DETERMINATION
OF AQUEOUS COSOLVENTS[a]

Cosolvent	Percent	Flash Temperatures ($^\circ$F)
Acetone	100	-3, -4
	30	11, 12
	20	37
DMF	100	138, 139
	30	>215
	20	>212
DMSO	100	198
	30	>215
	20	>215

(a) Determined in a TAG closed-cup tester (ASTM D56).

The selection process described below evaluates the three general solubilization concepts individually.

7.1 Cosolvents

Aqueous acetone was omitted from further considerations due to its prohibitive flammability potential, even though the limited solubility data collected indicated it was potentially the most effective cosolvent system. Aqueous DMF was somewhat more effective in solubilizing the three "guide" explosives than was aqueous DMSO. Solubilization values of 0.03 to 0.13 percent were achieved with 30 percent DMF whereas solubilization values of 0.02 to 0.06 percent were achieved with 30 percent DMSO for these guide explosives. Solubilization values of 0.5 to 1.4 percent were achieved with 60 percent DMF whereas solubilization values of 0.3 to 0.4 percent were achieved with 60 percent DMSO for these explosives. These solubilization magnitudes for DMF and DMSO were assumed to be large enough to allow effective chemical decontamination of non-dissolved explosives by contact with 30 percent (or higher) DMF or DMSO solutions which contained chemical decontaminants. Both DMF and DMSO at 30 percent or greater concentrations are recommended as trial reaction solvents either in Subtasks 4 (Prescreening of Chemical Decontamination Concepts) or amended Subtask 6 (Evaluation of Candidate Decontamination Concepts on Steel and Concrete).

7.2 Surfactants

Surfactants were not evaluated further since they were judged to have little potential for solubilizing explosives to a greater extent than was demonstrated when they were tested as 5 percent solutions. The solubilities which were determined with the three "guide" explosives ranged from 0.04 to 0.16 percent in terms of using the best surfactant for each explosive. These solubilizations, which resulted from using 5 percent surfactant solutions, generally exceeded the solubilizations demonstrated by using 30 percent aqueous cosolvents. However, since some foaming was observed at these concentrations with simple stirring, it is expected that significant foaming would

result if higher concentrations of surfactants were employed in solutions which were applied by spraying on building surfaces.

Some limited surfactant testing was also performed with 5 percent Triton X-100 (apparently the most effective surfactant based on work with the "guide" explosives) on the explosives 2,6-DNT, tetryl and HMX. The resultant solubility values are shown in Tab¹ 11. It can be seen that HMX is negligibly solubilized by this surfactant system. Thus another reason for not employing surfactants is that they (specifically Triton X-100) do not have universal applicability in terms of application to all target explosives.

Another factor to be considered is that surfactants typically require solution shear or agitation to dissolve solid particles. Whereas the solubilization of explosives by surfactants was not studied under static conditions, if solution shear is required it would imply that explosives which had diffused behind building surfaces into the building matrix would not be effectively solubilized for further decontamination. On the other hand, we have demonstrated in several cases that explosives which were not stirred in the presence of aqueous cosolvents were solubilized to the same extent when stirring was employed. Thus explosives present within the building matrix may be solubilized better with cosolvent systems than surfactants.

7.3 Complexing Agents

By far the greatest aqueous solubility enhancements resulted from the solubilization of TNT by the complexing agents DEA and HEP. The solubilization magnitudes for TNT were approximately 4.0 percent and (greater than) 11.7 percent with 30 percent DEA and HEP respectively. The solubilization magnitudes for 2,4-DNT were 1.2-1.4 percent with these two complexing agents. However, complexing agents were not recommended for further study for several reasons.

One reason is that the complexing agents are not universally effective since RDX was found to be solubilized only to a marginal extent by these complexing agents.

It was also noted that the solid sediments which remained after treatment of TNT with these complexing agents were soluble in water, in

TABLE 11.   SOLUBILIZATION OF EXPLOSIVES BY FIVE PERCENT
AQUEOUS TRITON X-100 (GRAMS/100 ML SOLUTION)[a,b]
HPLC ANALYTICAL METHOD

Explosive	Time	
	24 Hr	48 Hr
2,6-DNT	0.24	0.27
Tetryl	0.040	0.013
HMX	0.0016	0.0027

(a) Temperatures were maintained
at 27 C in thermostatted and
jacketed beakers.

(b) Analytical samples were removed
after initial centrifugation
followed by filtration through
Millex SR 50 micron millipore
filters.

contrast to TNT itself which is quite water insoluble. These results imply that TNT has been converted to another distinct chemical species which presumably involves a new covalvent bond from the amine nitrogen atom to the aromatic nitro groups. The probable formation of modified nitro groups presents a dilema in terms of subsequently applying chemical decontaminantion concepts which the literature uncovered in Phase I indicates are reactive towards unaltered nitro groups or specific explosives. Thus, one reason for not using these complexing agents is that non-precedented chemistry may be encountered in further chemical decontamination work.

Another factor is the fact that TNT and 2,4-DNT (unlike RDX) formed dark black solutions upon contact with these complexing agents which presumably results from charge transfer interactions. It is not known if this color would persist after decontamination had occurred and whether subsequent washing would be required to remove these colored species. It should also be mentioned that DEA is listed as an irritant but the toxicities of DEA and HEP are unknown to us at this time.

8.0 VERIFICATION OF THE AQUEOUS COSOLVENT SOLUBILIZATION OF 2,6-DNT, TETRYL AND HMX

The solubilization of these three remaining target explosives were tested in aqueous DMF and DMSO to determine if these cosolvents were appropriate for the solubilization of these explosives. The solubilization data are shown in Tables 12, 13 and 14. It is apparent where data are available that equilibrium solubilities are nearly established at two hours (or before).

The solubilities of 2,6-DNT in 30 and 60 percent DMF ranged from 0.14 to 2.3 percent respectively. The solubilities of 2,4-DNT in the same solvent and relative concentrations ranged from 0.13 to 1.4 percent respectively. The solubilities of 2,6-DNT in 30 and 60 percent DMSO ranged from 0.07 to 0.7 percent respectively whereas the solubilities of 2,4-DNT in the same solvents and relative concentrations ranged from 0.06 to 0.5 percent respectively.

Similar analyses indicate that tetryl has approximately the same solubilities as TNT does in 60 percent DMF and DMSO mixtures but tetryl has

TABLE 12. SOLUBILIZATION OF 2,6-DNT BY AQUEOUS
COSOLVENTS (GRAMS/100 ML SOLUTION)
HPLC ANALYTICAL METHOD

Cosolvents	Percent	Time		
		2 Hr	25 Hr	48 Hr
DMF	30	0.105	0.146, 0.145	0.143, 0.137
	60	--	2.34[c],[d]	2.04[e]
DMSO	30	0.060	0.070, 0.066	0.070, 0.072
	60	--	0.68[c],[d]	0.63[e]

[a] Temperatures were maintained at 25 C in thermostatted beakers.

[b] Analytical solutions were removed after initial centrifugation of samples followed by filtration of supernatant through cotton filters.

[c] Determined after 27 hours.

[d] Run at ambient air temperature of 27 C.

[e] Run at ambient air temperature of 25 C.

TABLE 13. SOLUBILIZATION OF TETRYL BY AQUEOUS COSOLVENTS
(GRAMS/100 ML SOLUTION)[a,b] HPLC ANALYTICAL METHOD

| Cosolvents | Percent | Time | | |
		2 Hr	25 Hr	48 Hr
DMF	30	--	0.027, 0.026[d]	0.025, 0.025
	60	--	0.79[c,e]	0.68
DMSO	30	0.022	0.018, 0.018	0.018, 0.018
	60	--	0.33[c,e]	0.31

(a) Temperatures were maintained at 25 C in thermostatted beakers.

(b) Analytical solutions were removed after initial centrifugation of samples followed by filtration of supernatant through cotton filters.

(c) Determined after 27 hours.

(d) Filtered through Millex SR 50 micron filters.

(e) Run at ambient air temperature of 27 C.

TABLE 14. SOLUBIILZATION OF HMX BY AQUEOUS COSOLVENTS
(GRAMS/100 ML SOLUTION)[a,b] HPLC ANALYTICAL METHOD

| Cosolvents | Percent | Time | | |
		2 Hr	25 Hr	48 Hr
DMF	30	0.157	0.156, 0.153	0.144, 0.144
	60	--	0.22[c,e]	0.21
DMSO	30	--	0.006, 0.006[d]	0.012, 0.012
	60	--	0.14[c,e]	0.16

(a) Temperatures were maintained at 25 C in thermostatted beakers.

(b) Analytical solutions were removed after initial centrifugation
of samples followed by filtration of supernatant through cotton
filters.

(c) Determined after 27 hours.

(d) Filtered through Millex SR 50 micron filters.

(e) Run at ambient air temperature of 27 C.

slightly lower solibilities than TNT in 30 percent DMF and DMSO. DMF affords approximately twice the solubilization of tetryl than DMSO does at both the low and high concentrations tested.

Inspection of the solubilization data for HMX indicates that increasing the DMF percentage from 30 to 60 percent only increases the solubility approximately 40 percent (from .15 to .22), instead of the approximately 20 fold increases which were observed in the case of RDX and TNT with the same solvent change. The solubility of HMX is approximately 20 fold higher in 60 percent DMSO compared to 30 percent DMSO. Comparing the solubilization of HMX by DMF and DMSO at the 60 percent concentrations levels, it can be seen that DMF is approximately 50 percent more effective than DMSO.

9.0 SOLUBILIZATION OF EXPLOSIVES BY FREON 113*

The solubilities of all six target explosives were determined in Freon 113* at 25 C to determine if the Radkleen* process was applicable to the removal of explosives from building surfaces. The data is shown in Table 15 and indicates that 2,6-DNT, 2,4-DNT and TNT have solubilities ranging from .15 to 1.3 percent whereas tetryl has a solubility of only 0.005 percent but the solubilities of RDX and HMX are below 1×10^{-5} percent. Thus Freon 113* does not appear to be an appropriate general solvent for the extractive removal of the target explosives from building surfaces.

10.0 FINAL RECOMMENDATIONS

In general it appears that DMF is more effective than DMSO in solubilizing all six target explosives. However, it is recommended that both aqueous DMF and DMSO at minimum concentrations of 30 percent be used as trial solvent systems for the evaluation of chemical decontamination concepts in Subtask 4. One reason for also investigating DMSO as a cosolvent is that the use of DMSO as a solvent for Fenton's reaction has been shown to generate methyl radicals which have been implicated to be a highly reactive species involved in the decomposition of various explosives.

TABLE 15.   SOLUBILIZATION OF EXPLOSIVES BY FREON 113
(GRAMS/100 ML SOLUTION)[a,b] HPLC ANALYTICAL
METHOD

Explosive	Time			
	3 Hr	17 Hr	26 Hr	49 Hr
TNT	0.155	0.170	0.138	0.147
2,4-DNT	0.644	0.625	0.670	0.666
RDX	$<1 \times 10^{-5}$	$<1 \times 10^{-5}$	$<1 \times 10^{-5}$	$<1 \times 10^{-5}$
2,6-DNT	1.39	1.25	1.33	0.975
Tetryl	0.006	0.004	0.005	0.004
HMX	$<1 \times 10^{-5}$	$<1 \times 10^{-5}$	$<1 \times 10^{-5}$	$<1 \times 10^{-5}$

(a) Temperatures were maintained at 25 C in thermostatted beakers.

(b) Analytical solutions were removed after initial centrifugation of samples followed by filtration of supernatant through cotton filters.

For the purpose of solvent extraction of explosives, a minimum of 60 percent concentrations of DMF or DMSO is recommended. At this concentration, aqueous DMF is approximately 1.5 to 3 fold more effective in solubilizing all target explosive than DMSO.

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APPENDIX A

SUBTASK 3, TEST PLAN

TASK 4

SUBTASK 3

TEST PLAN

for

ENHANCED AQUEOUS SOLUBILIZATION

CONTRACT NUMBER DAAK11-81-C-0101

to

UNITED STATES ARMY
TOXIC AND HAZARDOUS MATERIALS AGENCY

from

BATTELLE
Columbus Laboratories

by

Herman P. Benecke

September 12, 1983

1.0 PURPOSE AND OBJECTIVES

Solubilization of explosives is required for the effective chemical decontamination in solution or the extractive removal of explosives. Water is the best potential solvent for the solubilization of explosives in terms of its minimal cost and the low safety hazards associated with its use. Each of the three chemical concepts which will be tested in Subtask 4.4 are compatible with and typically performed in aqueous based solvent systems. Furthermore, solvent extraction of explosives (studied in Subtask 4.5) by aqueous-based solvents would benefit from appreciable solubility in these solvents. However, each of the six explosives which will be investigated in these later studies have very low solubilities (about 0.01 percent or lower) in water. The

objective of this subtask is to evaluate those additives which may significantly increase the aqueous solubilities of each of these explosives.

Those additives which will be screened in this subtask for enhancement of the aqueous solubilities of explosives include the following type materials:

Surfactants. Cationic, anionic and nonionic surfactants will be investigated. Nonionic surfactants are further classified with respect to their hydrophilic, lipophilic balance (HLB). High, medium and low HLB nonionic surfactants will also be screened. Salts and organic additives will also be evaluated since they are known to enhance the solubilities of various solubilizates in surfactants. Those surfactants which are found to significantly enhance the aqueous solubilities of explosives will be tested further in Subtask 4.4 to determine their potential catalytic effect upon the rates of solution decontamination of the target explosives.

Complexing Agents. Those complexing agents which will be evaluated are diethanolamine and N-hydroxyethylpiperazine.

Cosolvents. The cosolvents dimethylsulfoxide (DMSO), dimethylformamide (DMF), and acetone will be evaluated.

These initial screening tests will be performed with the explosives TNT, RDX, and 2,6-DNT only since these explosives are known to have similar solubilities and are expected to respond similarily to solubilizing agents as would Tetryl, HMX, and 2,4-DNT respectively. Upon determining the optimum surfactant system, complexing agent, and cosolvent (and their optimum solubilizing concentrations) in prior screening tests, each of these three types of solubilizing agents will be then evaluated in the presence of the other two agents to evaluate their potential synergistic effects. All six target explosives will be examined in detail during the final cross evaluation of the three classes of solubilizing agents in the presence of each other.

2.0   TEST CONDITIONS AND PARAMETERS

Solubility Determinations

The solubility of an explosive will be determined by stirring
a slurry of that explosive at room temperature in the solvent system
being evaluated. The amount of explosive which has been solubilized may
be determined by either measuring the quantity of non-solubilized explo-
sive or by measuring the quantity of explosive which is present in the
liquid phase.

A variety of analytical methods, which are shown in
Figure 1, may be employed to perform the measurements. The most direct
method involves weighing the undissolved explosive (Method A). However,
this option is presently considered to be unsafe because of the poten-
tial for spark discharge during the drying and weighing of the undis-
solved explosive. Other potential methods involve spectrometric (UV
absorption), chromatographic, and calorimetric determinations. The
specific choice of analytical method may depend on the nature of the
additives employed and their predicted effect upon the analytical
method. For instance, complexing agents are expected to significantly
alter the spectral response of explosives in solution so that only
Method B would be appropriate in this case. Surfactants are also
expected to alter the explosives' spectral response in solution due to
micelle incorporation, so that micelle decomposition (by dilution with
organic solvent) will be required before final analysis in this case.
Preliminary evaluations will be performed at the initiation of this
subtask to determine which analytical methods give the most self-
consistent data and also may be most rapidly performed.

The time(s) at which solution saturation has been achieved
will be determined by successive sampling at time intervals until no
further increase in explosive concentration is observed. If the
saturation time is found to be predicted with confidence for a certain
solubilizing agent, intermediate sampling will be performed in a random
manner rather than in every case.

Explosive $\xrightarrow[\text{Additives}]{\text{Water}}$ Saturated Solution $\xrightarrow{\text{Filter}}$ Solid + Filtrate

```
                                                    /  \      /  |  \
                                                   A    B    C   D   E
```
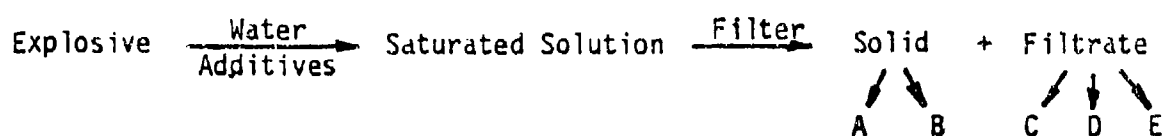
A. Wash with cold water, dry, and weigh.

B. Wash with cold water. Dissolve in organic solvent and quantitate by UV absorbance or DSC (Differential Scaning Calorimetry).

C. Direct HPLC analyses (Organic solvent composition required to dissociate micelles?).

D. Extract with organic solvent and quantitate by UV absorbance or DSC.

E. Direct UV or DSC quantitation (Organic solvent composition required to dissociate micelles?).

FIGURE 1. POTENTIAL SOLUBILIZATION ANALYTICAL METHODS

3.0 MEASUREMENTS

Complexing Agents and Cosolvents

It is anticipated that solution saturation will be achieved in a matter of minutes to hours at room temperature when explosives are stirred in the presence of complexing agents or organic cosolvents. Stirring will be performed in sealed vials with magnetic stir bars rotating at nearly constant rates. Three hundred milligrams of explosive will be used in each solubility measurement so that it constitutes 15 percent of the total mixture. This quantity will allow the determination of explosives' solubilities up to 15 weight percent. These solubilizing agents will be employed in quantities corresponding to 10, 20, and 30 weight percent of the total mixture. Higher percentages of these solubilizing agents will not be employed since it is estimated that higher concentrations would significantly reduce the solubilities of the inorganic decontamination agents which will be evaluated in Sub-task 4. A summary of the proposed variables matrix for evaluating complexing agents and cosolvents can be found in Table 1.

Surfactants

Solution saturation of explosives is expected to take significantly longer times in comparison to the time required to achieve saturation when cosolvents or complexing agents are employed. The solution process will be performed at room temperature in sealed vials in a laboratory rotating device. Since surfactant solutions will have appreciable viscosities, sample rotation will be required to provide equivalent mixing in different samples and also supply internal shear which will enhance the solubilization rate of the explosives. Traces of silicone oil will be employed to prevent foaming. Two hundred milligrams of each explosive will be used in each solubility measurement so that it constitutes 10 weight percent of the total mixture. This quantity will allow the determination of explosives' solubilities up to 10 weight

TABLE 1.   VARIABLES MATRIX FOR SCREENING COMPLEXING
AGENTS AND COSOLVENTS

Complexing Agent Concentration, (%)	Complexing Agent	
	Diethanolamine	N-Hydroxyethylpiperazine
10	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX
20	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX
30	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX
Number of Analyses	9	9

Cosolvent Concentration, (%)	Cosolvent		
	DMSO	DMF	Acetone
10	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX
20	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX
30	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX
Number of Analyses	9	9	9

percent. Sufactant loadings will correspond to 1 and 5 weight percent of the total mixture so that they exceed their published critical micellar concentrations (CMC). Upon determining the best anionic and nonionic surfactants, mixtures of the two types of surfactants will be tested to determine whether they act in synergism with each other (as has been observed elsewhere). The potentially beneficial effect of added ionic and organic additives will be evaluated either with this mixture of surfactants or the cationic surfactant, depending which system provides the greatest solubility enhancement of explosives. A summary of the proposed variables matrix for evaluating urfactants is shown in Table 2.

Cross Evaluation of Three Classes of Solubilizing Agents.

The potential synergistic effect of complexing agents, cosol-vents, and surfactants upon each other will be evaluated as a last stage of this subtask. All six explosives will be evaluated to validate that the explosives not tested until now respond similarily to solubilizing agents. A summary of the proposed variables matrix for this cross evaluation is shown in Table 3. Individual solubilizing agents will be employed at concentrations which are equal to and also are lower and higher than the optimum concentrations employed when these solubilizing agents were tested individually. These variations in concentration will allow the determination of the dependence of explosives' solubilities upon the relative concentration of the three types of solubilizing agents in the presence of each other. If it becomes obvious that combinations of solubilizing agents in certain concentrations lead to diminished solubility, those concentrations will not be utilized in further testing with other explosives.

TABLE 2. VARIABLES MATRIX FOR SCREENING SURFACTANTS

Surfactant Concentration, (%)	Anionic-1 (Carboxylate)	Anionic-2 (Sulfonate)	Cationic	Non-Ionic Low HLB	Non-Ionic Med. HLB	Non-Ionic High HLB
1	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT,RDX	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX	TNT; 2,6-DNT, RDX
5	"	"	"	"	"	"
Number of Analysis	6	6	6	6	6	6

	Best Anionic Surfactant (BAS)			
Best Non-Ionic Surfactant (BNIS)	TNT .5% BNIS*, .5% BAS 1% BNIS, 4% BAS 2.5% BNIS, 2.5% BAS 4% BNIS, 1% BAS	2,4-DNT .5% BNIS*, .5% BAS 1% BNIS, 4% BAS 2.5% BNIS, 2.5% BAS 4% BNIS, 1% BAS	RDX .5% BNIS*, .5% BAS 1% BNIS, 4% BAS 2.5% BNIS, 2.5% BAS 4% BNIS, 1% BAS	
Number of Analyses	4	4	4	

*Choices of total surfactant concentration (1% or 5%) will be guided by optimum concentration of individual surfactants

	Cationic or Best Surfactant Combination at Optimum Concentration Determined Above		
Ionic Additive (I.A.)	TNT 5% I.A. 10% I.A.	2,4-DNT 5% I.A. 10% I.A.	RDX 5% I.A. 10% I.A.
Organic Additive (O.A.).	TNT 5% O.A. 10% O.A.	2,4-DNT 5% O.A. 10% O.A.	RDX 5% O.A. 10% O.A.
Number of Analyses	4	4	4

TABLE 3. VARIABLES MATRIX FOR THE CROSS EVALUATION OF BEST COMPLEXING AGENTS, COSOLVENTS, AND SURFACTANTS WITH EACH OTHER*

| | Solubilizing Agent | | |
Explosive	Best Complexing Agent Relative Concentration	Best Cosolvent Relative Concentration	Best Surfactant System Relative Concentration
All Six Explosives	Optimum	Optimum	Optimum
	Low	Low	Low
	Low	Low	High
	Low	High	Low
	Low	High	High
	High	Low	Low
	High	Low	High
	High	High	Low
	High	High	High

Total Number of Analysis for Six Explosives   54

* Low and high concentrations in these cross evaluation tests refer to concentrations of surfactants which are 33 percent lower and higher than the optimum concentrations employed when these solubilizing agents were tested individually.

147

4.0 SCHEDULE OF TESTS

The estimated maximum number of solubility tests and final analyses which will be performed is 159. However, close to this number may also be performed in ascertaining the state of solution saturation during each solubility test. The eventual mode (or modes) of analysis which will be employed is not known at this time so that analytical through-put can not be estimated. More importantly, the cumulative times required to reach saturation is unpredictable and may be significant in the case of surfactants.

With these provisions in mind, the three screening studies will be performed in a staggered and overlapping sequence. The first screening study will involve surfactants and will tentatively start during the week of September 19. The experimental plan schedule is shown in Table 4. A letter test report detailing the results and the selection of the solubilization system which will maximize the aqueous solubility of all explosives will be submitted to USATHAMA for approval in the week of October 24, 1983.

5.0 CONTINGENCY PLANS

Contingency plans have been built into the test plan in appropriate areas. For example, Figure 1 lists a variety of analytical methods which will be initially evaluated for potential use. Various methods may be applicable to specific situations. Contingency plans were also incorporated into the schedule of tests to be run. Tests will be run simultaneously to most efficiently utilize time, personnel, and space without compromising laboratory safety.

TABLE 4. SCHEDULE OF EXPERIMENTS

Week 1	Week 2	Week 3	Week 4
Analytical Development			
Surfactants	Surfactants	Surfactants	
	Complexing Agents	Complexing Agents and Cosolvents	
			Mixtures of Solubilizing Agents

APPENDIX B

SOLUBILITY OF EXPLOSIVES IN ORGANIC SOLVENTS

APPENDIX B

SOLUBILITY OF EXPLOSIVES IN ORGANIC SOLVENTS

Explosive	Solvent	Temperature (°C)	Solubility (g/100 ml)
TNT	Aceton	20	109.0
RDX	Acetone	20	7.3
RDX	DMSO	25	41[a]
RDX	DMF	25	37[a]
HMX	DMSO	25	57[a]
HMX	DMF	25	forms insoluble solvate[a]
Tetryl	Acetone	20	75.0

(a) Sitzman, Michael E., et. al. Solubilities of High Explosives: Removal of High Explosives Fillers From Munitions by Chemical Dissolution. Naval Ordinance Laboratory NOLTR 73-186, November, 1973 (AD773078).

DEPARTMENT OF THE ARMY
US ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
ABERDEEN PROVING GROUND, MARYLAND 21010-5401

REPLY TO
ATTENTION OF

August 24, 1984

Technology Division

Dr. Eugene J. Mezey
Battelle
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Dear Dr. Mezey:

The test reports entitled "Enhanced Aqueous Solubilization of Explosives" and "Prescreening of Chemical Decontamination Concepts" have been reviewed and the Agency's comments are enclosed. These comments should be addressed in writing and incorporated into the final report where appropriate.

Sincerely,

Andrew P. Roach
Contracting Officer's
Technical Representative

Enclosures

Comments to Test Report Entitled
"Enhanced Aqueous Solubilization of Explosives"

Item	Page	Comment
1	2	While not totally incorrect, the discussion on rates of decontamination versus rates of solution or solubility is misleading. The rate of decontamination will be controlled by the lowest of three factors: the rate of the decontamination reaction, the rate of solution, or the solubility of the compound in the matrix. The rate equation for the decontamination reaction normally shows at least a first order dependence on the concentration of the compound which is a reflection of its solubility. Consequently, the higher the concentration is (the greater the solubility), the faster the rate of decontamination should be. Therefore, the statement in the report implying a solubility limit above which no increase in decontamination rates would be observed is probably erroneous. A similar situation does not exist when comparing the decontamination rate versus the rate of solution. Once the solution is saturated with the compound of interest, there is no advantage to a more rapid rate of solution than rate of reaction. However, starting at time zero, with the concentration of contaminant well below its saturation point, a rate of solution that is more rapid than the rate of decontamination will result in an increased concentration which will enable more rapid decontamination.
2	2, Sec 2.0	A comma is missing from 2,6-DNT.
3	3, Sec 3.0	The equipment name "Micrometrics" is misspelled.
4	4	Para 1, Line 4: Change "was" to "were."
5	4, Sec 4.0	Para 1, Line 2: Insert "of" so that the sentence reads "...a slurry of 200 mg of the explosive."
6	4	Para 2: The word "ylid" is misspelled."
7	5	Line 3: Insert "be" so that the sentence reads "...were presumed to be due to..."
8	5	Para 1, Line 7: The word "presumed" is misspelled.

Item	Page	Comment
9	6	A similar procedure for estimating solubility by weighing filtered residue was discussed at the top of page 5 and was found to yield non-reproducible results. These samples were filtered through Whatman filter paper, but no indication is given on the reproducibility of these results. Clarify if these results were any more reproducible. Also, the weight of the residue was used to estimate the quantity of explosive that was dissolved. However, indications are that the residue contained complexed explosive rather than neat explosive. Clarify if the additional weight of the complexing agent was compensated for in estimating the amount of undissolved explosive and, consequently, the amount of dissolved explosive. If so, clarify on what basis the portion of weight of the residue was estimated that was assigned to the complexing agent.
10	7, Sec 5.1.1	First sentence: Change "is" to "are."
11	7, Sec 5.1.1	Without knowing the exact mechanism of solution preparation, initial supersaturation of the solution followed by a decrease to equilibrium concentrations is difficult to believe. Supersaturation to an extent greater than three times the saturation concentration makes this phenomenon even more difficult to believe.
12	9, Sec 5.1.3	The "complexing" agents may be more decontaminants than complexing agents. Many explosives are known to react with basic compounds, and diethanolamine is highly basic. Depending on intended decontaminants, the amines may prove to be primary rather than secondary decontaminants.
13	16, Sec 5.21.	The argument provided to rationalize the observation of 2,4-DNT is not totally clear, and sufficient details of the "proof" are not given to demonstrate the validity of the conclusion. The absence of a significant 2,4-DNT peak in the 30% DMSO solution could indicate that the original TNT sample did not contain enough 2,4-DNT to leach out to yield the chromatogram shown in Figure 2. A more definitive test would have been to analyze the 30% DMSO solution over the time after 27 hours to show that no 2,4-DNT was being produced.
14	27, Sec 7.1	The word "amend" is misspelled.

February 15, 1985

Mr. Andrew P. Roach
U.S. Army Toxic and Hazardous
  Materials Agency
Building E-4585
Attn: DRXTH-TE-D
Aberdeen Proving Ground (EA)
  Maryland 21010

Dear Andy:

<div align="center">

Contract No. DAAK11-81-C09101
Novel Decontamination Technology
Task 4 Development of Techniques for
Explosive-Contaminated Facilities

</div>

This letter addresses USATHAMA's comments on the test report on Task 4,
Subtask 3 entitled "Enhanced Aqueous Solubilization of Explosives" dated
August 24, 1984. USATHAMA comments are attached for reference. This
letter is to be attached to the test report and covers the explanations or
actions taken in response to the Army's comments. The comments or actions,
addressed by item number, are as follows:

Item 1. The paragraph on page 2 of the test report was deleted in
order to avoid confusion based on speculation of the possible rate
limiting step during any chemical decontamination process. Battelle
adheres to the premise that decontamination of explosives by
chemicals that are most reactive when dissolved in water (whereas
explosives are sparingly soluble in water) requires that explosive
solubility be enhanced in water by some means. Once the explosive is
in solution the reaction between explosive and reagent can proceed.
It is obvious that the basic requisite is that the explosive be
solubilized so that reaction can occur with the water soluble
reagent. These objectives were clearly stated in the Test Plan for
this subtask attached to the test report.

Item 2-8 These comments have been incorporated into the report.

Item 9 The reproducibilities of the solubilities determined by the
filter, dry, and weigh method for TNT and RDX with complexing agents
are shown in Tables 6 and 9 respectively (Reader is now directed to
these sections).

As stated, we did have indications that the residue which was weighed did contain complexing agent in the case of TNT. Since we did not determine the relative contribution of the complexing agent towards the composition of the residue, the solubility values for TNT which are tabulated represent minimum rather than exact solubilities.

Item 10 Change has been made.

Item 11 We agree that initial supersaturation to values significantly greater than the true solubility are difficult to explain. However, we believe that the rapid stirring may have milled the particles to a small size which would have increased the surface area resulting in an apparent and temporary state of supersaturation. As the particle sizes grow, the equilibrium solubility is attained.
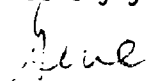
Item 12 We agree that amines could act as decontaminants (Section 7.3). However, the distinction between a complexing agent and a decontamination for explosives may be made by determining whether the interaction is reversible (a complexing agent) or not reversible (a decontaminant). We believed that resolution of such a destinction was beyond the scope of this work. Our description of this concept (p III-274 of Report DRX7H-TE-CR-83211) indicates that the interactions of explosives and the type of complexing agents which we employed would interact in a reversible fashion.

Item 13 We believe our tests conclusively demonstrated that TNT was not denitrated to 2,4-DNT during its solubilization in aqueous cosolvents since the same TNT was used in the solubilization studies in aqueous 30 percent DMSO (partial solubilization) and the solubilization studies which involved initial and complete solubilization in DMSO which was followed by dilution with water to give a 30 percent aqueous DMSO solution. The fact that only a trace of 2,4-DNT was evident in the resulting 30 percent solution after long periods of time (several weeks) showed that the appearance of significant 2,4-DNT in the former case was caused by selective leaching of the small quantities of the 2,4-DNT present in the TNT sample. If denitration of TNT were occurring, the process would be expected to proceed with time and more denitration product would be evident.

Item 14 Correction was made.

Should you have any questions on these responses, please call me at (614) 424-4995 or Dr. Herman Benecke at (614) 424-4457.

Sincerely yours,

E.J. Mezey
Project Manager

EJM/sj

Attachment

APPENDIX III-2

SUBTASK 4 - PRESCREENING OF CHEMICAL DECONTAMINATION
CONCEPTS - TEST REPORT

PREVIOUS PAGE
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TEST REPORT

for

TASK 4 SUBTASK 4
PRESCREENING OF CHEMICAL
DECONTAMINATION CONCEPTS

Contract DAAK11-81-C-0101

to

UNITED STATES ARMY
TOXIC AND HAZARDOUS MATERIALS AGENCY

February 15, 1985

by

H.P. Benecke
S.S. Harsh
and
E.J. Mezey

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

EXECUTIVE SUMMARY

The cosolvent systems of water and dimethyl sulfoxide (DMSO) or demethylformamide (DMF) judged most suitable in Subtask 3 for dissolution of the six explosives under study were used for the evaluation of three chemical decontamination reactions. The decontamination effectiveness of solutions of sodium hydroxide, sodium sulfide, sodium disulfide and Fenton's Reagent were determined. The variables studied with 2,4-DNT, TNT and RDX were water to solvent ratio, temperature, and reagent concentration. The nature of the reaction products were also determined. The most acceptable chemical decontamination concept for explosives is judged to be a solution of sodium hydroxide in aqueous DMSO (30% or 75% DMSO). A solution of sodium sulfide is even a more effective decontaminant but was rejected because of toxic characteristics of the reaction products.

TABLE OF CONTENTS

LIST OF TABLES

TEST REPORT

for

TASK 4 SUBTASK 4

PRESCREENING OF CHEMICAL DECONTAMINATION CONCEPTS

Contract DAAK11-81-C-0101

to

UNITED STATES ARMY

TOXIC AND HAZARDOUS MATERIALS AGENCY

from

BATTELLE

Columbus Laboratories

February 15, 1985

1.0 INTRODUCTION

Decontamination of previously utilized explosives manufacturing, loading and assembly facilities is necessary to allow the Department of the Army to restore the facilities for alternate use or to dispose of them in excessing actions. Included in the facilities are the buildings, structures, sumps, processing equipment, underground and above-ground storage tanks, and associated transfer systems. Facility decontamination involves not only the decontamination of exposed surfaces but also the decontamination of trace quantities of explosives which may have penetrated into the material through pores, cracks, or other openings.

The development of novel concepts for the decontamination of explosives-contaminated buildings and equipment is being carried out by Battelle Columbus Laboratories (BCL) for the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) under Contract No. DAAK11-81-C-0101. The explosives of interest are TNT, 2,4-DNT, 2,6-DNT, RDX, HMX, and tetryl. In the previous phase (Task 2), ideas were systematically developed into concepts for decontaminating buildings and equipment. These concepts were evaluated and ranked with respect to technical and economic factors. Three concepts were selected for evaluation in Task 4: Use of sodium hydroxide

solution, solutions of sulfur-based reductants, and Fenton's reagent solutions. The screening decontamination evaluations were only performed with TNT, 2,4-DNT and RDX since they are expected to respond similarily to decontaminants as would tetryl, 2,6-DNT, and HMX respectively. This report details the results of the preliminary laboratory evaluation of these three chemical decontamination concepts with these three explosives (See Test Plan, Appendix A).

2.0 OBJECTIVES

The objective of this study was to provide preliminary evaluation and determination of working conditions of the three proposed chemical decontamination concepts so that 1-3 concepts may be recommended for further evaluation in Subtask 6. The chemical system concepts which have been evaluated are:

- Sodium hydroxide solutions
- Sulfur-based reductants - both sodium sulfide and disulfide solutions
- Fenton's reagent solutions.

The primary criterion for evaluation was the decontamination efficiency of each system. This efficiency was primarily measured by determining the time needed to reach concentrations of explosives which were reduced by 1000 fold (or greater) from that of the initial explosives concentration. The secondary criterion used for concept evaluation was the identification of the major products formed during the decontamination reactions and an assessment of their potential toxicity based on our general awareness of toxic compounds. Since the objective of this study was to study building decontamination, those concepts which were judged to produce potentially toxic products were to be eliminated from further consideration. Identification of major reaction products is to be done by gas chromatography/mass spectroscopy (GC/MS).

3.0 TEST EQUIPMENT AND MATERIALS

The RDX was received as a suspension in isopropanol from Holston AAP (Batch No. 4RC 14-71). Prior to use, approximately 2 gram samples were dried

under vacuum in a shielded dessicator to remove the alcohol. The 2,4-DNT was received from Aldrich Chemical Company. The TNT used in these studies was munition grade and was obtained through internal transfer from another Army project.

A Lauda Model B-1 circulating constant temperature bath was used for decontamination studies performed at 25 C. For decontamination studies performed at 65 C, three water jacketed beakers each containing mineral oil were connected in series to the constant temperature bath. The beakers were insulated with cotton and foil. The 147 C run was performed in an oil bath heated by a hot plate.

Explosive concentrations were determined with High Performance Liquid Chromatography (HPLC). The HPLC system used for analyzing the kinetic samples consisted of the following instrumentation and parameters:

Instrumentation

- Altex 110 A pump
- LDC Spectromonitor III - UV detector
- Micrometrics 725 Autoinjector
- Hewlett Packard 1000 Computer with Computer Assisted Laboratory System (CALS) Chromatographic Software

Parameters

- Column: Spherisorb OD 5M, 25 cmx4.6 mm I.D.
- Mobile Phase: methanol/water, 50/50 isocratic
- Flow Rate: 1 0 ml/min
- Detector: UV @ 254 nm
- Attenuation: unattenuated output to computer
- Injection volume: 10μl

GC/MS analysis was performed on a Finnigan Model A 1020 using chemical ionization (either with ammonia or methane) as the mode of ionization. A fused silica capillary column coated with SE-52 (30 meters x 0.025 mm) was used for GC separation.

4.0 EXPERIMENTAL PROCEDURES

A USATHAMA approved test plan (see Appendix A) was used as the basis for testing the various decontamination concepts.

Prior to beginning decontamination studies using TNT, 2,4-DNT, and RDX, stock solutions of the explosives, as well as solutions of quench materials, were prepared in both 30% dimethylformamide (DMF) and 30% dimethyl sulfoxide (DMSO). The stock explosive solutions (500 ml) were formulated by first dissolving the explosive (125 mg of TNT and 2,4-DNT and 100 mg of RDX) in neat organic solvent (150 ml) and then diluting with water (350 ml). Solutions were maintained at 25 C in a water bath (Fisher Model 125) throughout the testing period. The explosive solutions were monitored by HPLC over a period of a week to ascertain that the concentrations remained constant. The different explosives concentrations were chosen so they did not exceed the solubilities previously determined in Subtask 3 studies for the two cosolvent systems.

Stock solutions of explosive in combination with surfactants were also made up as described by initially dissolving the explosives and surfactants in neat organic solvents before dilution with water.

Decontamination determinations were conducted using two methods. For tes' using 30% cosolvent, the stock explosive solution (40 ml) was pipetteteinto the reaction vessel and the decontamination agent (10 ml) added. Glass volumetric pipets were used for dispensing the solutions. After use, they were rinsed with acetone, soaked in 20% nitric acid overnight, washed with water and with acetone and dried with a stream of nitrogen. The reaction vessels used were 100 ml three neck round-bottom flasks equipped with a mechanically stirred Hirschberg paddle. These reactions were conducted in a constant temperature bath maintained at 25 C. For testing at elevated temperatures and for tests involving higher percentages (>30%) of cosolvent, weighed amounts of explosive were placed in a 50 ml one-neck round bottom flask equipped with a water cooled West condensor and magnetic stirrer. The decontamination solutions were added directly to the solid explosive and solubilization was complete in less than a minute.

During each of the kinetic/decomposition experiments, samples (1 ml) were removed periodically by means of an Eppendorf pipet. The 1 ml kinetic samples were added to the appropriate quench solution (generally 100 µl quantities of chilled solution) also measured by Eppendorf pipet. The pH of the quenched material was checked using color pHast* indicator sticks,

pH 0-14. The pH of the kinetic solutions was measured with a Radiometer pH Meter 26. The quenched aliquots were either diluted with measured amounts of methanol and submitted for HPLC analysis or analyzed without further dilution.

All kinetic experiments were accompanied by a blank prepared using the same ratios of constituents but omitting the decontaminating agent and substituting it with water. The percentage of decontamination was calculated by dividing the HPLC response area count of the explosive remaining compared to the area of the time zero blank. The minimum detectable area counts (unitless) were determined for each explosive and found to be 2.9, 6.8, and 6.3 for RDX, TNT and 2,4-DNT respectively.

4.1 Experiments With Sodium Hydroxide
In Thirty Percent DMSO or DMF

Forty milliliters of explosive stock solutions (0.025 weight/volume percent for TNT and 2,4-DNT and 0.020 weight/volume percent for RDX) in 30 percent aqueous cosolvents were initially added to three-necked 100 ml round-bottom flasks in a 25 C water bath and allowed to equilibrate for ten minutes. Ten milliliters of 0.50M or 0.005M sodium hydroxide in 30 percent aqueous cosolvent were then added to give kinetic solutions which were 0.10M or 0.001M in sodium hydroxide respectively. The resulting final concentration of TNT and 2,4-DNT were 0.020 percent and the final concentration of RDX was 0.016 percent. Time zero was taken when one half of the stock sodium hydroxide solution had been added. The more concentrated kinetic solutions were found to remain at pH 13 throughout the reactions. One ml samples were withdrawn and added to one dram vials containing 100 µl of either 1.0M or 0.01M cold hydrochloric acid for the higher or lower concentrations of sodium hydroxide solutions respectively. The pH of the quenched solutions was found to be 6 or lower. Samples for HPLC analysis were prepared by diluting 100 µl of the quenched solution with 1900 µl of methanol. HPLC analyses of the decontamination of 2,4-DNT, TNT, and RDX with 0.10M sodium hydroxide are shown in Tables 1,2, and 3 respectively. The analytical results for the decontamination of TNT with 0.001M sodium hydroxide are shown in Table 4. In

TABLE 1. HPLC ANALYSIS OF 2,4-DNT DECONTAMINATION WITH 0.1 MOLAR SODIUM HYDROXIDE

Decon. No. 1 30 Percent DMSO			Decon. No. 2 30 Percent DMSO(a) Plus CPBr			Decon. No. 3 30 Percent DMF			Decon. No. 4 30 Percent DMF Plus CPBr		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	847.8	0	0	843.8	C	0	817.9		0	821.0	0
17	770.4	9.1	17	78.9	90.6	17	865.4	None	17	771.1	6.1
30	799.6	5.7	30	44.3	94.7	30	842.1	None	30	777.7	5.3
45	744.1	12.2	45	13.0	98.5	45	836.6	None	45	836.6	0
70	643.3	24.1	70	BDL	>99.2	70	875.9	None	70	784.8	4.4
185	309.7	63.5									

Decon. No. 5 30 Percent DMSO			Decon. No. 6 30 Percent DMSO Plus CPBr			Decon. No. 7 30 Percent DMSO Plus CEME(b)		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	15544.0	0	0	801.3	55.9	0	964.0	0
30	12762.6	17.9	4	353.0	55.9	4.25	507.8	47.3
120	8468.4	45.5	6	245.0	69.4	6.08	389.5	59.6
240	4084.6	73.7	8	191.6	76.1	8	305.1	68.4
516	516.4	96.7	12	131.3	83.6	12	177.3	81.6
1260	79.2	99.5	16	97.7	87.8	16	127.6	86.8
1441	48.5	99.7	24	77.3	90.4	24	40.8	95.8
1627	73.9	99.5	30	42.9	94.6	30	20.2	97.9
						45	BDL	>99.3
						70	BDL	>99.3

(a) CPBr = Cetyl pyridinium bromide

(b) CEME = Cetyl-N-ethyl morpholinum ethosulfate

TABLE 2. HPLC ANALYSIS OF THE DECONTAMINATION WITH 0.1 MOLAR SODIUM HYDROXIDE

Decon. No. 1 30 Percent DMSO			Decon. No. 2 30 Percent DMSO Plus CPBr(a)			Decon. No. 3 30 Percent DMF			Decon. No. 4 30 Percent DMF Plus CPBr		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	695.5	0	0	732.1	0	0	679.1	0	0	674.7	0
4	265.6	61.8	4	69.2	90.5	4	547.1	19.4	17	220.2	67.4
6	140.9	75.7	6	58.3	92.0	6	484.9	28.0	30	166.7	75.3
8	192.1	72.4	8	48.9	93.3	.8	456.6	32.8	45	84.9	84.4
12	86.9	87.5	12	52.9	92.8	12	361.9	46.7	70	8.9	98.7
16	104.2		16	54.4	92.6	16	287.3	57.7			
24	69.2		24	87.8	83.0	24	205.6	69.7			
45	58.0		30	45.7	93.3	30	159.0	76.6			
70	70.9	89.8									

Decon. No. 5 30 Percent DMSO			Decon. No. 6 30 Percent DMSO Plus CPBr			Decon. No. 7 30 Percent DMSO Plus CEME(b)		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	15433.3	0	0	728.9	0	0	775.3	0
30	1735.7	88.8	4	75.8	89.6	4	29.1	96.2
120	1229.9	92.0	6	66.6	90.9	6	29.1	96.2
241	904.6	94.1	8	55.1	92.4	8	20.1	97.4
516	688.1	95.5	12	47.5	93.5	12	14.5	98.1
1260	344.2	97.8	16	42.2	94.2	16	8.4	98.9
1440	309.2	93.0	24	33.6	95.4	24	BDL	>99.1
1629	589.7	95.2	30	34.6	95.3	30	BDL	>99.1
			45	25.8	96.5	45	BDL	>99.1
			70	22.2	97.0	70	BDL	>99.1

(a) CPBr = Cetyl pyridinium bromide

(b) CEME = Cetyl-N-ethyl morpholinum ethosulfate

TABLE 3. HPLC ANALYSES OF RDX DECONTAMINATION
WITH 0.1 MOLAR SODIUM HYDROXIDE

30 Percent DMSO		
Time	Area	% Decon
0	5657.1	0
10	298.0	94.7
30	3.0	99.95
60	1.9	99.97
120	BDL	> 99.95

TABLE 4.   HPLC ANALYSES OF TNT DECONTAMINATION
WITH 0.001 MOLAR SODIUM HYDROXIDE

	Decon. No. 1 30 Percent DMSO			Decon. No. 2 30 Percent DMSO + CPBr	
Time	Area	% Decon	Time	Area	% Decon
0	710.8	0	0	625.3	0
8	652.5	8.2	8	280.8	55.1
17	611.5	13.9	17	214.7	65.7
30	597.6	15.9	30	129.5	79.3
45	463.3	34.8	45	120.0	80.8
70	485.4	31.7	70	160.5	74.3
100	350.1	50.7	100	130.6	79.1
130	299.4	57.9	130	134.2	78.5

decontamination number 5 of Table 1 (2,4-DNT), a more concentrated sample for HPLC analysis was generated by diluting the entire quenched solution with 400 µl methanol. The quench solutions of decontaminations number 5 of Table 2 (TN) and number 1 of Table 3 (RDX) were analyzed without dilution with methanol.

4.2 Experiments With Sodium Sulfide and Sodium Disulfide in Thirty Percent DMSO

A stock solution of sodium sulfide nonahydrate was prepared as a 25 percent (w/v) solution in 30 percent DMSO. A 1.25 percent solution of sodium sulfide nonahydrate in 30 percent DMSO was also prepared by making a 1 to 20 dilution of the 25 percent stock solution.

The kinetic reactions, were initiated by adding ten milliliters of the sodium sulfide solutions to forty milliliters of the 30 percent DMSO stock solutions of explosives. These dilutions resulted in a final sodium sulfide nonahydrate concentrations of 5.0 and 0.25 percent.

The TNT and 2,4-DNT concentrations in the kinetic reaction mixture was 0.02 percent whereas it was 0.016 percent for RDX. Both reactant stock solutions and all explosive stock solutions were maintained at 25 C before and during use.

Kinetic reaction mixtures were quenched by withdrawing 1 ml samples and adding this to 100 µl of 4.2M HCl. This process converted all excess sodium sulfide to hydrogen sulfide and produced a milky solution. Before anlaysis by HPLC, samples were filtered using a Millex SR-0.5 micron disposable filter. Results of the HPLC analysis of quenched samples from the reaction of 2,4-DNT, TNT, and RDX with sodium sulfide (both concentrations) are shown in Table 5. In decontamination number 1 (2,4-DNT), the sample was prepared for HPLC analysis by mixing 500 µl of the quenched sample with 1500 µl of methanol. This dilution produced a clear solution and was not filtered. In decontamination number 4 (2,4-DNT), the sample was prepared for HPLC analysis by mixing 1000 µl of the quenched sample with 500 µl of methanol and the filtering solid from this mixture. In decontamination numbers 2 and 5 (TNT) and number 3 (RDX), samples were prepared for HPLC analysis by diluting

TABLE 5. HPLC ANALYSIS OF 2,4-DNT, TNT AND RDX DECONTAMINATION WITH SODIUM SULFIDE

Decon. No. 1 30% DMSO 2,4-DNT/5% Sodium Sulfide			Decon No. 2 30% DMSO TNT/5% Sodium Sulfide (Aged)			Decon No. 3 30% DMSO RDX/5% Sodium Sulfide		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	4371.6	0	0	11305.3	0	0	4164.9	0
2	19.3	99.6	10	4453.9	60.6	10	13.6	99.7
4	411.7	90.6	30	1713.4	84.8	30	BDL	>99.93
6	141.8	96.8	60	1885.0	83.3	60	BDL	>99.93
8	413.8	90.5	120	1657.5	85.3	120	BDL	>99.93
12	511.6	88.3	180	1233.3	89.1			
16	482.4	88.9	325	544.9	95.2			
24	474.9	89.1	1140	BDL	>99.94			
36	284.6	93.5						
70	126.2	97.5						

Decon. No. 4 30% DMSO 2,4-DNT/5% Sodium Sulfide			Decon. No. 5 30% DMSO/5% Sodium Sulfide (Fresh)			Decon No. 6 30% DMSO TNT/0.25% Sodium Sulfide		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	13536.5	0	0	11385.8	0	0	7902.2	0
10	10779.2	20.4	10	3601.9	68.4	2	5531.9	30.5
30	7002.2	48.3	30	1804.5	84.2	4	3437.1	56.5
60	2220.5	83.6	60	1521.1	86.6	8	1933.2	75.5
120	85.1	99.4	120	1412.8	87.6	16	808.8	89.7
180	BDL	>99.95	180	1412.8	87.6	16	808.8	89.7
240	BDL	>99.95	363	547.3	95.2	24	497.4	93.7
390	BDL	>99.95	1128	BDL	>99.94	32	354.4	95.5
			1490	BDL	>99.94	45	222.8	97.2
						70	134.6	98.3

1100 µl with 500µl of methanol and then filtering this mixture. For comparison purposes, decontamination number 2 was performed using an aged 25 percent sodium sulfide stock solution (approximately two months) and decontamination number 5 was performed using a stock solution of fresh 25 percent sodium sulfide. Decontamination number 6 (TNT) was performed using a stock solution of 1.25 percent sodium sulfide. The concentration of sulfide in the reaction mixture was 5 and 0.25 percent respectively. Samples (1 µl) from this decontamination were quenched with 100 µl of 1.0 M hydrochloric acid. The sample was prepared for HPLC analysis by diluting 1000 µl of the quenched solution with 1000 µl of methanol. Since no cloudiness developed, no filtration was performed.

The decontamination effectiveness of sodium disulfide was also studied. Sodium disulfide was prepared according to Gabel and Shpeier. (J. Gen. Chem 17, 2277-8 (1947)). Sodium sulfide (24.0g) nonahydrate was placed in an Erlenmeyer flask equipped with a magnetic stirrer and 300 ml of ethanol was added. After heating to boiling, the solution was cooled and the undissolved solid was filtered, dried and weighed to give 2.6g. (10.8%). Sublimed sulfur (2.85g) was added to the boiling solution. After boiling off excess ethanol, the precipitated solid was filtered, washed, and vacuum dried to give 7.05g solid (39.5%). Analyses by Galbraith Laboratories indicated that the percent of sodium and sulfur were 20.99 and 29.08 respectively which corresponds closely to the elemental analysis expected for sodium disulfide hexahydrate. This material was used to prepare standard stock solutions of 25 percent and 1.25 percent in 30 percent DMSO.

The results of the HPLC analysis of quenched samples from the decontamination of 2,4-DNT and TNT with sodium disulfide are shown in Table 6. One decontamination was performed using a stock solution of 25 percent sodium disulfide with TNT (starting disulfide concentration of 5 percent). Samples were prepared for HPLC analysis by adding one milliliter of the decontamination solution to 100 µl of 4.2 M hydrochloric acid. This mixture was filtered through Millex GS-22 micron filters. These quenched solutions (100 µl) were diluted with 1900 µl of methanol prior to HPLC analysis. Two decontamination studies were made between 0.25 percent sodium disulfide and TNT and 2,4-DNT. For quenching the more dilute sodium disulfide solutions, 1.0M HCl was used. It was unnecessary to filter these quenched samples since

TABLE 5. HPLC ANALYSIS OF 2,4-DNT AND TNT
DECONTAMINATION WITH SODIUM DISULFIDE

TNT 30% DMSO/5% Sodium Disulfide			TNT 30% DMSO TNT/0.25% Sodium Disulfide			2,4-DNT 30% DMSO/0.25% Sodium Disulfide		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	701.0	0	0	8299.8	0	0	13538.5	0
2	24.2	55.5	2	4571.8	44.9	2	13099.5	3.2
4	16.4	67.7	10	60.3	99.3	10	13043.7	3.7
7	12.2	98.3	30	44.9	99.5	30	13048.1	3.6
9	0.5	98.7	60	39.4	99.5	60	12976.2	4.2
11	BDL	>99.0	120	42.3	99.5	120	13037.7	3.7
14	BDL	>99.0	180	44.2	99.5	180	12904.4	4.7
17	BDL	>99.0	240	50.5	99.4	240	13017.2	3.9
			405	32.6	99.6	390	12974.8	4.2
			1297	38.5	99.5	570	12874.7	4.9
						1180	12928.4	4.5

no cloudiness developed. In the case of T , the HPLC sample was prepared from 1 ml of quenched solution plus 1 ml of ethanol. For 2,4-DNT, 1 ml of quenched solution plus 500 μl of methanol was used.

4.3 Experiments with Fenton's Reagent in Thirty Percent DMSO

Stock solutions of ferrous sulfate and hydrogen peroxide were prepared. Twenty grams of ferrous sulfate heptahydrate and 5.2 ml of concentrated sulfuric acid were diluted to 100 ml with 30 percent aqueous DMSO. A 1.08M solution of hydrogen peroxide was prepared in 30 percent DMSO. A 100 ml solution containing 0.1 ml of Catalase (Sigma, 750,000 units per 2 ml) was also prepared in pH7 buffer and used to quench the reaction.

The experiments with Fenton's Reagent were performed by first adding 5 ml of the 20 percent ferrous sulfate solution to 40 ml of stock explosive solution in a 100 ml round bottom flask after which 5 ml of the hydrogen peroxide solution was immediately added. Quenched samples for HPLC analysis were prepared by adding 1 ml of the kinetic solutions to 140 μl of 2.5M sodium hydroxide (to adjust the pH to 7). Then 860 μl of catalase/buffer solution was added. This amount of catalase was estimated to be in significant excess over the amount needed to decompose all hydrogen peroxide which was initially present. These quenched solutions were filtered through Millex SR-0.5 micron filters before analysis. The results for 2,4-DNT, TNT, and RDX are shown in Table 7 and show that maximum decontamination was reached in the first 2-10 minutes of the 1300-1517 minute monitoring period. These decontaminations efficiencies ranged from 55 to 98 percent.

To determine if these same reaction mixtures would undergo further decomposition by reapplication of Fenton's reagent, the following experiments were performed. To 8 ml of the original decontamination solutions was added sequentially 1 ml of 20 percent ferrous sulfate solution followed by 1 ml of 1.08M hydrogen peroxide. The reaction was carried out in an 11 dram vial with reagents equilibrated at 25 C. HPLC samples were taken at 8 and 30 minutes, as described above. A blank was prepared from one ml of original kinetic plus 250 μl of H_2O. To 1 ml of this solution was added 140 μl of H_2O and 860 μl of catalase. The results of this additional treatment performed on all three explosives as shown in Table 8.

TABLE 7. HPLC ANALYSIS OF 2,4-DNT, TNT AND RDX DECONTAMINATION WITH FENTON'S REAGENT

2,4-DNT 30% DMSO			TNT 30% DMSO			RDX 30% DMSO		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	10170.4	0	0	8432.2	0	0	3135.7	0
2	1544.4	84.8	10	174.8	97.9	2	1319.3	57.9
6	1547.7	84.8	30	139.6	98.3	6	1676.6	46.5
10	1778.1	82.5	60	254.8	97.0	10	898.5	71.3
30	1424.1	86.0	120	228.1	97.3	30	1291.0	58.8
60	1636.3	83.8	180	165.8	98.0	60	1199.4	61.8
120	1673.2	83.5	240	198.6	97.6	120	1422.4	54.7
180	1624.0	84.0	405	251.9	97.0	180	1300.3	58.5
305	989.3	90.3	1300	201.1	97.6	307	1153.8	63.2
545	1316.6	87.1				547	1337.9	57.3
1515	1504.9	85.2				1517	1405.7	55.2

TABLE 8. SECONDARY TREATMENT OF TNT, RDX, AND 2,4-DNT WITH FENTON'S REAGENT

	TNT			RDX			2,4-DNT	
Time (Min)	HPLC Area	% Decomposition	Time (Min)	HPLC Area	% Decomposition	Time (Min)	HPLC Area	% Decomposition
0	825	0	0	1925	0	0	4938	0
8	263	68.1	8	1352	29.8	8	2317	53.1
30	244	70.4	30	1600	16.9	30	2214	55.2

4.4 Experiments with DS2

DS2 (which is composed of 70 percent diethylenetriamine, 28 percent ethylene glycol mono-methyl ether and 2 percent sodium hydroxide) was tested as a decontamination reagent for 2,4-DNT, TNT, and RDX. Fifty ml of DS2 were placed in 100 ml round bottom flasks and accurately weighed amounts of explosive (approximately 50 mg or 0.1 percent) were added. One ml of the decontamination solutions was quenched with 1200 µl of conc. HCl (with considerable spattering & foaming) to give a final pH of approximately 7. Samples for HPLC analysis were prepared by diluting 500 µl of the quenched solutions with 500 µl of DMSO. DMSO rather than methanol was used to prevent precipitation of a salt from these solutions. Blanks were prepared using weighed amounts of explosive (~10 mg) in 10 ml of DMSO. One ml was diluted with 1 ml DMSO to prepare the sample for HPLC analysis. The time zero area counts were estimated by calculating the area counts the blank samples would have if they had the same concentration as the explosive in the DS2 solution. The results for this study are shown in Table 9.

4.5 Experiments With Sodium Hydroxide in High
Percentages of DMSO

Various compositions of sodium hydroxide in DMSO/water were prepared to determine the percentage of water necessary to maintain the solubility of 0.1M sodium hydroxide. The minimum percentage of water found necessary to solubilize 0.1M sodium hydroxide in DMSO was found to be 25 percent.

Weighed amounts of explosive (12.5 mg of TNT and 2,4-DNT, and 10.0 mg of RDX) were treated with 40 ml of 0.1M sodium hydroxide in 75 percent aqueous DMSO at three temperatures: 25, 65 and 148 C. The low temperature decontaminations were conducted in thermostatted water baths in 11 dram capped vials equipped with stir bars. The elevated runs were done in thermostated oil baths in round bottom flasks equipped with stir bars and water cooled condensors. For HPLC analyses, one ml of the decontamination solutions were quenched with 100 µl 1.0 hydrochloric acid. No further dilution with methanol was made for HPLC analyses. The results for the three explosives in 75 percent DMSO at these three temperatures are given in Table 10.

TABLE 9. HPLC ANALYSIS OF 2,4-DNT, TNT AND RDX
DECONTAMINATION WITH DS2

2,4-DNT			TNT			RDX		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	22312.8	0	0	17148.5	0	0	8224.1	0
30	1285.4	94.2	31	919.1	94.6	10	15.4	99.8
60	1368.2	93.9	60	1728.3	89.9	30	BDL	>99.96
115	1302.8	94.2	115	730.3	95.7			
340	780.6	96.5	340	1277.8	92.5	120	15.5	99.8
973	624.1	97.2	974	610.7	96.4			

TABLE 10. HPLC ANALYSIS OF 2,4-DNT, TNT AND RDX WITH
0.1 MOLAR SODIUM HYDROXIDE IN 75 PERCENT DMSO

TEMPERATURE: 25 C

	2,4-DNT			TNT			RDX	
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	2893.19	0	0	23139.8	0	0	8647.3	0
30	1183.0	95.9	30	14918.0	35.6	30	6.9	99.9
60	276.1	99.1	60	12060.5	47.9	60	8.6	99.9
180	103.7	99.6	180	7352.4	68.2	180	10.3	99.9
300	77.3	99.7	300	5205.6	77.5	300	8.7	99.9
685	BDL	>99.98	688	1797.8	87.9	682	8.9	99.9
1212	BDL	>99.98	1215	981.6	95.8	1209	7.4	99.9

TEMPERATURE: 65 C

	2,4-DNT			TNT			RDX	
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	28931.9	0	0	23139.8	0	0	8647.3	0
59	BDL	>99.98	62	125.2	99.5	56	9.9	99.9
118	BDL	>99.98	119	54.4	99.8	118	BDL	>99.97
364	76.9	99.7	367	66.6	99.7	361	7.9	99.9
1042	29.6	99.9	1045	80.3	99.7	1039	8.6	99.9

TEMPERATURE: 147 C

	2,4-DNT			TNT			RDX	
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	28931.9	0	0	23139.8	0	0	8647.3	0
160	30.4	99.9	157	846.2	96.3	159	23.8	99.7
300	16.1	99.94	307	4.6	99.98	304	6.2	99.93
923	13.8	99.95	919	7.4	99.97	922	7.8	99.91
1449	9.4	99.96	1445	6.9	99.97	1448	BDL	>99.97

4.6 Experiments With Saturated Sodium Hydroxide In Neat DMSO

Two grams of pelleted sodium hydroxide were stirred in 100 ml of neat DMSO overnight at room temperature. The mixture was then heated in a hot water bath and recooled to room temperature. The solution, which was decanted from the sodium hydroxide was assumed to be saturated. The literature indicates that the solubility of sodium hydroxide is 7×10^{-3}M in DMSO.

The studies were performed in a similar manner to these performed in 75 percent DMSO. Weighed amounts of explosives (12.5 mg TNT and 2,4-DNT, and 10.0 mg of RDX) were combined with 40 ml of saturated sodium hydroxide in DMSO and decontamination studies were performed at 25 and 65 C. One series of decontaminations were performed at 25 C after the saturated DMSO solution was diluted by a factor of 1 to 5. Samples (1.0 ml) were quenched with 100 ul 1.0M HCl and the quenched solutions were used directly for HPLC analysis. The results of HPLC analyses are shown in Table 11.

5.0 DISCUSSION OF THE CHEMICAL DECONTAMINATION RESULTS

Aqueous solvent systems which contain DMSO and DMF were recommended for use in Subtask 4 decontamination studies as a result of Subtask 3 studies on the solubilization of explosives. The minimum concentration of organic solvent which was estimated to give useful solubilities of all target explosives was 30 percent.

The initial decontamination studies focused on the decontamination of 2,4-DNT and TNT with sodium hydroxide. In these initial studies, emphasis was placed on determining the half-lifes of these reactions by studying these reactions in a kinetic fashion. The potential advantage of determining a reaction half-life for a well behaved reaction is that the time required to achieve any desired degree of decontamination may be readily calculated. These reactions were performed under pseudo first-order conditions in which the initial concentration of decontaminants was at least ten times the concentration of explosives so the concentration of decontaminants remained essentially constant during the course of these reactions. Therefore, a reaction which is first order in both decontaminant and explosive (second

TABLE 11. HPLC ANALYSIS OF 2,4-DNT, TNT AND RDX DECONTAMINATION WITH DMSO SATURATED WITh SODIUM HYDROXIDE

TEMPERATURE: 25 C

2,4-DNT			TNT			RDX		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	31282.5	0	0	24009.3	0	0	10337.0	0
61	19734.4	36.9	62	17999.1	25.0	58	BDL	
118	15092.4	51.8	120	16003.8	33.3	115	35.7	99.9
525	538.7	98.3	527	8379.1	65.1	522	BDL	
1042	244.8	99.2	1043	4730.7	80.3	1039	BDL	

TEMPERATURE: 25 C (1/5 Dilution)

2,4-DNT			TNT			RDX		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	18376.3	0	0	14402.0	0	0	5670.0	0
30	12418.4	32.4	30	13456.3	6.6	30	336.5	94.1
60	7658.5	58.3	60	12743.4	11.5	60	211.3	96.3
120	2203.1	88.0	120	11479.9	20.3	120	187.6	96.7
240	204.2	98.9	240	9316.9	35.3	240	146.8	97.4
405	100.3	99.5	405	7396.7	48.6	405	115.3	97.9
727	BDL		727	5063.7	64.8	725	83.9	98.5
1320	43.7	99.8	1320	2699.9	81.3	1320	64.4	98.9

TEMPERATURE: 65 C

2,4-DNT			TNT			RDX		
Time	Area	% Decon	Time	Area	% Decon	Time	Area	% Decon
0	31282.5	0	0	24009.3	0	0	10337.0	0
30	7653.8	75.5	31	1873.2	92.2	30	303.3	97.1
60	3350.1	89.3	60	781.9	96.7	60	BDL	-
120	286.7	99.1	120	738.1	96.9	120	65.4	99.4
183	4707.6	84.9	186	592.1	97.5	179	44.5	99.6
309	3006.7	90.4	312	195.9	99.2	305	3.9	99.9
667	1533.5	95.1	670	436.0	98.2	663	2.2	99.9
1323	134.3	99.6	1326	5068.8	78.9	1321	73.9	99.3

order overall) will reduce to a reaction which is zero order in the decontaminant and is first order in explosive and behave as a pseudo first order reaction. If a decontamination reaction is following pseudo first order kinetics, a plot of the natural logarithm of the explosives concentration versus reaction time will give a straight line. Pseudo first order rate constants were determined as the negative slopes of such plots and the half-lifes were calculated by dividing the natural log of 2 by the rate constants.

However, it was found in initial studies with sodium hydroxide that some reactions fit pseudo first order expressions very well but other reactions did not follow pseudo first order kinetics. Therefore, subsequent decontamination reactions were followed by determining the time/percent decomposition profile of a reaction. The decontamination reactions were evaluated in terms of the time required to reach high decontamination efficiencies (usually 98.9 percent or better).

5.1 Decontamination of Explosives With Sodium Hydroxide

5.1.1 Thirty Percent Aqueous DMSO and DMF

Kinetic data for those decontamination reactions with sodium hydroxide which were performed in thirty percent aqueous DMSO or DMF at 25 C and evaluated by kinetic analysis are shown in Table 12. Only 2,4-DNT and TNT were utilized in these early studies since RDX was not yet available. Sodium hydroxide concentrations of 0.10 and 0.0010M were employed and the catalytic effects of the cationic surfactants cetyl pyridinium bromide (CPBr) and N-cetyl-N-ethyl morpholinium ethosulfate (CEME) were studied. As mentioned earlier, these kinetic studies were performed under pseudo first-order conditions in which the initial concentration of sodium hydroxide was significantly in excess of the initial concentrations of explosives. The analytical data was entered into pseudo first order kinetic plots by employing Least Squares Analyses so correlation coefficients could be determined for each kinetic run. (A -1.00 correlation coefficient represents a perfect linear fit for all data points).

TABLE 12. KINETIC DATA FOR THE PSEUDO FIRST-ORDER DECOMPOSITION OF EXPLOSIVES

Explosive	Organic Solvent (30%)	Sodium Hydroxide Conc. (M)	Cationic Surfactant (.1%)	Kinetic Correlation Coefficient	Half-Life (min)	No. of Half-Lives Followed
2,4-DNT	DMSO	0.100	None	-0.99	126	1.5
2,4-DNT	DMSO	0.100	CPBr[a]	-0.99	9.2	5
2,4-DNT	DMSO	0.100	CEME	-0.99	5.50	5.5
2,4-DNT	DMF	0.100	None	No Decomposition		
2,4-DNT	DMF	0.100	CPBr	No Decomposition		
TNT	DMSO	0.001	None	-0.98[b]	107	1.2
TNT	DMSO	0.001	CPBr	-0.65[c]	--	--
TNT	DMSO	0.100	None[a]	-0.68[c]	--	--
TNT	DMSO	0.100	CPBr[a]	-0.47[d]	--	--
TNT	DMSO	0.100	CPBr[a]	-0.66[c]	--	--
TNT	DMSO	0.100	CEME[a]	-0.99[e]	6.42	2.5
TNT	DMF	0.100	None[a]	-0.99	14.4	2
TNT	DMF	0.100	CPBr[a]	-0.98	11.8	6

(a) A precipitate appeared after several days.
(b) The loss of TNT appeared to level out at approximately 30 minutes.
(c) The loss of TNT appeared to level out at approximately 24 minutes.
(d) The loss of TNT appeared to level out at approximately 8 minutes.
(e) Does not include time zero point.

It can be seen that the decontamination reactions of 2,4-DNT in DMSO follow pseudo first order kinetics as evidenced by correlation coefficients of -0.99. The reaction half-lifes are significantly dependent on the nature of the solvent as well as the presence of cationic surfactants. The decontamination of 2,4-DNT in DMSO with 0.10M sodium hydroxide was found to be accelerated by factors of 14 and 23 when 0.1 percent CPBr or CEME respectively were used as catalysts. However, in the case of CPBr, a precipitate formed after several days which could be a complex between the surfactant and either 2,4-DNT or its reaction products. This precipitate was not analyzed to determine its composition.

Aqueous DMF was omitted from future consideration when it was found that 2,4-DNT did not decompose at any measurable rate even when CPBr was used as a catalyst.

It can be seen that the decontamination of TNT with 0.001M sodium hydroxide follows pseudo first order kinetics (through 1.2 half-lifes) but non-first order kinetics were followed when CPBr was used and when 0.1M sodium hydroxide was employed both with and without CPBr. However, first order kinetics (through 2.5 half-lifes) was observed in the decontamination of TNT with 0.10M sodium hydroxide when CEME was used as the catalyst. In those reactions where first order kinetics were not followed, the loss of TNT appeared to level out within 30 minutes reaction time. Prec, tate formation was observed in each case where catalysts were employed in the decomposition of TNT with 0.10M sodium hydroxide. Even though both cationic surfactants showed significant catalytic activity, they were omitted from use in subsequent studies because of the possibility that both surfactants would initiate precipitation of TNT when 0.10M sodium hydroxide was employed.

These kinetic studies were followed through only 1-6 half-lifes. In those cases where non-linear pseudo first order kinetics were observed, the percent decompositions were typically low. Therefore, further studies with 0.10M sodium hydroxide were performed at 25 C for much longer time intervals to determine the reaction times which were required to reach high decontamination percentages. Selected data points for the decontamination reactions of TNT, 2,4-DNT, and RDX carried out for long time periods are shown in Table 13. It can be seen that RDX is by far the most reactive of these explosives since the concentration of remaining RDX at 120 minutes was below

TABLE 13. PERCENT DECONTAMINATION OF EXPLOSIVES AT SELECTED
TIME INTERVALS IN 30% AQUEOUS DMSO AT 25 C

Decontamination Concepts

Explosive	Five Percent Sodium Sulfide		0.25 Percent Sodium Sulfide		0.25 Percent Sodium Disulfide		0.1 M Sodium Hydroxide		DS2		Fenton's Reagent	
	Time (min)	Percent Decon.	Time (min)	Percent Decon.	Time (min)	Percent Decon.	Time (min)	Percent Decon.	Time (min)	Percent Decon.	Time (min)	Percent Decon.
TNT	180	89.1	12	85.0	10	99.3	241	94.1				
	325	95.2	70	98.3	3J	99.5	516	95.5	31	98.3	10	97.9
	1140	>99.94			1297	99.5	1629	96.2	974	98.8	1300	97.6
2,4-DNT	60	83.6	--	--	10	3.7	240	73.7	30	96.9	2	84.8
							516	96.7				
	390	>99.95	--	--	1180	4.5	1627	99.5	973	98.5	1515	85.2
RDX	10	99.66	--	--	--	--	10	94.7	10	99.9	2	57.9
	30	>99.93	--	--	--	--	120	>99.95	30	>99.99	1517	55.2
									120	99.9		

the detectable limit (greater than 99.97 percent decontamination) after a 120 minute reaction time. 2,4-DNT is next most reactive and TNT is the least reactive towards this reagent. The decontamination percents observed for these two explosives leveled off with time. The decontamination efficiency with TNT leveled out at approximately 94-96 percent between 241-1629 minutes but the decontamination efficiency ranged from 96.7-99.5 percent with 2,4-DNT between 516-1627 minutes. Therefore it appears that TNT is the least reactive of the three trial explosives under these conditions.

5.1.2 Seventy-Five Percent Aqueous DMSO

Studies were also performed in 75 percent aqueous DMSO with 0.10M sodium hydroxide to determine if the reactivies towards sodium hydroxide would be increased over the reactivities in 30 percent aqueous DMSO. Another objective of using a higher percentage of DMSO was to determine if this change would enhance the denitration process which was indicated to occur (through product analysis studies, Section 6) to a limited extent in thirty percent aqueous DMSO. The results of experiments which were performed at 25 C, 75 C and 147 C are shown in Table 14. It appears that the decontamination efficiency of TNT at 25 C is lower in 75 percent aqueous DMSO than it was in thirty percent aqueous DMSO. (See Tables 2 and 10). Conversely, the decontamination efficiency of 2,4-DNT at 25 C appears to be significantly enhanced in 75 percent aqueous DMSO than it was in thirty percent aqueous DMSO. Decontamination of RDX by sodium hydroxide was shown to be effective in both solvents and data points at earlier times would be needed to discriminate between 30 and 75 percent DMSO. As expected, the decontamination efficiencies increased when the reactions were performed at higher temperatures.

5.1.3 Saturated Sodium Hydroxide In Neat DMSO

The solubility of sodium hydroxide in neat DMSO is only 0.007 molar(0.03%) whereas the 0.1M sodium hydroxide which was used in thirty percent aqueous DMSO corresponds to 0.4 percent solutions. The results of decontamination experiments with saturated sodium hydroxide in

TABLE 14. PERCENT DECONTAMINATION OF EXPLOSIVES AT SELECTED
TIME INTERVALS BY 0.10 MOLAR SODIUM HYDROXIDE IN
75 PERCENT AQUEOUS DMSO

Explosive	Temperature (C)	Time (min)	Decontamination Effectiveness Percent Decontamination
TNT	25	300	77.5
		688	87.91
		1215	95.76
TNT	65	62	99.46
		119	99.76
		1045	99.65(a)
TNT	147	157	96.34
		307	>99.98
2,4-DNT	25	30	95.91
		300	99.73
		685	>99.98
2,4-DNT	65	59	>99.98
		364	99.73(a)
2,4-DNT	147	160	99.89
		300	99.94
RDX	25	30	99.92
		1209	99.91
RDX	65	56	99.89
		118	>99.97
RDX	147	159	99.72
		304	99.93

(a) Anomaly presumed to be due to formation of a product which had the same
retention time during HPLC analysis as the explosive.

DMSO at 25 C and 65 C are shown in Table 11. It can be seen that lower decontamination efficiencies were realized in this system compared to the decontamination systems which contain 0.10M sodium hydroxide in thirty and seventy-five percent aqueous DMSO. These results imply that the potential enhanced reactivity of sodium hydroxide in neat DMSO is more than neutralized by the 57 fold lower concentration of sodium hydroxide in neat DMSO.

5.2 Decontamination of Explosives
With Sodium Sulfide and Sodium Disulfide
In Thirty Percent DMSO at 25 C

A summary of the decontamination effectiveness of these two reducing agents at various concentrations is shown in Table 13. It is evident that 0.25 percent sodium disulfide was significantly more reactive than 0.25 percent sodium sulfide in decontaminating TNT (as was indicated in the literature). However, it can be seen that 0.25 percent sodium disulfide appears to be very ineffective in decontaminating 2,4-DNT. However, the possibility exists that a product peak has the same HPLC retention time as 2,4-DNT which would give the appearance of negligible decomposition. Nevertheless, sodium disulfide was dropped from further consideration because of its apparent lack of reactivity with 2,4-DNT. It can be seen that five percent sodium sulfide (as the nonahydrate) has general applicability toward TNT, 2,4-DNT, and RDX since all three explosives were reduced to concentrations below their detectable limits within reasonable periods of time (between 30-1140 minutes). The order of reactivity of these explosives appears to be RDX >> 2,4-DNT > TNT. It was also found that freshly prepared five percent sodium sulfide gave the same decontamination profile with TNT as two month old five percent sodium sulfide.

5.3 Decontamination of Explosives With Fenton's Reagent
In Thirty Percent DMSO at 25 C

A summary of the decontamination efficiencies of Fenton's Reagent towards the three target explosives is shown in Table 13. These reactions were characterized by effecting various levels of decontamination in very

short time intervals (2-10 minutes) which then did not increase with time. It can be seen that order of reactivity of explosives towards Fenton's Reagent is TNT > 2,4-DNT > RDX. These decontaminations were achieved by adding hydrogen peroxide to a solution of the explosive which also contained acidic ferrous sulfate. However, when hydrogen peroxide and ferrous sulfate were premixed and then added to a solution of TNT, negligible decontamination resulted. It was established that further decontamination of all three explosives could be effected by further sequential treatment of the spent explosives decontamination solutions with acidic ferrous sulfate followed by hydrogen peroxide (Table 8). These results indicate that successive and sequential spraying of a contaminated surface with ferrous sulfate followed by hydrogen peroxide could give rise to effective decontamination. It is also probable that greater levels of decontamination could be achieved by using increased concentration of reagents. However, a significant liability of Fenton's Reagent is that, unlike the use of bases and reductants, the explosives must be initially solubilized before the needed reagents are sequentially added.

5.4 Decontamination of Explosives With DS2

A summary of the decontamination efficiencies of DS2 at 25 C toward the three target explosives is shown in Table 13. It appears that DS2 had slightly greater initial reactivity than 0.1M sodium hydroxide at the same temperature but produced approximately equal percent decontamination after extended time intervals. DS2 contains diethylenetriamine and methyl cellosolve, both of which are considered toxic. Since the decontamination efficiency of DS2 does not appear to be markedly greater than sodium hydroxide in DS2, the further use of DS2 was dropped from consideration.

6.0 IDENTIFICATION OF DECONTAMINATION PRODUCTS

6.1 Literature Precedents For Decontamination Products

6.1.1 Sodium Hydroxide

Literature precedents exist for the reaction of nitroaromatics in general as well as the explosives TNT, tetryl and the DNT isomers with various

bases. One type of reactivity mode involves the acid-base reactions of nitroaromatics with these bases as well as the addition of these bases to the nitroaromatic systems. Another reactivity mode involves the displacement of nitro groups with hydroxide functionality as well as with solvent generated nucleophiles.

TNT is known to rapidly deprotonate in a variety of solvents to initially form the 2,4,6-trinitrobenzyl anion[1]. At high base concentrations, the addition of the basic species to the aromatic ring to form Meisenheimer complexes becomes an important process. When TNT is in excess over base, the subsequent reaction of 2,4,6-trinitrobenzyl anions with un-reacted TNT molecules to form Janovsky complexes is reported to become a dominant process[1,2]. Another type of reactivity involves the addition of up to three moles of potassium hydroxide to TNT in aqueous solvents to produce a tripotassium salt of TNT. The 2,4,6-trinitrobenzyl anion may also be involved in further reactions which do not involve ionic products. A precedent for one type of reactivity involves the reaction of nitrobenzene in basic DMSO solution (which also contains t-butyl alcohol) to give high yields of o- and p-nitrobenzoic acids[3]. This reaction involves the initial attack of the DMSO anion on nitrobenzene to give an adduct which eliminated CH_3SO^- to give o- and p-nitrotoluene. These isomeric nitrotoluenes were converted to the corresponding benzyl anions which were shown to undergo one-electron donation to nitrobenzene to give the nitrobenzene radical anion and the isomeric nitro-benzylic free radicals. The nitrobenzylic free radicals were postulated to then react with oxygen to give the isomeric nitrobenzoic acids.

In basic DMSO solution (in the presence of t-butyl alcohol), TNT was also shown to produce radicals and react with oxygen to presumably yield trinitrobenzoic acid[4] which probably involves the electron transfer from 2,4,6-trinitrobenzyl anion as described above. The same study showed that p-nitrotoluene dimerized to p,p'-dinitrodibenzyl presumably thorough a p-nitro-benzylanion intermediate. Dimerization of TNT to give dibenzyl derivatives also has been reported under basic conditions[5] which presumably involves the intermediary of the 2,4,6-trinitrobenzyl anion. Dibenzyl and stilbene derivatives have also been reported to be formed from the reaction of TNT and aqueous alkalai metal hydroxides[6]. When TNT was treated with barium

hydroxide, a product was isolated which was assumed to have a tetrameric salt structure which involves aryl, aryl bonding[7]. The reaction of TNT with aqueous sodium carbonate has also been reported to form complex polymeric materials.

Another type of reactivity of TNT involves displacement of nitro groups by various nucleophiles. The reaction of TNT and potassium hydroxide resulted in the detection of nitrite ion[8] which implies that nitro group are being displaced by hydroxide ions. Trinitrophenol and 3,5-dinitrophenol have been reported to be formed from the reaction of TNT and aqueous sodium hydroxide by hydroxide displacement reactions[9]. However, these structural assignments are suspect because this relied primarily on comparisons of paper chromatography R_f values. However, another study which was based on a spectroscopic investigation, indicated that a nitro group may be replaced by a hydroxy function in the reaction of p-dinitrobenzene with sodium hydroxide in aqueous DMSO at room temperature[10]. These results indicate that the displacement of nitro groups by hydroxide or other reactive nucleophiles (such as the DMSO anion) is a feasible method to decontaminate nitroaromatic explosives even though dimerization reactions may occur. The above precedents indicate that if the DMSO anion were to successfully displace nitro groups, the resulting adducts would then be transformed into methyl groups which could then readily be oxidized to carboxylic acid functions under basic reaction conditions in the presence of oxygen.

The reaction of RDX and HMX with aqueous base is reported to lead to nearly complete decomposition to gaseous and inorganic products[11,12].

The initial product resulting from the reaction of tetryl and hydroxide ion is picric acid which results from the displacement of the nitramine group by hydroxide ion[13].

6.1.2 Sodium Sulfide

The reduction of TNT with sulfide ion is reported to give a variety of compounds in which variable numbers of nitro groups have been reduced. 4-amino-2,6-dinitrotoluene has been made by reacting TNT with ammonium

sulfide in dioxane[14] Reduction of TNT with ethanolic ammonium sulfide yields 2,4-diamino-6-nitrotoluene[15]. Hydroxylamines have also been isolated in the reduction of nitroaromatic compounds in several instances as seen in the conversion of dimethyl 5-nitroisophthalate to dimethyl 5-(hydroxyamino)isophthalate[16].

The products resulting from the reduction of RDX have not to our knowledge been determined in previous studies, but were assumed to be hydrazine derivatives.

6.1.3 Fenton's Reagent

Nitroaromatic and nitramine explosives have been reported to be decomposed to gaseous products by hydroxyl and methyl radicals which have been generated from the photochemically induced decomposition of hydrogen peroxide and acetone respectively[17]. Fenton's Reagent was evaluated in these decontamination studies because hydroxyl and methyl radicals were reported to be chemically generated by this reagent. However, the reaction of certain nitro-aromatics with Fenton's Reagent has been shown to lead to the introduction of hydroxyl functions on the aromatic ring[18]. The formation of dibenzyl derivatives has also been reported in the reaction of certain toluene derivatives with Fenton's Reagent.

6.2 Analytical Methodology

Gas chromatography/mass spectroscopy (GC/MS) was employed for the preliminary analysis of the mixtures resulting from all decontamination reactions. Since electron-impact mass spectral detection of explosives usually result in an abundance of spectra with low molecular ions (except for nitroaromatics), chemical ionization was used in the mass spectral detection system. This approach readily allows the determination of the molecular weights of the components separated by GC. However, when chemical ionization is employed, degradation ions are generally formed in low relative abundances which precludes detailed structural studies of these components. Nevertheless, the determination of product molecular weights limits the variation

in functionality which may be present in decomposition products and allows tentative structural assignments. Both methane and ammonia were used as reagent gases for the chemical ionization detection system. Methane was found to generally be more responsive than ammonia in detecting the variety of products which were produced in the decontamination reactions. When methane was used as the reagent gas, the molecular weight of the parent molecules (M) was determined by the presence of characteristic M+1, M+29, and M+41 peaks. Ammonia was also used as a reagent gas because this reagent gas has been indicated to replace nitro groups with amino functions which results in characteristic M-29 peaks. The determination of residual nitro groups in various products was useful in assigning tentative structures of various products. When ammonia was used as a reagent gas, the molecular weights of the parent molecules was determined by the presence of characteristic M+1 and M+18 peaks.

The quenched decontamination solutions were extracted with tert-butyl methyl ether. The sodium hydroxide and Fenton's Reagent initiated decontamination solutions were adjusted to approximately pH 1 prior to extraction. Quenched sodium sulfide solutions were adjusted to approximately pH 13 prior to extraction.

Method development studies led to the preferred use of a capillary column packed with SE-52 for the GC/MS studies. Three standards were employed with this column to ascertain that their chromatographic behavior and retention times were appropriate and that they could be readily detected when methane was used as the reagent gas. These standards were 2,6-dinitrocresol, 4-amino-2,6-dinitrotoluene, and 2,4-diaminotoluene.

6.3 Decontamination Products From TNT

The tentative structural asignments of products resulting from the decontamination of TNT with sodium hydroxide, sodium sulfide, and Fenton's Reagent are shown in Table 15.

6.3.1 Sodium Hydroxide

The major type of postulated products derived from TNT result from the displacement of nitro groups by hydroxide or DMSO anions, the oxidation of

TABLE 15. GC/MS PRODUCT ANALYSIS FROM TNT DECONTAMINATIONS
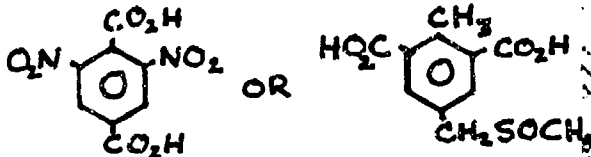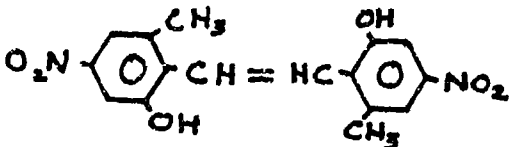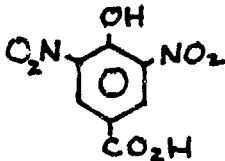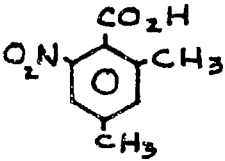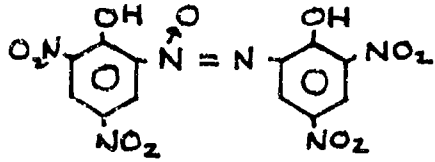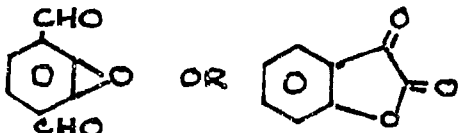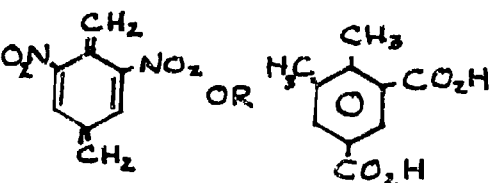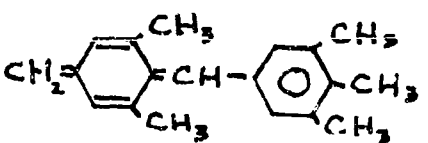
Reactant/Solvent Systems	Molecular Weights	Relative Amounts	Tentative Structural Assignments (Or Structural Isomers)
A. 0.1M Sodium Hydroxide in 30% DMSO (25°C)	148	Minor	Phthalic anhydride
	256	Minor	

	278	Major	Dibutyl phthalate
	330	Minor	

0.1M Sodium Hydroxide in 75% DMSO (65°C)	148	Minor	Phthalic anhydride
	228	Minor	

	254	Minor	Benzene tetracarboxylic acid
	256	Major	As above
	264		Unknown
	278	Medium	Dibutyl phthalate
	312	Minor	Phthalate derivative
Saturated Sodium Hydroxide in 100% DMSO (65°C)	116	Minor	Unknown
	211	Minor	Unknown
	195	Major	

	256	Medium	As above
	278	Minor	Dibutyl phthalate

TABLE 15. (continued)

Reactant/Solvent Systems	Molecular Weights	Relative Amounts	Tentative Structural Assignments (Or Structural Isomers)
	312	Minor	Phthalate derivative
	350,364	Minor	Unknown (note homolgous series)
	378,392		
	406,420		
B. 5% Sodium Sulfide in 30% DMSO (25°C)	167	Major	2,4-diamino-6-nitrotoluene
	174	Minor	Unknown
	278	Minor	Dibutyl phthalate
	283	Minor	Unknown
	337	Medium	Presumably a reduced dimer of TNT
	410	Minor	

C. Fenton's Reagent in 30% DMSO (25°)	148	Major	
	166	Medium	Unknown
	180	Minor	Unknown
	194	Medium	

| | 250 | Medium | |

methyl groups to carboxylate groups, and the dimerization (and subsequent oxidation) to stilbene derivatives. As indicated earlier, these processes have precedents in the literature. The attack by DMSO anions is postulated to give rise to methyl groups by subsequent cleavage of the initially formed adduct by hydroxide ion. It was assumed that carboxylic acids would not be retained by the SE-52 packed GC column so that their structures may be validly assigned. However, this assumption was not validated by determining if known carboxylic acids could be detected by the GC/MS system.

One reason for investigating the decontamination effectiveness of sodium hydroxide in increased percents of DMSO and at increased temperatures was to determine if a greater amount of nitro-group displacement occurred at these modified conditions. However, based on the limited structural data available, an increased degree of nitro-group displacement is not evident when these modified conditions were employed. A compound with a molecular weight of 256 (a dicarboxylic acid derivative) which was the minor product in 0.1M sodium hydroxide in 30% DMSO at 25 C, became the major component in 75% and neat DMSO at 65 C.

6.3.2 Sodium Sulfide

The reduction of TNT leads to one major product which is presumably 2,4-diamino-6-nitrotoluene. This specific diamino isomer is postulated because the 4-nitro group of TNT is usually reduced more readily than the 2- (or 6-) nitro groups. Presumably minor amounts of reduced dimers of TNT as well as a specific azoxy derivative (shown) are also formed during reduction with sodium sulfide.

6.3.3 Fenton's Reagent

A major component observed in the reaction with TNT has the same molecular weight (148) as phthalic anhydride. However, in a preliminary experiment the retention time of authentic phthalic anhydride was shown not to correspond to the retention time of this 148-molecular-weight species. (The retention time comparisons need to be repeated for verification purposes.)

The postulated structures for this component may be plausibly derived from the reactive methyl and hydroxyl radicals which are generated in Fenton's Reagent. The other higher molecular weight structures which are postulated have varying degrees of nitro-group substitution. The predicted phenolic compounds were not detected.

6.4 Decontamination Products From 2,4-DNT

The tentative structural assignments of products resulting from the decontamination of 2,4-DNT with sodium hydroxide, sodium sulfide, and Fenton's reagent are shown in Table 16.

6.4.1 Sodium Hydroxide

The same general type of products were observed in the decontamination of 2,4-DNT as were observed in the decontamination of TNT with sodium hydroxide. The postulated sulfenic acid derivative and cycloheptane derivative (which would originate from cyclization of a dibenzyl intermediate) were not observed when the reaction conditions were changed from 30% DMSO at 25 C to 75% and neat DMSO at 65 C. As can be seen from the tentative structures, it is difficult to postulate that the utilization of elevated temperatures and higher DMSO percentages lead to a greater degree of nitro-group substitution.

6.4.2 Sodium Sulfide

The major product observed is 2,4-diaminotoluene which results from the complete reduction of both nitro-groups. However, minor products which are produced correspond to the partial reduction of 2,4-DNT to hydroxylamine, nitro derivatives. Other products which are postulated incorporate functional groups which result from the introduction and oxidation of methyl groups which may readily occur as a result of the highly basic nature of the sodium sulfide solutions.

TABLE 16. GC/MS PRODUCT ANALYSIS FROM 2,4-DNT DECONTAMINATIONS
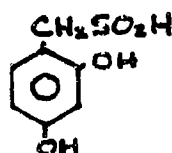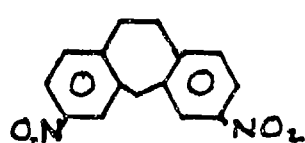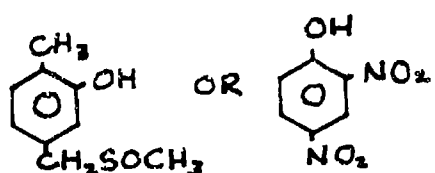
Reactant/Solvent Systems	Molecular Weights	Relative Amounts	Tentative Structural Assignments (Or Structural Isomers)
A. 0.1M Sodium Hydroxide in 30% DMSO (25°C)	188	Major	(Although Ammonia reagent gas gave M-29 peak)
	256	Minor	Phthalate derivative
	278	Minor	Dibutyl phthalate
	284	Minor	
	337	Minor	Unknown
0.1M Sodium Hydroxide in 75% DMSO (65° C)	101	Minor	Unknown
	148	Minor	Phthalic anhydride
	167	Major	Nitrobenzoic acids
	171	Major	Unknown has one nitrogen atom but may not have nitro group.
	184	Major	
	256	Minor	Unknown
	278	Minor	Dibutyl phthalate
	312	Minor	
	410	Medium	Presumably a dimer of DNT with an even number of nitrogen atoms
Saturated Sodium Hydroxide in 100%	101	Minor	Unknown
	167	Minor	Nitrobenzoic acid

TABLE 16. (continued)

Reactant/Solvent Systems	Molecular Weights	Relative Amounts	Tentative Structural Assignments (Or Structural Isomers)
DMSO (65°C)	184	Major	As above
	256	Minor	Dibutylphthalate
B. 5% Soduim Sulfide in 30% DMSO (25°C)	111	Minor	Unknown
	122	Major	2,4-diaminotoluene
	168	Minor	(structure: toluene ring with CH_3, $NHOH$, NO_2 substituents)
	182	Minor	Not 2,4-DNT (structure: benzene ring with CO_2H, NH_2, NO_2) OR (toluene ring with CH_3, CH_3, CH_2SOCH_3)
	312	Minor	
Fenton's Reagent in 30% DMSO (25°C)	111	Minor	Unknown (Also generated in sodium sulfide)
	182	Major	2,4-DNT
	194	Major	(structure: ring with NO_2, NO_2 substituents)
	195	Minor	Unknown
	278	Medium	Dibutylphthalate

6.4.3 Fenton's Reagent

The major product which is observed in the reaction with Fenton's Reagent is a dinitro styrene derivative which could plausibly be formed from 2,4-DNT in the presence of methyl radicals. However, this species would be expected to be readily polymerized in the presence of various radicals. Therefore, if this assignment is correct, some fraction of this compound may have undergone a radical-induced polymerization.

6.5 Decontamination Products From RDX

Tentative structural assignments of products resulting from the decontamination of RDX with sodium hydroxide, sodium sulfide, and Fenton's Reagent are shown in Table 17.

6.5.1 Sodium Hydroxide

The reaction of RDX with aqueous base is reported to produce formaldehyde, formate ion, dinitrogen oxide, ammonia and nitrogen.[11] The nature of this predicted product composition was verified when, upon acidification and extraction of the spent reaction mixtures, the GC traces were very noisy and component peaks were barely detectable. Usable GC/MS responses were obtained when the samples were concentrated and the GC/MS instrument was operated in the splitless mode. It is not possible to measure the amount of RDX which was converted to gaseous products without adding an inert reference compound to the decontamination solutions. However, based on the low GC/MS response, it appears that only a very small amount of RDX was not converted to gaseous products. HPLC analysis of the spent RDX decontamination solutions also indicates that a large percentage of the RDX decomposition products are mainly gaseous because these solutions have a low area count compared to the starting solutions. The only structures which were postulated for these residual products correspond to rearranged RDX isomers. These rearranged RDX isomers could readily be formed by the reaction of expelled nitrite ion and the unsaturated intermediate known to be formed in the initial reaction between RDX and base.[11]

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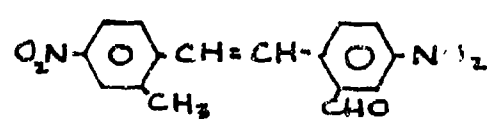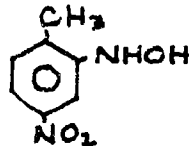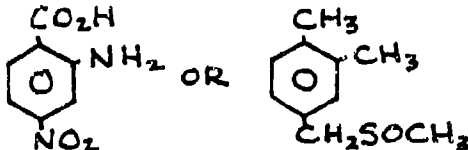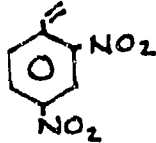TABLE 17. GC/MS PRODUCT ANALYSIS FROM RDX DECONTAMINATIONS
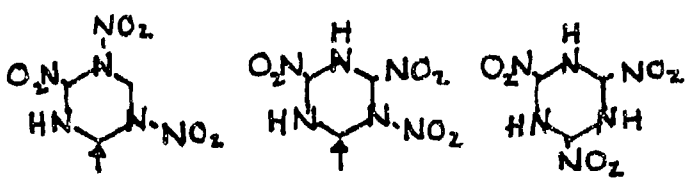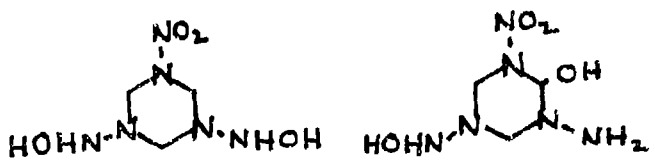
Reactant/Solvent Systems	Molecular Weights	Relative Amounts	Tentative Structural Assignments (Or Structural Isomers)
A. 0.1M Sodium Hydroxide in 30% DMSO (25°C)	--	-	GC response was so low that mass spectral scans were not recorded.
0.1M Sodium Hydroxide in 75% DMSO (65°C)	116	Minor	Unknown
	222	Medium	Structural isomers of RDX (5 isomers were observed).

plus isomers

	256	Major	Unknown
	278	Major	Dibutyl phthalate
	312	Medium	Unknown
	337	Minor	Unknown
	370	Minor	Unknown
Saturated Sodium Hydroxide in 100% DMSO (65°)	149	Minor	Unknown
	157	Minor	Unknown
	228	Minor	Unknown
	256	Major	Unknown
	283	Minor	Unknown
	278	Medium	Dibutyl phthalate
	326	Minor	Unknown
	410	Minor	Unknown
B. 5% Sodium Sulfide in 30% DMSO (25°C)	194	Major	(2 isomers were observed)

	278	Minor	Dibutyl phthalate

TABLE 17. (continued)

Reactant/Solvent Systems	Molecular Weights	Relative Amounts	Tentative Structural Assignments (Or Structural Isomers)
	312	Minor	Unknown
	337	Minor	Unknown
	410	Minor	Unknown
C. Fenton's Reagent in 30% DMSO (25°C)	194	Major	(Same retention time and spectrum observed with RDX in sodium sulfide reaction.)
	278	Minor	Dibutyl phthalate
	312	Minor	Unknown
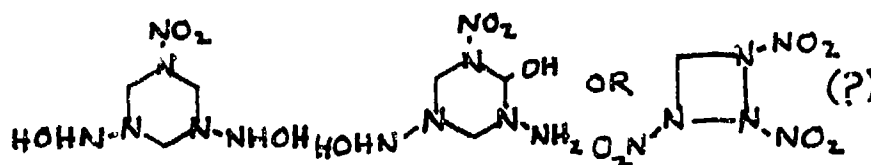
6.5.2 Sodium Sulfide

The major product formed from the reduction of RDX may either be a di-hydroxylamine or an amino, hydroxylamine, hydroxy derivative of RDX. The nature of the postulated structures of these products indicates that complete reduction of all three nitro groups does not occur.

6.5.3 Fenton's Reagent

The major product from the reaction of Fenton's Reagent with RDX appears to be the identical compound which was detected in the reduction of RDX with sodium sulfide (194 molecular weight). That being the case, it is difficult to postulate that the four-membered ring compound shown could be formed in decontamination reactions of RDX with both sodium sulfide and Fenton's Reagent.

7.0 EVALUATIONS AND COMPARISONS

The decontaminants which were ranked the highest in terms of decontamination efficiencies were sodium sulfide followed by the basic reagent sodium hydroxide in aqueous DMSO solutions. Sodium disulfide was dropped from consideration because of its apparent lack of reactivity with 2,4-DNT. DS2 was dropped from further consideration since it was assumed to be toxic and its decontamination effectiveness was approximately the same as sodium hydroxide in aqueous DMSO. Fenton's Reagent was dropped from further consideration because its mode of action requires that the explosive be previously solubilized before the ferrous sulfate and hydrogen peroxide are sequentially added. These requirements would not allow the decontamination of explosives located within a building material matrix.

The complete evaluation of these concepts must take into account the potential toxic effects of the decontamination products.

The structural assignments which are most definite in our GC/MS studies are derived from the sodium sulfide-based reduction of TNT and 2,4-DNT in which it was indicated that 2,4-diamino-6-nitrotoluene and 2,4-diaminotoluene, respectively, were almost exclusively formed. The structural

assignments of the reduction product from RDX are not as definitive but partially reduced hydroxylamine derivatives of RDX appear to have been formed. Nevertheless, it is difficult to recommend sodium sulfide (standing alone) as a candidate for structural decontamination (versus inerting) since aromatic amines which result from the nitroaromatic explosives have been implied to cause cancer in animals.

The tentative structural assignments of the decontamination products from the reaction of the nitroaromatic explosives with sodium hydroxide indicate that a significant degree of nitro group displacement has occurred. Both GC/MS and HPLC analysis indicate that a large percentage of decontamination products from the reaction of RDX with sodium hydroxide are gaseous products. At least some of the compounds remaining in solution are presumed to be rearranged RDX structural isomers.

It thus appears that the only decontamination reagent which provides effective decontamination of explosives both on the surface and within the matrix of building materials and also may yield products of acceptable toxicity is sodium hydroxide in aqueous DMSO. A logical extension of this base-initiated hydrolysis concept is to employ elevated temperatures which has been demonstrated to improve the decontamination efficiencies toward all explosives (which is needed in the case of TNT to effect a high decontamination efficiency). The limitations of our structural studies did not allow us to assess whether increased displacement of residual nitro groups occurred at higher temperatures. A practical approach which could be employed to treat contaminated surfaces at moderately elevated temperatures would be to employ polymeric thickening agents which would allow continued contact time for extended periods of time. Another extension would be to soak the contaminated building material with sodium hydroxide solutions prior to hot-gas treatment. This modification of direct thermolysis would probably allow much lower decontamination temperatures especially for HMX (and RDX) which has been found to require the highest temperatures for thermal decomposition in Subtask 5 studies.

However, it should be stressed that we recommend the use of sodium hydroxide towards nitro aromatic explosives with some reservations. One basis of this reservation is the fact that structural assignments based on the GC/MS analytical approach are speculative in nature and in no manner confirm

the structure of a compound. Furthermore, as indicated earlier, nitro-aromatics (and TNT in particular) have been reported to form a number of complexes as well as oligomers or polymers in the presence of base. The complexes may not dissociate (even at low pH values employed in the extraction process) and polymeric material would not volatilize so that these potential decontamination products would not be detected with GC/MS. One assumption is that tertiary butyl methyl ether which is used as an extractant will effectively extract the explosives products from aqueous DMSO solutions. Another potential analytical limitation is that the extracted decontamination products may not pass through the GC column under the conditions employed and/or these products may be undergoing thermal decomposition upon injection and passage through the GC column. The fact that, in certain cases, GC peaks with identical retention times and identical mass spectra were observed in the product mixtures from the decontamination of the same explosives with different decontaminants is consistent with this latter possibility. To illustrate, the reaction of 2,4-DNT with sodium sulfide and Fenton's Reagent produce the same 111 molecular weight component in both reaction mixtures which have identical retention times and mass spectra. Furthermore the same 256 molecular weight component (same retention time and mass spectra) was observed in the sodium hydroxide initiated decontamination of both TNT and RDX. This occurrence is difficult to understand unless some high temperature pyrolysis were to convert these distinctly different explosives into the identical compound.

An alternate analytical approach for the separation and isolation of products formed from the decontamination of explosives with sodium hydroxide would use HPLC for the initial separation of products. This separation method would not be prone to the potential recovery problems associated with extraction techniques and would not lead to potential thermal decomposition of products formed. Upon isolation of these product fractions, elemental analyses in combination with various spectral methods would be used for structural characterization and functional group analysis of the products. This analytical approach may lead to a far better determination of the exact nature of decomposition products than is possible with the direct GC/MS method currently being employed on the sodium hydroxide reaction products.

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APPENDIX A

TEST PLAN FOR PRESCREENING
OF CHEMICAL DECONTAMINATION CONCEPTS

TASK 4, SUBTASK 4

TEST PLAN FOR
PRESCREENING OF CHEMICAL DECONTAMINATION CONCEPTS

to

U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
CONTRACT NO. DAAK11-81-C-0101

by

Herman P. Benecke

1.0 INTRODUCTION AND OBJECTIVES

The purpose of this study is to provide a closely controlled
evaluation in solution of the three proposed chemical decontamination
concepts identified in Task 2 studies. These three concepts which will
be evaluated are:

- Sulfur-Based Reduction
- Base-Initiated Decomposition
- Radical-Initiated Decomposition.

The primary criterion for concept evaluation will be the
relative destruction efficiency of the explosives by the three concepts.
The destruction efficiency will be determined by monitoring the percent
explosive remaining at various time periods. Key reaction conditions
will be varied to help determine the optimum conditions required for
decomposing the target explosives. The products of the various decon-
tamination reactions will also be determined to help evaluate these con-
cepts in terms of the known toxicity of identified reaction products.
We prefer that the preliminary identification of the reaction products
be performed during this subtask rather than during Subtask 5 (on stain-
less steel) since the same solutions used to determine decontamination
efficiency also could be used for product determination. Later work
during Subtask 5 would verify the presence of the products of concern
formed on stainless steel.

As was the case in Subtask 3 concerning solubilization of explosives, determination of optimum conditions will be performed only with TNT, RDX, and 2,4-DNT. The applicability of these optimized reaction conditions will then be validated on tetryl, HMX, and 2,6-DNT. If conditions cannot be found for the effective decomposition of explosives by any one of the above concepts, an alternate replacement concept will be chosen with USATHAMA approval and its effectiveness will be determined in the same manner.

The principal analytical technique which will be used to follow the decomposition of explosives is reverse phase, high pressure liquid chromatography (HPLC). Preliminary product identification will be performed either with HPLC/MS (Mass Spectrometry) or GC (Gas Chromatography)/MS techniques.

2.0 TEST CONDITIONS AND PARAMETERS

2.1 Solvent Systems

Additives are currently being evaluated (Subtask 3) which are expected to enhance the solubility of explosives in aqueous solvent systems. Water is presumed to be a necessary component of the solubilization/decontamination solvent system to permit the appreciable solubilization of the various inorganic decontamination reagents. The chemical decontamination concepts will be evaluated in the aqueous-based solvent system(s) which appears to have the highest merit for all six target explosives.

2.2 General Methods for Evaluating Decontamination Concepts

All decontamination will be performed under pseudo first-order conditions wherein the decontaminant concentrations will be significantly greater than the concentrations of explosives employed. The reaction vessels containing the solutions will be thermostated at 25.0 C and analysis times will be chosen so that four concentration

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determinations of residual explosive will be performed within the 25 to 95 percent decomposition range. If linear plots of the natural logarithm of explosives concentrations versus time are achieved as expected, pseudo first-order kinetics and conditions will have been demonstrated. The slopes of these plots will equal the pseudo first-order rate constants (negative) whose magnitudes will allow a quantitative comparison of decomposition efficiencies. Half-lives may be directly derived from these rate constants and be used to specify the time needed for various levels of decontamination. If linear plots are not achieved, determinations of relative destruction efficiencies of each concept will be performed by comparing the percent of explosive which has been decomposed at common decomposition times.

Product compositions for each explosive and decontamination method will be determined only for those sets of conditions which give the highest rate of decomposition for any decontamination method.

2.3 Decontamination Reagents

2.3.1 Sulfur-Based Reductants

The two sulfur-containing reductants which will be screened are sodium sulfide and sodium disulfide. The literature indicates that sodium disulfide is much more reactive than sodium sulfide but it is not commercially available. However, sodium disulfide is reported to be readily synthesized from sodium sulfide. Both reductants will be evaluated at two basic pH values. The potential catalytic effect of the addition of a cationic surfactant (cetylpyridinium bromide) will be evaluated at the pH value which gave the most rapid rate of reaction for each reductant. The amounts of non-decomposed explosives remaining will be determined by withdrawing aliquots which will be quenched by cooling and acidification before HPLC analysis. Control reactions will be performed to ascertain that no further decomposition occurs after the reaction mixtures have been acidified.

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A summary of the proposed test conditions for TNT, RDX, and 2,4-DNT are found below in Table 1.

TABLE 1. VARIABLES MATRIX FOR TNT, RDX, AND
2,4-DNT WITH SULFUR-BASED REDUCTANTS

Test Conditions	Na_2S	Na_2S_2
Expls. Concentration	0.5%	0.5%
Reductant Concentration	10%	10%
pH	9,11	9,11
Surfactant Concentration	0.1%	0.1%

It can be seen that 6 reactions must be performed to evaluate both reductants towards each explosive so that 18 reactions must be performed to evaluate all three explosives. Validation of these conditions for the other three explosives will involve the best of the two pH values previously determined, followed by surfactant evaluation so a toatl of 12 more reactions will be performed. The total number of reactions (30) imply that approximately 120 analyses will be performed to monitor these reactions.

2.3.2 Sodium Hydroxide

The aqueous alkaline hydrolysis of explosives will be evaluated by subjecting explosives to high hydroxide ion concentrations. The potential catalytic effect of the addition of a cationic surfactant (cetylpyridinium bromide) will be evaluated at the best pH. The decontamination efficiency of DS2 (a basic agent decontamination system) will also be evaluated. The progress of these reactions will be followed by withdrawing aliquots which will be quenched by cooling and acidification

before HPLC analysis. Control reactions will be performed to ascertain that decomposition of explosives does not occur after quenching.

A summary of the proposed test conditions for TNT, RDX, and 2,4-DNT are found below in Table 2.

TABLE 2. VARIABLES FOR TNT, RDX, AND 2,4-DNT
WITH BASIC REAGENTS

Test Conditions	NaOH	DS2
Expls. Concentration	0.5%	0.5%
pH	11,13	--
Surfactant Concentration	0,1%	none

It can be seen that three reactions will be performed to evaluate sodium hydroxide and one reaction will be employed to test DS2 with each explosive, so that twelve reactions must be performed to evaluate the above three explosives. Validation of these conditions for the other three explosives will involve only the optiomum pH value (sodium hydroxide only) followed by surfactant evaluation (sodium hydroxide only) so a total of nine more reactions will be performed. The total number of reactions (21) imply that approximately 84 analyses will be performed to monitor these reactions.

2.3.3 Radical Sources (Fenton's Reagent)

Hydroxyl radicals will be chemically generated in situ and their effectiveness towards initiating the decomposition of explosives will be evaluated. The hydroxyl radicals will be generated from Fenton's reagent which is a mixture of ferrous ion and hydrogen peroxide maintained at low pH. The experimental technique will involve the

addition of an acidic solution of hydrogen peroxide to an acidic
solution containing the explosive and ferrous ion. The reaction will
primarily be evaluated in the presence of dimethyl sulfoxide (DMSO)
since hydroxyl radicals are known to react with DMSO to give methyl
radicals which have been reported to effectiveles initiate the decompo-
sition of explosives. Alcohols will not be used as a cosolvent since
hydroxyl radicals readily oxidize alcohols. The effect of a non-ionic
surfactant (Triton X-100) will also be investigated at the best
conditions.

It is not known with certainty at this time which of the
following conditions will effectively quench these reactions. Repre-
sentative reaction mixtures will be neutralized to neutral pH and
monitored to determine if the component composition undergoes further
change with time. If the reaction composition is found to change before
analyses typically can be performed, mild reducing agents such as
methanol will be added to reduce hydrogen peroxide to water.

A summary of the proposed test conditions are found below in
Table 3.

TABLE 3. VARIABLE MATRIX FOR TNT, RDX, AND
2,4-DNT WTIH FENTONS REAGENT

Test Conditions	
Expls. Concentration	0.5%
Ferrous Perchloriate	1%, 5%
Perchloric Acid	.1M
30% Hydrogen Peroxide	equal molar amounts as ferrous ion concentration
Surfactant	0%, 1%

It can be seen that three reactions will be performed to evaluate each explosive so nine reactions must be performed to evaluate the above three explosives. Validation of these conditions for the other three explosives will require six additional reactions so a total number of 15 reactions must be performed which implies that approximately 60 analyses will be performed to monitor these reactions.

3.0 SCHEDULE OF TESTS

The estimated total number of reactions which will be performed to evaluate all concepts is 66. The corresponding number of HPLC analyses may be estimated to be 264. The analyses will probably limit the rate of the data output rate. The times needed for method development are predicted to be small.

The screening studies for the three concepts will be performed in a staggered and overlapping sequence. Use of a second HPLC instrument will expedite analyses and allow the tests to be completed in the 6-week time span estimated in the Task 4 Work and Resource Plan. The startup date will be approximately November 28, 1983 if RDX and HMX were to arrive soon. The interval between the completion of the solubilization studies and the initiation of this subtask would allow conclusions to be drawn concerning the selection of solubilizing agents for these decontamination studies. These results would be communicated verbally to the COTR for approval. We would anticipate that the results of these chemical decontamination screening tests would be completed and reported to you by January 16, 1984. The experimental plan schedule is shown in Table 4.

4.0 CONTINGENCY PLANS

Various contingencies have been anticipated for different aspects of these decontamination studies which are described below. One

common factor of our experimental approach is that all reactions will be
initially performed at 25 C. However, if reactions proceed too slowly
to be followed effectively at these temperatures, then these reactions
will be studied at a higher common temperature. Another common factor
is that cetylpyridium bromide is to be investigated as a micellar
catalyst. If this compound does not effectively function as a catalyst,
then other selected cationic catalysts will be substituted.

TABLE 4. SCHEDULE OF EXPERIMENTS

Week 1	Week 2	Week 3	Week 4	Week 5	Week 6
Reductants	Reductants	Reductants	Reductants		
		Hydroxide, DS2	Hydroxide, DS2	Hydroxide, DS2	
				Radicals	Radicals

DEPARTMENT OF THE ARMY

US ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY

ABERDEEN PROVING GROUND, MARYLAND 21010-5401

REPLY TO
ATTENTION OF

August 24, 1984

Technology Division

Dr. Eugene J. Mezey
Battelle
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Dear Dr. Mezey:

The test reports entitled "Enhanced Aqueous Solubilization of Explosives" and "Prescreening of Chemical Decontamination Concepts" have been reviewed and the Agency's comments are enclosed. These comments should be addressed in writing and incorporated into the final report where appropriate.

Sincerely,

Andrew P. Roach
Contracting Officer's
Technical Representative

Enclosures

Comments on Test Report Entitled
"Prescreening of Chemical Decontamination Concepts"

Item	Page	Comment
1	2, Sec 2.0	The toxicity of the decomposition products is a consideration in the selection of a treatment concept. It cannot be called a criterion. This paragraph leads the reader to expect a more detailed evaluation of the toxicity of the decomposition products. It should be clearly stated that the scope of this study includes identification of major decomposition products and an assessment of their toxicity based on data available in the literature.
2	16, Sec 4.5	Was solubility at 25 percent verified by cloud point measurement?
3	23, Sec 5.1.1.	Was the CPBr precipitate observed during the Enhanced Solubilization Study? If it is an insoluble decomposition product it should be identified by GC/MS.
4	25, Sec 5.1.1.	Para 1, second sentence: Delete "...which was observed..." so that the sentence reads "...the percent decompositions were typically low."
5	25, Sec 5.1.2.	If denitration is only occurring to a limited extent, what are the primary decomposition products in the 30 percent DMSO solution?
6	28	Line 4: It should be clarified that both 30 percent and 75 percent DMSO were effective in decomposing RDX and that additional data at times below some value (perhaps 30 min) would be required to discriminate between the solvent systems.
7	45	Line 1: The sentence beginning "We are not aware..." is misleading. It would be appropriate to include a general but brief discussion of the relative toxicity of the various substituted aromatics to indicate that this concern was addressed.

February 15, 1985

Mr. Andrew P. Roach
U.S. Army Toxic and Hazardous
    Materials Agency
Building E-4585
Attn: DRXTH-TE-D
Aberdeen Proving Ground (EA)
    Maryland 21010

Dear Andy:

<div align="center">
Contract No. DAAK11-81-C09101
Novel Decontamination Technology
Task 4 Development of Techniques for
Explosive-Contaminated Facilities
</div>

This letter addresses USATHAMA's comments on the test report Task 4, Sub-task 4 entitled "Prescreening of Chemical Decontamination Concepts" dated August 24, 1984 USATHAMA's comments are attached for reference. This letter is to be attached to the test report and covers the explanation or actions taken in response to the Army's comments. The comments or action, addressed by item number, are as follows:

Item 1   The collection of literature concerning the toxicities of the major decontamination products was beyond the scope of this project. Estimates of toxicities of classes of compounds typical of the decomposition products identified were based on our general awareness of the toxicities of those class of compounds (e.g., aromatic amines).

Item 2   The solubility of sodium hydroxide in aqueous DMSO was determined by visual determination of solubility and was not verified by cloud point measurement.

Item 3   The CPBr precipitate was not observed in the Enhanced Solubility Study but that study utilized 100 percent water as opposed to 30 percent aqueous DMSO or DMF which was used in these studies. Since these precipitates were presumed to have incorporated this sur-factant they were judged to be poor candidates for study by GC/MS (i.e., low volatility and easily thermally decomposed).

<div align="center">225</div>

Item 4 Correction made.

Item 5 The postulated products of this reaction are shown in Tables 15, 16, and 17.

Item 6 Appropriate additions were made to the text.

Item 7 This sentence has been omitted. Please see the response to Item 1 concerning the potential toxicities of identified decontamination products.

Should you have any questions on these responses, please call me at (614) 424-4995 or Dr. Herman Benecke at (614) 424-4457.

Sincerely,

Eugene J. Mezey
Program Manager

EJM/sj

Attachment

APPENDIX III-3

SUBTASK 5 - STAINLESS STEEL SURFACE DECONTAMINATION BY
HOT GASES - TEST REPORT

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TEST REPORT

for

TASK 4, SUBTASK 5
STAINLESS STEEL SURFACE
DECONTAMINATION BY HOT GASES

Contract No. DAAK11-81-C-0101

to

UNITED STATES ARMY
TOXIC & HAZARDOUS MATERIALS AGENCY

August 3, 1984

by

D.R. Hopper, A.J. Killmeyer
and
E.J. Mezey

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

SUMMARY

The concept of using hot gases to decontaminate stainless steel surfaces was evaluated for its effectiveness in decomposing/removing explosives contamination from stainless steel surfaces. Tests were conducted with 5-inch square stainless steel coupons spiked with 400 mg of one of six explosives (TNT; 2,4 DNT, 2,6 DNT, Tetryl, RDX, or HMX). The results of these tests have indicated that the removal of explosives from stainless steel by this concept occurs through a complex combination of vaporization and decomposition. While the less volatile explosives (HMX and RDX) are the most difficult to remove, sufficiently mild conditions (500 F for 1 hour) were shown to effect 99.97 percent removal of any of the six explosives. Consequently BCL recommends that the effectiveness of this concept to decontaminate more porous matricies such as concrete or painted surfaces be evaluated in subsequent subtasks.

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FOR

TASK 4, SUBTASK 5

STAINLESS STEEL SURFACE

DECONTAMINATION BY HOT GASES

Contract No. DAAK11-81-C-0101

to

UNITED STATES ARMY

TOXIC AND HAZARDOUS MATERIALS AGENCY

August 3, 1984

1.0 INTRODUCTION

Facilities previously used by the United States Army for th manufacture or loading of explosives must be decontaminated or inerted before the facilities can be restored for alternate use or safely excessed. The development of novel concepts for the decontamination of such facilities is being conducted by Battelle Columbus Laboratories (BCL) for the United States Army Toxic and Hazardous Materials Agency (USATHAMA) under Contract No. DAAK11-81-C-0101. In Task 2 of this contract,[1] ideas were systematically developed into concepts for the decontamination of buildings. These concepts were also evaluated and ranked with respect to selected technical and economic factors. Five concepts were selected for further evaluation and knowlege gaps pertaining to the implementation of these concepts were identified. Task 4 was then established to permit laboratory resolution of these knowledge gaps.[2] Subtask 5 is the third of four experimental subtasks established in Task 4. The test plan for this subtask describing the experimental effort that was planned is included with this report as Appendix A.

2.0 PURPOSE AND OBJECTIVES

The designed purpose of this subtask was to provide a preliminary evaluation of the five concepts that had been selected as the most promising

during previous experimental subtasks. The concepts scheduled for evaluation in this subtask included:

- Hot Gases
- Vapor/Solvent Circulation (The solvent to be selected from Subtask 3)
- Three Chemical Concepts (Selected from the results of Subtask 4).

These five concepts were to be evaluated for their ability to decompose or remove each of six explosives (TNT, 2,4 DNT, 2,6 DNT, Tetryl, RDX, and HMX) from stainless steel coupons. The objective of this task was to rank order the tested concepts and select a maximum of three for subsequent evaluation in Subtask 6. However, due to delays in the availability of data from Subtasks 3 and 4 as well as the desire to provide an economy of effort in proceeding with Subtasks 5 and 6, evaluation of the vapor/solvent as well as the chemical concepts was deferred to Subtask 6. Consequently, the objectives of Subtask 5 were modified and only the Hot Gases Concept was evaluated.

3.0 FACILITIES, MATERIALS, AND EQUIPMENT

Due to the hazardous nature of the explosives and the desire to provide test conditions closely resembling field conditions, special test equipment was designed and fabricated.[3] This equipment was then installed in a laboratory equipped for conducting experimental work with hazardous and toxic materials. As shown in Figure 1, the test equipment consists of the gas delivery, test chamber and effluent handling systems that are described in more detail below. For safety purposes and to assure custody, stock solutions containing less than 5 grams of explosives were prepared for laboratory use. The storage and handling of the bulk explosives was performed by trained personnel in accordance with procedures described in BCL's custodial plan.[4]

GAS
DELIVERY
SYSTEM

TEST
CHAMBER

EFFLUENT
SAMPLING
SYSTEM
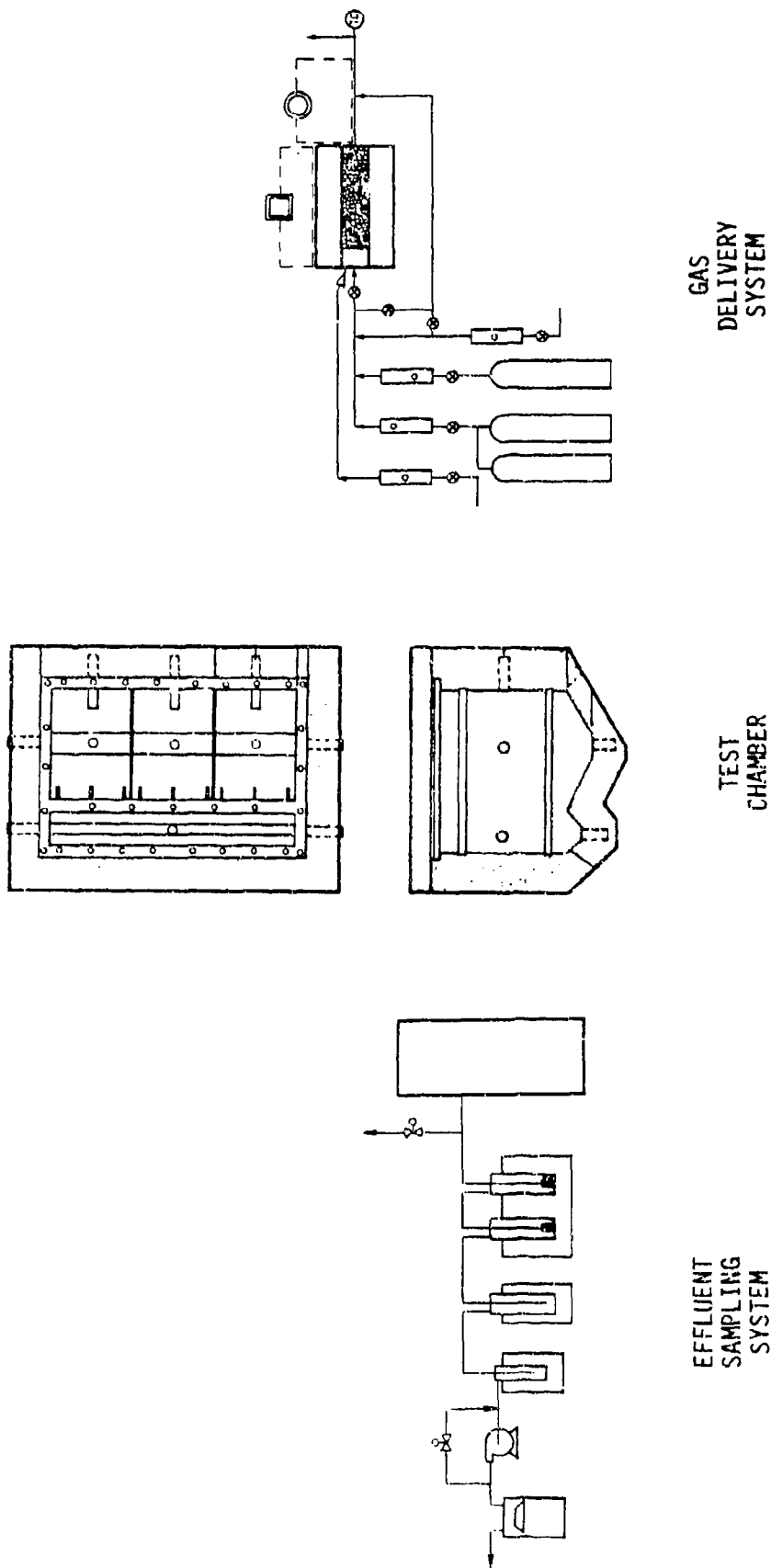
FIGURE 1. OVERALL EQUIPMENT LAYOUT

3.1 Explosives

Six conventional high explosives, 2,4, DNT, 2,6 DNT, TNT, Tetryl, RDX, and HMX were used during these tests. Both 2,4 DNT and 2,6 DNT were received from Aldrich Chemical Company. The 2,4 DNT was broken down to a smaller and uniform particle size by rolling the material with a wooden dowel. The 2,6 DNT was used as received. The TNT and tetryl were munition grade explosives obtained through an internal transfer from other Battelle projects. The RDX (Batch No. 4RC 14-71) and HMX (Batch No. 6ABCH 14-1) were received from Holston AAP. These materials were shipped wetted with isopropyl alcohol. This alcohol was removed prior to use by placing the explosives in a desicator and evacuating at high vacuum for one day.

3.2 Test Chamber

As shown in Figure 2, the test chamber consisted of a two compartment box of roughly rectangular geometry. This chamber was constructed of 16 gauge stainless steel sheet and supported, reinforced with 1/4 x 1 1/2 x 1½ inch angle iron and sized to fit within a laboratory hood. All seams were welded and the removable top sealed with a gasket. To minimize heat losses, the entire chamber was covered with a 4 inch thick layer of Cera* wool insulation. Brief descriptions of the three major components of the test chamber (i.e., exposure chamber, collection chamber, and coupon holder) are given below.

3.2.1 Exposure Chamber

This 2.5 cubic foot chamber served to contain the hot gas atmosphere to which the sample coupons were exposed. Access to this chamber as well as the collection chamber was provided by the removable top. This top was sealed with a gasket made from 1/4 inch diameter Gortex* rope. This material remains pliable and is thermally stable at temperatures up to 370 C.

The bottom of the chamber was formed into three sumps which collected any condensate as well as solutions used to wash down the chamber
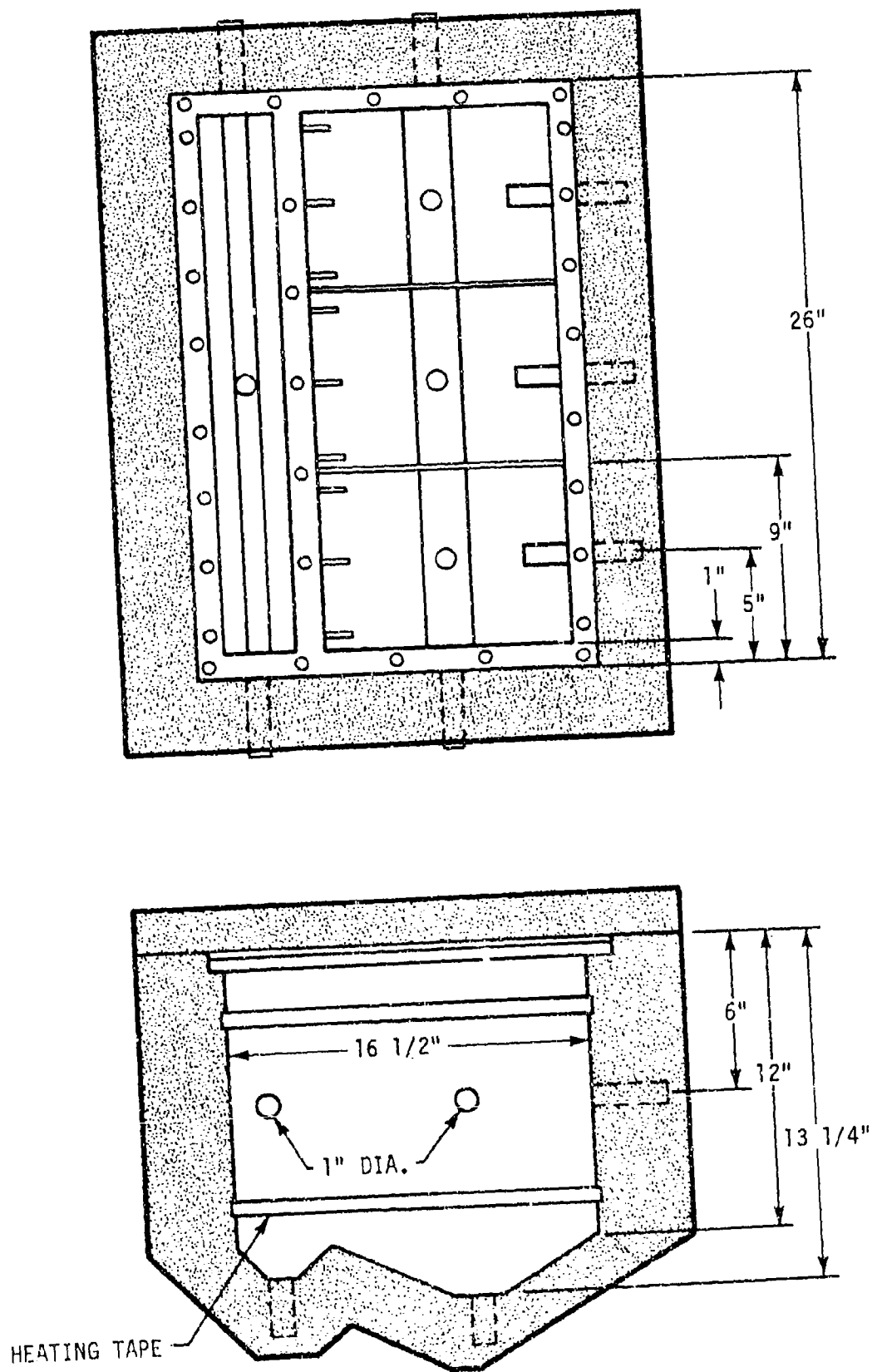
FIGURE 2. TEST CHAMBER

after the test was completed and the chamber cooled. These three sumps were connected to a common 1/4 inch stainless steel drain line from which the samples were collected for analysis.

The heated gases which provided the hot gas environment entered the chamber through a one inch pipe nipple located on the center of one side of the chamber and exited through a similar nipple on the opposite side. A deflector, placed within the inlet gas stream, provided good mixing of the hot gases and hence good temperature distribution within the exposure chamber. The exit line passed through a gate valve. During tests this valve was kept partially closed to maintain a small positive pressure (~2 inches of water) within the chamber. This assured that no uncontrolled diluting gases could leak into the chamber. The metered hot gas inlet stream could therefore be considered to represent the entire gas volume flowing into and out of the chamber. A metered sample taken upstream of the exit valve was assumed to be representative of the entire gaseous effluent.

The rear wall of the exposure chamber contained three 6-inch square openings evenly spaced on 8 inch centers. Eight bolts, welded to this wall around each opening functioned to attach the coupon holders. In this manner, the coupons covered the openings effectively separating the exposure and collection chambers during the tests. To provide rigidity to support the weight of the coupon and holders and not buckle under the thermal stresses, this wall was fabricated from 1/4-inch thick stainless steel.

3.2.2 Collection Chamber

This chamber was designed to collect liquids and gases that permeated through the test coupons. Since the stainless steel coupons were considered impermeable, in these tests the collection chamber only served to provide access for thermocouples used to measure the coupon temperatures.

3.2.3 Coupon Holders

The purpose of the sample holders was to hold the sample coupons being decontaminated in such a manner so one verticle plane surface is exposed

to the decontamination environment. In order to do this task while simultaneously sealing each of the 6-inch openings between the exposure and collection chambers three two piece holders identical to that represented in Figure 3 were constructed. These holders were designed to bolt to the rear chamber wall while holding a 5-inch square sample coupon between the back up plate and the retaining plate. Centered in both plates were 4-inch circular openings that regulated the area of the coupon that would be exposed to the atmosphere of both chambers. The sharp edge design of these openings prevented the buildup of liquids on the coupon surface.

The back up plate was constructed from a 7-inch square, 1/4 inch thick plate. Eight holes on the edges of this plate when aligned with the bolts welded to the back chamber wall served to attach the holder to the chamber. A flat Gortex® gasket placed between the wall and the plate provided a leakproof seal when the holder was in place. The backup plate also contained a groove for a Gortex O-ring gasket to provide a seal between the plate and the coupon. Four 1/4 x 2 inch bolts were welded to this plate to permit attachment of the retaining plate.

The retaining plate was constructed from a 5½ x 3/32 inch thick plate. Four holes, one in each corner, aligned with the 1/4-inch bolts attached to the back up plate. Tightening these bolts serves to hold the coupon in place while compressing the O-ring gasket and sealing the chamber.

3.2.4 Thermocouples

To provide measurement of the temperatures of interest, Chromel/ Alumel thermocouples were located at strategic sites within the chamber. The exposure chamber was monitored by a thermocouple inserted through the pipe in the center section of the front wall of the chamber. The collection chamber temperature was monitored by a thermocouple similarly inserted into the side wall port of this part of the chamber. In addition, thermocouple leads entered this port for thermocouple that were attached to the rear of the test coupons. These thermocouples provided monitoring of the critical coupon temperature parameter.

FIGURE 3. COUPON HOLDER

BEVELED EDGE

RETAINING PLATE

COUPON

BEVELED EDGE

O-RING

6" SQUARE OPENING WITH PLASTIC SEAL

FRONT PLATE

CHAMBER WALL
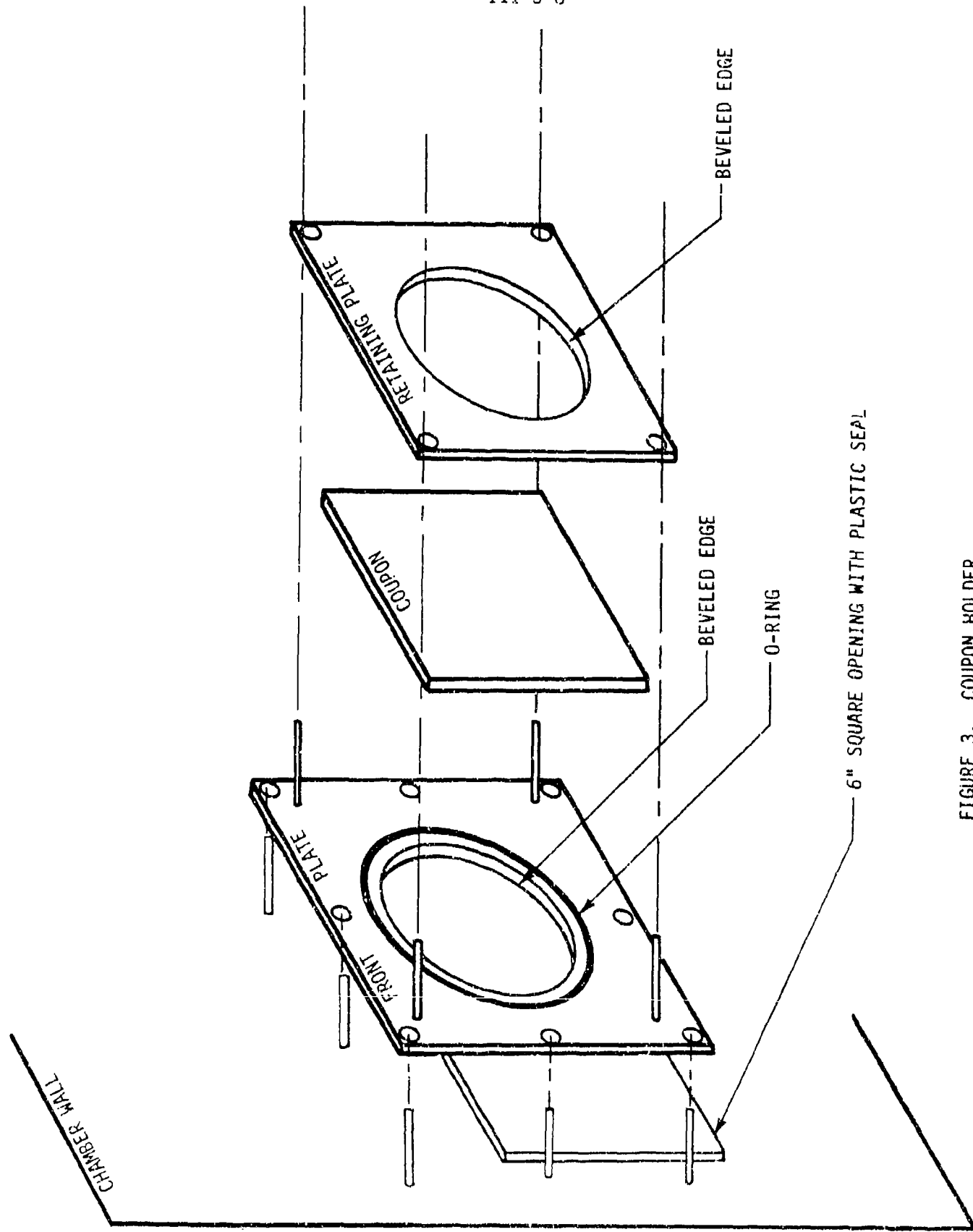
3.3 Gas Delivery System

In field application of the hot gas concept, the hot gases will be produced through the combustion of natural gas in air. In a worst case scenario, complete combustion of a stoichiometric mix of natural gas and air produces an atmosphere containing 71 percent nitrogen, 10 percent carbon dioxide and 19 percent water vapor. Allowing for normal heat losses, such an atmosphere would most likely enter the building at a temperature of approximately 800-820 C. The gas flow rate would be sufficient to deliver enough sensible heat to the building to achieve the desired temperature (~300 C) in less than 3 hours.

The gas delivery was designed to supply a comparable atmosphere to the test chamber. As can be seen in Figure 4, this system consisted of gas supplies, flow metering hardware, a gas heater and a heated chamber inlet pipe.

The gas supply consisted of bottled nitrogen and carbon monoxide and air from the laboratory compressed air supply. The nitrogen, the major constituent of the chamber atmosphere, was passed through a charcoal filter prior to use to remove any trace organics. The city water service in the laboratory was used to supply water in a liquid state to the system where it was vaporized in the furnace.

A constant and measured flow of 56.6 liters/min (2 scfm) of these gases was assured through the use of Dryer Rate Master Flowmeters. The control valves on these meters were adjusted to provide 40.1 liters/min of N_2 gas and 9.4 liters/min of CO_2 gas to a common manifold. This manifold was attached to the gas heater inlet. The water flow rate was adjusted to supply 8 ml/min of liquid water which was then introduced into the gas heater via a 0.02 inch ID nozzle.

The water was vaporized and the resulting gas mix was brought to the desired temperature by passing it over a bed of alumina chips that was contained in a 1½ inch diameter stainless steel pipe. Heat was supplied to this unit by a 2500 watt tube furnace. The heated gases left heater and passed into the chamber via a 24 inch long 1 inch diameter pipe. A heating tape controlled by a 10 amp Variac* provided heat to the inlet pipe and trim control of the chamber inlet gas temperature.
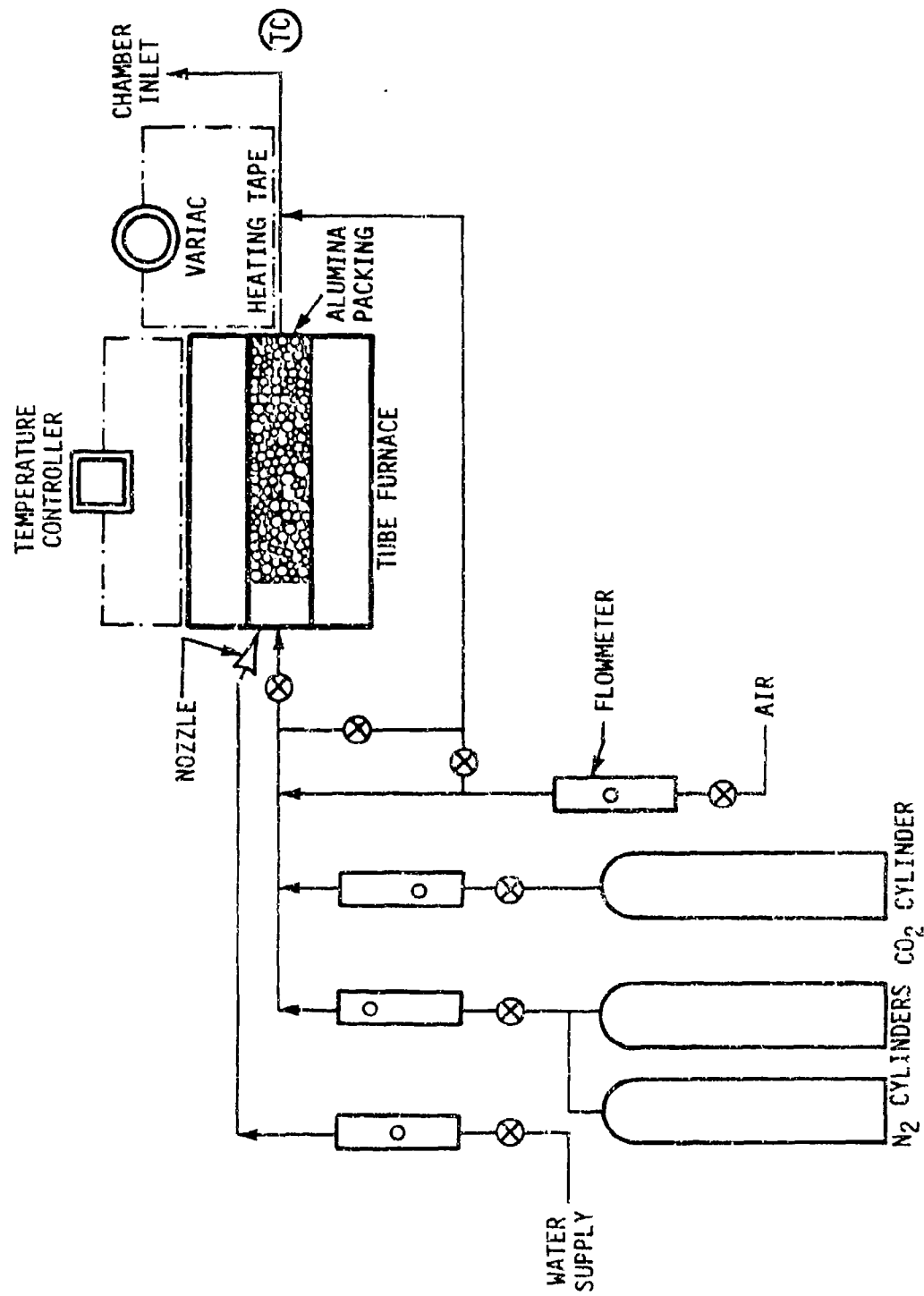
FIGURE 4. GAS DELIVERY SYSTEM

3.4 Effluent Sampling System

The effluent sampling system, which is represented graphically in Figure 5, was designed to sample approximately 1/15 of the chamber effluent, or 2.6 liters/hour. The first stage of this system consisted of two standard 500 ml impingers with fritted gas sparging tubes. These impingers were filled with solvent (usually acetonitrile) and chilled in an ice bath. As such they served to collect most of the explosive and other high molecular weight organics that were contained in the gas sample.

After the impingers, the gas sample passed through two moisture traps that were chilled in a solid carbon dioxide/acetone bath. The first trap, an empty 500 ml impinger with the fritted sparging tube removed, served to collect any solvent that was carried over from the impingers. The second trap, a cold finger, then chilled the gases sufficiently to remove any remaining water vapor by freezing.

The driving force to pull the sample gas through the impingers and traps was provided by a stainless steel bellows pump. A bypass line with a needle valve in line regulated of the sampling flow rate while permitting the pump to run efficiently at full capacity.

The final element of the sampling system was a dry gas test meter. This totalizing flow meter gave a cumulative reading of the total volume of dry gas sampled during the test.

3.5 Solvents

Acetonitrile was used to trap explosives in the gas sampling train impingers, to wash down the chamber after a test, and to extract explosives from coupons. It was also used as the base solvent for stock solutions of HMX. Acetone was used as the base solvent for the stock solutions of the other five explosives. In addition, acetone was used in the gas sampling train for the first few tests with 2,4 DNT. In all cases UV grade distilled in glass solvents purchased from Burdick and Jackson Laboratories Incorporated was used. This grade solvent is commonly used in trace analysis by HPLC and Gas Chromatography.
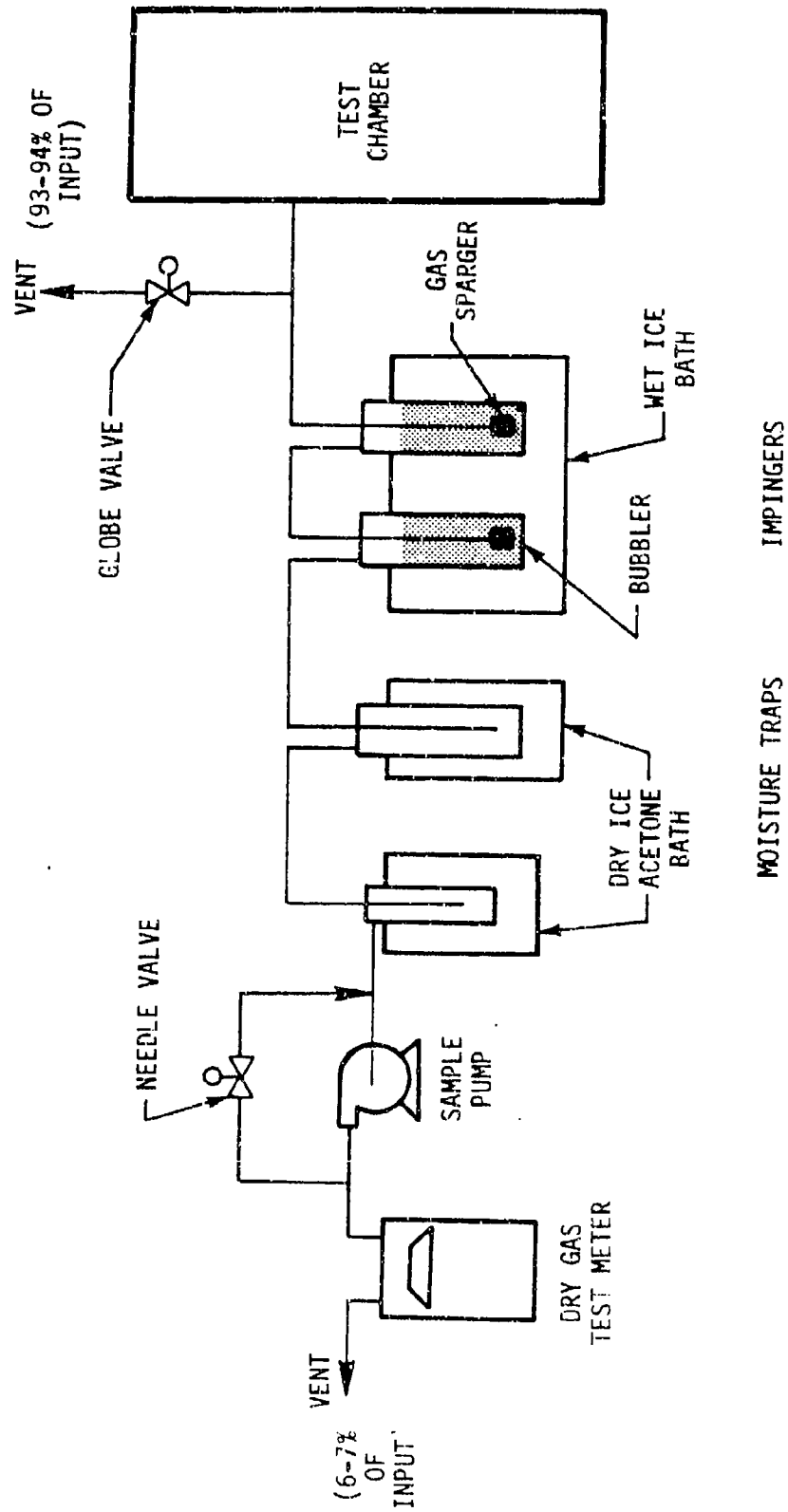
FIGURE 5. EFFLUENT SAMPLING SYSTEM

3.6 Analytical Hardware

Analysis of sample coupons and effluents from the chamber for explosives was performed with High Performance Liquid Chromatography (HPLC). Analysis of samples for decomposition products was performed using a gas chromatograph/mass spectrometer (GC/MS) system. The equipment as well as the procedure used for these analyses are described in Appendix B, Analytical Methods.

3.7 Test Coupons

To evaluate the effectiveness of the concept on removing explosives from stainless steel, explosives were placed on 5-inch square coupons sheared from 18 gauge 304 stainless steel sheet stock.

4.0 EXPERIMENTAL PROCEDURES

The approach used to implement the hot gas concept as well as the rationale behind the test schedule are detailed in the Subtask 5 Test Plan which is attached to this report as Appendix A. Brief discussions of the actual procedures are given below.

4.1 Coupon Spiking

The reported analytical detection limit for each of the six explosives is approximately 2 mg/M^2.[1] Therefore, in order to be able to be certain that >99.9 percent decontamination/removal of explosive were obtained when a belo. detectable limit concentration of explosive was reported by the post test analysis of the coupons, it was necessary to apply 2 gm/M^2 of explosive to the 10.16 cm (4 inch) diameter circle that was being exposed to the hot gases. As described below, this was done by preparing stock solutions of the explosive in an appropriate solvent, slowly applying a sufficient quantity of the solution to the surface of the coupon, and allowing the solvent to evaporate leavings crystals of the explosive on the coupon surface.

4.1.1 Stock Solution

Stock solutions used to spike coupons with the six explosives were prepared by weighing the proper amount of explosive into a volumetric flask, adding enough solvent to dissolve the explosive, and then diluting the resulting solution to the desired total volume with additional solvent. The concentration of explosive in the stock solutions was then confirmed by submitting an aliquot of the stock solution for analysis by HPLC.

Acetone was used as the base solvent for five of the explosives, because of its rapid rate of evaporation at ambient conditions. TNT, Tetryl, 2,4 DNT and 2,6 DNT stock solutions were prepared at a concentration of 5 grams of explosive per 100 ml of solution. Due to its lower solubility, RDX was prepared at a concentration of 2 grams of RDX per 100 ml of solution.

The solubility of HMX in acetone was too low to permit practicle use of such a solution. In addition, the acetone solvent was found to contain an unidentified specie which eluted in the HPLC analysis with the same retention time as the HMX. While this specie would not interfere with the spiking procedure, its presence did prevent quantitative analysis of the spiking solution. For these reasons, other solvents were used. The first HMX stock solution was prepared with cyclohexane as the base solvent. However, the slow rate of evaporation of this solvent made its use impractical for spiking. Subsequent HMX solutions were prepared in acetonitrile which, though providing a more dilute solution and therefore the application of a larger volume of solution, evaporated much more readily than did cyclohexane. The acetonitrile solution was prepared at 1.5 grams of HMX per 100 ml of solution.

4.1.2 Spiking Methods

The stainless steel test coupons were first washed with acetone to remove any oil or other organic contamination. The test coupon was mounted in a sample holder before spiking to assure that the 10.16 cm circular area exposed to the hot gases and the area spiked with explosive coincided. The sample holder and mounted coupon were then placed in a laboratory hood with the retaining plate side of the assembly up. A 50 ml burrett containing the

stock solution was then placed above the sample holder and centered over the exposed circular area of the test coupon. The stock solution was then applied to the coupon by slowly opening the stopcock and allowing approximately 0.5 ml of stock solution to drip onto the surface. This volume of solution would typically wet the majority of the exposed surface without contacting the retaining plate. The air flowing into the hood evaporated the solvent from the coupon surface. Then more solution was applied to the coupon and the procedure repeated until the total desired quantity of stock solution (8 ml of a 5 gm/100 ml stock solution) had been applied. A small quantity of solvent was used after each application to wash the tip of the burrett and transfer any residual explosive from the burrett to the coupon.

To assure that all of the explosives placed on the coupon had crystallized, the combined coupon/holder assembly was then placed into a -10 F freezer for a minimum of 30 minutes. The assembly was stored in the freezer until loaded into the chamber for testing. The quantitative application of explosive to stainless steel coupons by this technique was confirmed by spiking 4 coupons in this manner and then determining the quantity of explo sive that could be recovered by the standard analytical methods described elsewhere in this report. Greater than 85 percent recovery was obtained in each of these tests. In addition, no interferants were observed in any of three analytical blanks similarly run.

4.2 Chamber Test Procedures

To conduct an evaluation of the hot gas concept, three sample holders containing stainless steel coupons that had been spiked with the same explosive in the manner described above were bolted to the chamber wall. They were mounted so that the spiked coupon surface faced the exposure chamber. During this operation the coupon/holder assembly was handled with care to avoid dislodging crystallized explosives from the coupon. Once the coupons were mounted in this manner, the lid was placed on the chamber and the retaining bolts tightened uniformly to assure that the lid sealed the chamber. The chamber containing the three spiked coupons remained in this condition until the test was initiated. This time interval varied from 1 to 16 hours.

Prior to starting the flow of hot gases the electrical power to the tubular furnace which supplied the heat to the gases was turned on and the furnace temperature allowed to rise to 871 C (1600 F). Since a minimum of four hours was required to reach this temperature from ambient temperatures, the furnace was normally left on overnight at a temperature of 590 C. It should be noted that during equipment shakedown tests as well as the experimental evaluations, no increase in the coupon or chamber atmosphere temperature was observed until such time as the gas flow was started. Since the explosives are thermally stable at ambient temperatures, the variability of the total time that the coupons remained in the test chamber prior to initiating the flow of hot gases was not considered of importance to the removal process.

Once sufficient temperature of the air heater was achieved (871 C), a fresh impinger train was attached to the effluent sample line and iced down. After recording the initial reading on the dry gas meter, the flow of hot gases and the sample pump were started simultaneously. The input gas flows were adjusted to yield a total gas flow to the chamber of 56.6 liters/min in the ratio of 71 percent nitrogen, 10 percent CO_2, and 19 percent water vapor. The sample flow rate was adjusted to be approximately 1/15 of the total flow or 2.6 liters/min. The coupon temperatures were monitored continuously and all temperatures and the dry gas meter reading were recorded in a BCL record book at least every 30 minutes. (A copy of a typical data sheet can be found in Appendix C.)

As the coupon temperature were observed to approach the desired temperature, the power was cut back to the heating tapes and the furnace temperature set point was lowered to a valve approximately 50 C above the desired coupon temperature. In this way the heat up time was minimized (~2 hours) without producing excessive overshoot of the desired coupon temperature. Plots of the temperature-time data for each test can be found in Appendix C. The coupons were held at the desired temperature for the desired time interval by adjusting the furnace temperature and/or the power to the heating tape.

After the coupon had been exposed to the desired temperature for the selected time, a run was terminated by stopping the gas flows, turning off the

sample pump, and shutting off the heating tapes and the furnace. It was initially planned to cool the coupon by flowing ambient temperature air into the chamber. In equipment shakedown tests it was demonstrated that this procedure required in excess of two hours. Therefore, during the experimental program, the chamber was cooled by immediately removing the lid and allowing the sensible heat of the chamber and its contents to dissipate to the hood environment. It was thus possible t adequately cool the chamber in less than one hour. After the chamber was c l, the holders containing the treated coupons were removed from the chamber and placed horizontally on a lab bench with the spiked surface facing upward. The coupons were then inspected and the observations recorded in the lab book. The retaining plate was then loosened and the coupons were removed from the holder and placed in a plastic sample box until they could be analyzed for explosives by the procedures given in Appendix B.

In order to determine the quantity of residual explosives contained within the chamber, the chamber surfaces were rinsed with a measured volume of acetonitrile. A sample of this rinse solution as well as samples from the impingers and traps on the effluent sampling train were analyzed for explosives content. The procedures used to prepare these samples and perform the analysis are described in Appendix B.

4.3 Calculations

The HPLC analytical method for the analysis of explosives determined the concentration of the explosives in the liquid solutions (μg/ml). The total weight of explosive (mg) contained in the liquid samples was determined by simply multiplying this concentration by the volume of the liquid sampled. For coupon extracts and the chamber rinse solution, this weight was assumed to represent the total mass of residual explosive found in these locales. However, the effluent sampling system collected only a fraction (~1/15) of the total effluent. It was therefore necessary to multiply the weight of explosives determined in the above manner by the ratio of the total gas volume passing through the chamber during the test to the gas volume sampled (~15/1) in order to determine the total weight of explosive that was contained in the entire effluent.

The quantity of explosive placed on the coupon during sampling was calculated by multiplying the concentration of the spiking solution (determined by HPLC analysis) by the total volume of solution placed on the coupon during spiking.

5.0 DISCUSSION OF RESULTS

As stated previously, the objective of these tests was to determine the viability of the hot gas concept and to select operating conditions (time and temperature) that were sufficient to remove/decontaminate all six explosives from stainless steel. Following a review of the decomposition rate and other physical properties data contained in the Task 2 Final Report[1] and summarized below in Table 1., 2,4 DNT and HMX were selected as the worst case explosives with which to determine sufficient conditions. As described in the test plan (Appendix A), a matrix of tests was conducted with both of these explosives. The results of this matrix was used to select conditions which were to be used in tests with the remaining four explosives (2,6 DNT, TNT, Tetryl, and RDX) to confirm that a hot gas treatment would in fact remove/decontaminate all six explosives from a stainless steel surface. Selected samples from tests conducted under these conditions were then submitted for analysis by GC/MS to attempt to identify products. The results of these tests are discussed in detail below. Sample data, including time vs. temperature profiles can be found in Appendix C. The actual data can be found in BCL record book No. 39324.

5.1 2,4 DNT Tests

The relatively long time predicted to achieve 99 percent thermal decomposition of 2,4 DNT was the property for which this explosive was selected for testing. The results of the six tests conducted with coupons spiked with 2,4 DNT are presented in Table 2. During these tests, acetone was used as the solvent in the effluent sample train impingers.

TABLE 1. VOLATILITY AND THERMAL STABILITY OF EXPLOSIVES

Explosive	Predicted Time[a] For 99 Percent Decomposition At 500 F	Vapor Pressure At 482 F (Torr.)
2,6-DNT	6 days	44
2,4-DNT	2 days	169
TNT	45 minutes	56
HMX	10 minutes	< 1
RDX	50 seconds	5
Tetryl	25 seconds	(b)

(a) Task 2 Report, DRXTH-TE-CR-83211.

(b) Reportedly explodes at 369 F.

The temperature/residence times tested included 1,3, and 6 hours at approximate 218 C (400-450) F, 1 hour at 300 C (550-600 F), and 1 and 3 hours at 320 C (600-620 F). The heat up time required to achieve these temperatures ranged from 120 minutes (218 C) to 150 minutes (320 C). In none of these tests was any visible residues observed on the coupon or chamber surfaces. As can be seen in Table 2, all of the conditions tested reduced the 2,4 DNT level on the spiked coupons to levels below the detection limit of the analytical method. At the milder conditions, small quantities of 2,4 DNT were found in the chamber washdown. This could be attributable to a combination of mechanisms including run off of melted explosives, condensation of volatilized explosive or simply crystals of explosive that fell from the spiked coupons. More severe temperature and time conditions reduced the concentration in the chamber wash down to below detectable limits. Of more interest is the presence of significant levels of 2,4 DNT in the flue gas. This observation as well as the relatively high vapor pressure reported for this explosive (169 torr at 250 C) indicate that volatilization is an important mechanism in the removal/decontamination of 2,4 DNT. This property results in operating conditions that are sufficient to remove the explosive being milder than anticipated.

5.2 HMX Tests

HMX, while being the thermally least stable of the six explosives, has a high melting point and extremely low vapor pressure within the temperature range projected for the hot gas concept. Consequently the mechanism for removal of this explosive was expected to be markedly different than the mechanism for 2,4 DNT.

The results of the six tests conducted with this explosive are presented in Table 3. Prior to these tests it was discovered that the acetone solvent produced an interference with the HPLC analysis for HMX. Consequently, the impingers in the flue gas sample train were filled with acetonitrile for these and all future tests.

After treatment, all HMX-contaminated coupons were coated with a reddish brown residue. Since this residue flaked easily, flakes sometimes

TABLE 2. HOT GAS DECONTAMINATION OF 2,4-DNT ON STAINLESS STEEL RESULTS

Spiking Level 400 mg 2,4-DNT/Coupon(a)

Run No.	Treatment Conditions	Recoveries					
		Coupons(b)		Chamber Washdown		Flue Gas	
		mg	%	mg	%	mg	%
1	1 hr at 600-620 F	<0.05	<0.01	0.16	0.01	(c)	(c)
2	3 hr at 600-620 F	<0.05	<0.01	<0.125	<0.01	155	12.9
3	1 hr at 550-600 F	<0.05	<0.01	0.23	0.02	91	7.6
4	1 nr at 400-450 F	<0.010	<0.003	0.24	0.02	232	19.3
5	3 hr at 400-450 F	<0.010	<0.003	0.03	0.003	131	10.9
6	6 hr at 400-450 F	<0.010	<0.003	<0.025	<0.002	143	11.9

(a) Equivalent to 49 g/m^2

(b) Average of three coupons

(c) Solution volume not determined

TABLE 3. HOT GAS DECONTAMINATION OF HMX ON STAINLESS STEEL RESULTS

Spiking Level - 400 mg/coupon(a)

Run No.	Treatment Conditions	Recoveries									
		Coupons						Chamber			
		Exit		Middle		Entrance		Wash Down		Flue Gas	
		mg	%	mg	%	mg	%	mg	%	mg	%
7	1 hr at 410 F	193	48.3	100(b)	25.0	212	53.0	5.73	0.5	5.01	0.4
8	1 hr at 500	<.01(c)	<.003	<.01(c)	<0.003	<0.01(c)	<0.003	3.3	0.3	<0.7(c)	<0.06
9	6 hrs at 410	47.8	12.8	110	27.5	21.9	5.5	90.5	7.5	10.4	0.87
10	1 hr at 460	112	28.0	70	17.5	32.1	8.0	159	13.3	18.39	1.5
11	12 hrs at 410	3.3	0.75	2.0	0.5	16.7	4.2	93.5	7.8	5.29	0.4
12	6 hrs at 460	0.054	0.014	<0.01(c)	<0.003	0.030	0.007	2.58	0.22	31.62	2.63

(a) equivalent to 49 g/m²

(b) coupon momentarily reached 460 F

(c) below detectable limit

fell off the coupon and collected in the bottom of the chamber. Removal of the HMX from the coupons to below dectable limits by treatment with hot gases was more difficult than removal of 2,4 DNT. As can be seen in Figure 6 accomplishing such removal required either brief exposure to high temperatures (1 hour at 260 C) or long exposure to more moderate temperatures (e.g., six hours at 238 C). Little HMX was found in the flue gas suggesting that if volatilization occurs, the HMX rapidly decomposes in the gaseous state. Considering the low vapor pressure and high melting point (270 C) of HMX, it is more likely that decomposition on the surface in the solid/liquid state is the important mechanism.

5.3 Confirmation Tests

Based on the 2,4 DNT and HMX tests, exposure to 500 F for 1 hour was selected as sufficient operating conditions for the hot gas concept. The results of the tests to confirm removal/decontamination of the remaining four explosives from stainless steel are presented along with results from comparable tests with 2,4 DNT and HMX in Table 4. Treatment with hot gases of 500 F for 1 hour removed greater than 99.97 percent of the spiked quantity (~400 mg) of any of the six explosives. Both volatilization and decomposition appear to be important mechanisms in the removal of explosives from stainless steel. In Table 5, this is demonstrated by comparing the content of explosive in the flue gas with the thermal properties of the explosives. The three most volatile explosives (TNT, 2,4 DNT and 2,6 DNT) are found in the greatest quantity in the flue gas. However, these quantities are ordered to match their thermal stabilities. Therefore, the most stable explosive (2,6 DNT) is found in the greatest quantity even though it is less volatile than TNT or 2,4 DNT. Only small quantities of HMX and RDX, which have low vapor pressures and are thermally less stable, are found in the flue gas. Coupons spiked with tetryl were coated with a heavy carbonaceous residue after the hot gas treatment. This explosive is the thermally least stable of the six studied. Though no vapor pressure data is available for this explosive as it reportedly decomposes rapidly at 187 C, the higher content in the flue gas indicates that it is more volatile than either RDX or HMX.
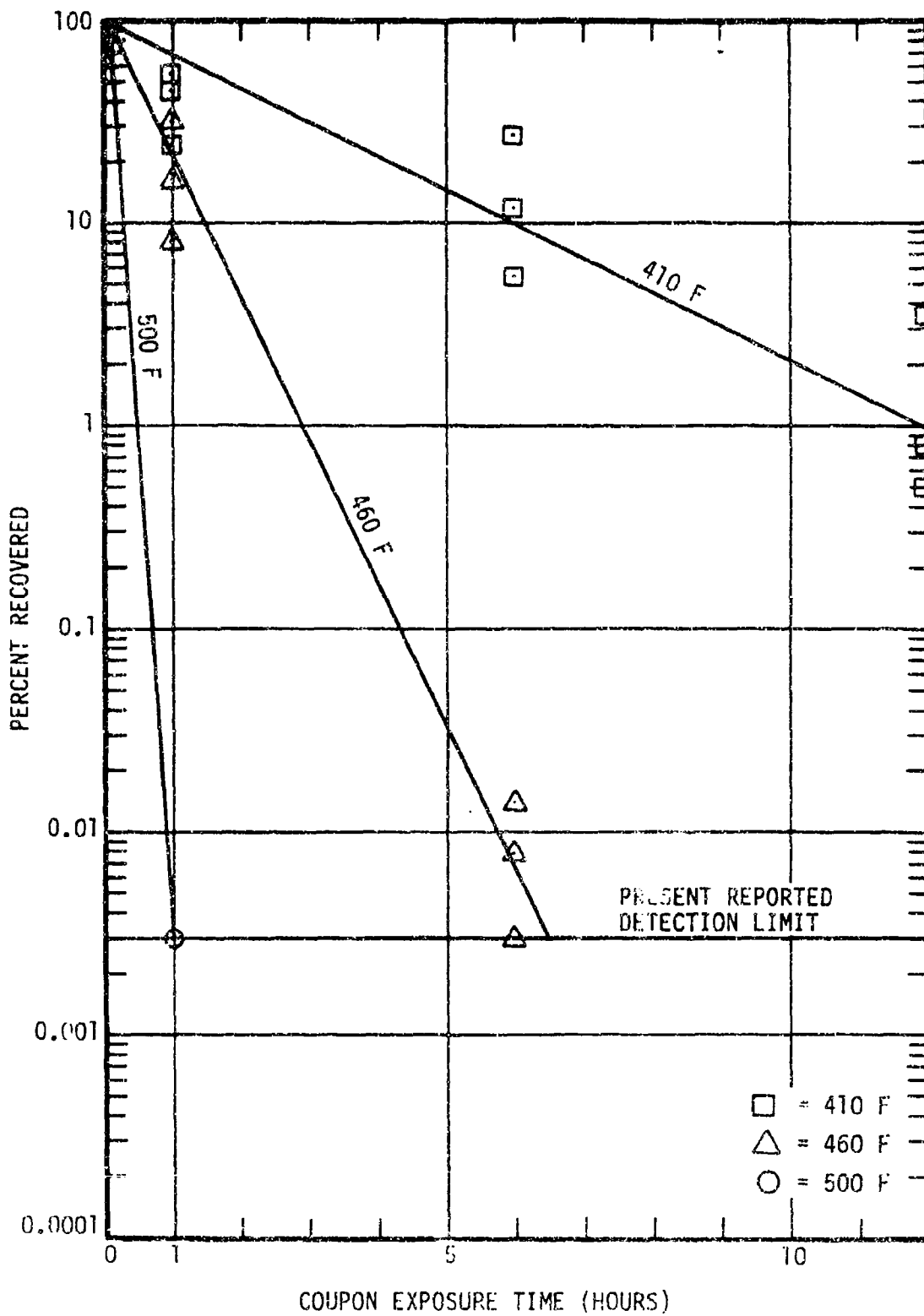
FIGURE 6. PERCENT HMX RECOVERED vs TIME

TABLE 4. HOT GAS DECONTAMINATION CONFIRMATION RESULTS

Test Conditions: Coupon heated to 500°F in approximately 2 hours and then held at temperature for 1 hour.

			Recoveries								
			Coupon Position in Chamber						Chamber		Flue Gas	
			Exit		Middle		Entrance		Wash Down			
Run No.	Explosive	Spiked Level mg/coupon	mg	%	mg	%	mg	%	mg	%	mg	%
4(a)	2,4-DNT	400	<0.010(b)	<0.003	<0.010(b)	<0.003(b)	<0.010(b)	<0.003	0.24	0.02	232	19.3
8	HMX	400	<0.01(b)	<0.003	<0.01(b)	<0.003	<0.01(b)	<0.003	3.3	0.28	<0.7(b)	<0.06
14	Tetryl	416	0.083	0.020	0.101	0.024	0.041	0.010	<0.025(b)	<0.002	8.91	0.71
15	TNT	352	<0.005(b)	<0.0014	<0.005(b)	<0.0014	<0.005(b)	<0.0014	<0.012(b)	<0.0011	65.5	6.20
17	2,6-DNT	404	0.012	0.0030	0.013	0.0032	0.012	0.0030	0.063	0.0052	309	255
18	RDX	412	<0.01(b)	<0.0024	<0.01(b)	<0.0024	<0.01(b)	<0.0024	0.173	0.014	1.54	0.12

(a) 450 for 1 hour
(b) Below detectable limit

TABLE 5. VOLATILITY AND THERMAL STABILITY VS. FLUE GAS CONTENT

Explosive	Predicted Time[a] For 99 Percent Decomposition At 500 F	Vapor Pressure At 482 F (Torr)	Percentage of Spiked Explosive Recovered In Flue Gas
2,6-DNT	6 days	44	25.5 percent
2,4-DNT	2 days	169	19.3 percent
TNT	45 minutes	56	6.2 percent
HMX	10 minutes	<1	0.06 percent
RDX	50 seconds	5	0.12 percent
Tetryl	25 seconds	(b)	0.71 percent

(a) Task 2 Repor., DRXTH-TE-CR-83211.

(b) Reportedly explodes at 369 F.

5.4 Product Identification

While no decomposition products were observed during the HPLC analysis, a further attempt to identify products of the thermal decomposition of the explosives, was made by selecting samples from successful tests and submitting them for analysis by gas chromatography/mass spectrometry (GC/MS) using the procedures described in Appendix B. The mass spectrometric data is presented in Appendix C. No product peaks were observed that could attribute to more than 3 mg of material (<0.75% of the spiked explosive).

This information supports the predicted result that the decomposition products are either light weight gaseous compounds or heavy carbonaceous compounds (soot) that are insoluble in acetonitrile and not chromatographable.

6.0 CONCLUSIONS AND RECOMMENDATIONS

The results of these tests have indicated that the removal of the six explosives from stainless steel by the hot gas concept occurs through a complex combination of at least two mechanisms -- vaporization and decomposition. Further, it appears that, with stainless steel, the lower limit of satisfactory operating temperatures is determined by the explosives that do not readily volatilize, namely HMX. This may not be true with a more porous matrix such as concrete where mass transfer limitations might restrict the volatilization. However, regardless of the mechanism, the removal effected by this technique is sufficient to warrant its inclusion in Subtask 6 studies.

Selection of the optimum conditions for hot gas decontamination/ removal of these six explosives from building materials requires a much broader matrix of tests as well as a complete engineering evaluation. However, the limited testing conducted in this task indicates that treatment at 500 F for 1 hour is sufficient for >99.97 percent removal from stainless steel of any of the six explosives. It is recommended that testing of these conditions be performed on the more porous and/or painted materials.

REFERENCES

1) "Development of Novel Decontamination and Inerting Techniques for Explosive Contaminated Facilities" Phase I Final Report to USATHAMA Contract DAAK11-81-C-0101, July 1983, by H.P. Benecke et.al., Battelle Columbus Laboratories.

2) Work and Resource Plan for "Task 4 - Development of Novel Decontamination and Inerting Techniques for Explosive Contaminated Facilities - Phase II", to USATHAMA, Contract DAAK11-81-C-0101, August 29, 1983, by H.P. Benecke and D.R. Hopper, Battelle Columbus Laboratories.

3) "Test Plan for Design and Construction of the Agent/Explosives Decontamination Concepts Test Chambers", to USATHAMA, Contract DAAK11-81-C-0101, September 12, 1983, by D.R. Hopper et. al., Battelle Columbus Laboratories.

4) "Custodian Plan for Physical Security of Sensitive Explosives at Battelle Columbus Laboratories" to USATHAMA, Contract DAAK11-81-C-0101, September 22, 1982, by E.J. Mezey, Battelle Columbus Laboratories.

APPENDIX A

TEST PLAN

TASK 4, SUBTASK 5

TEST PLAN

for

STAINLESS STEEL SURFACE
DECONTAMINATION SCREENING

Contract No. DAAK11-81-C-0101

to

UNITED STATES ARMY
TOXIC AND HAZARDOUS MATERIALS AGENCY

November 15, 1983

1.0 INTRODUCTION

The development of novel concepts for the decontamination and inerting of explosives-contaminated buildings is being carried out by Battelle's Columbus Laboratories (BCL) for the United States Army Toxic and Hazardous Materials Agency (USATHAMA) under Contract No. DAAK11-81-C-0101. In a previous phase of this contract (Task 2), ideas were systematically developed into concepts for decontaminating and inerting buildings and equipment.[1] These concepts were also evaluated and ranked with respect to selected technical and economic factors and five were selected for laboratory evaluation. Knowledge gaps pertaining to these concepts were identified. Task 4 was then established with four experimental subtasks designed to permit laboratory resolution of these knowledge gaps[2]. This subtask is the third of these experimental subtasks to be conducted.

2.0 PURPOSE AND OBJECTIVES

The purpose of this subtask is to provide a preliminary evaluation of the five candidate concepts that were selected as the most

promising during the previous subtasks. The concepts to be evaluated in this subtask include:

- Hot gases
- Vapor/solvent circulation
- Three chemical concepts to be selected in Task 4, Subtask 4

These five concepts will be evaluated for their ability to decompose or remove explosives placed on the surface of stainless steel coupons. Stainless steel was selected because it is the substrate from which the analytical techniques achieve the highest recoveries of explosives and therefore yield an accurate measurement of the decontamination/removal accomplished by the process. The final objective of this task is to rank-order the tested concepts and select a maximum of three to be subsequently evaluated on the more difficult steel and concrete substrates.

3.0 FACILITIES

In order to evaluate decontamination/removal concepts in a controlled and safe manner, a test chamber to submit contaminated coupons to various decontamination processes has been designed and constructed[3,4]. Performance of this chamber will be determined prior to conducting concept evaluation.

Due to the toxic nature of the small quantities of explosives used in these tests and to assure custody of the explosives, the test chamber will be placed in a hood, which is located in Battelle's Toxic Substance Laboratory (TSL). This is a restricted access facility designed to permit the conduct of experimental work with toxic materials while preventing the release of such materials to the environment. Plans for the custodial security of the explosives have been previously submitted[5].

4.0 EXPERIMENTAL PROCEDURES

The five proposed concepts are described in detail in the Task 2 Final Report[1]. As can be seen in these descriptions, each concept has its own characteristics and operating parameters of importance. While the detailed test procedures will be varied to reflect these differences, the following general testing concept will remain the same.

Stainless steel coupons will be spiked with a known quantity of explosive (TNT, RDX, HMX, 2,4 DNT, 2,6 DNT, or tetryl). The concentration level to which the coupons will be spiked will be based on the detection limit of the analytical methods and the desire to prove decontamination of at least 99.9 percent (see Section 6.0, Coupon Spiking). Spiked coupons will then be mounted in the test chamber, the test chamber will be sealed, and the decontamination treatment applied while collecting all gaseous and liquid effluents. The chamber will then be opened, the coupons removed, and all internal surfaces of the chamber washed with acetonitrile to remove residual explosives. The explosives content of the coupons and the gaseous and liquid effluents (including the chamber washdown) will then be determined. When appropriate, attempts will also be made to determine reaction products that may appear in these samples. These data will determine the destruction/removal efficiency of the process and lend assistance in determining the fate of the explosives during treatment. The detailed descriptions of the tests for each concept are given below.

4.1 Hot Gases

4.1.1 Test Description

This concept utilizes hot inert gases, such as burner exhaust gases, to heat and thermally decontaminate a building. The process variables of importance are the gas temperature and flow rate and the surface temperature of the coupons.

The process must be designed to thermalize any of the six explosives of interest. The approach to be taken is to determine conditions sufficient to thermalize the worse case explosives and then confirm decontamination/removal of the remaining explosives under these conditions. In selecting the worse case explosives, consideration must be given to both the kinetics of decomposition as well as the rate of volatilization. The available kinetic data indicate that the two isomers of DNT require markedly longer decomposition times at the same temperature than the other explosives. There are questions about the validity of these data and experimental confirmation is needed. Review of the available vapor pressure data indicates the DNT isomers have appreciable vapor pressure at 300 C and could conceivably be removed by volatilization rather than decomposition. The vapor pressure of HMX at this temperature is 0.1 torr. The preliminary tests will be conducted with HMX and 2,4 DNT to assure that both mechanisms have been investigated. Collection and analysis of all residues should determine the fate of the explc 'es and thereby the mechanism of removal.

To determine the effectiveness of this concept in decontamination/removing explosives, the test chamber will be arranged in the configuration illustrated in Figure 1. To simulate the atmosphere produced by combustion of stoichiometric ratios of natural gas and air, a gas mixture of seven parts N_2 to one part CO_2 will be humidified by passing it through a water column heated to 60 C, heated to approximately 620 C by an electric heater, and then directed into the inlet port of the insulated test chamber where it will heat the chamber and the test coupons. From the chamber, the gases will be vented through an impinger train designed to collect volatilized explosives and/or products of decomposition. The gas inlet temperature and the total flow rate will be adjusted to provide a rapid heatup with a minimal coupon temperature overshoot. Once the coupon has reached the desired temperature (300 C), the flow rates will be modulated to maintain that temperature. It is estimated that under these conditions, the gas temperature in the chamber will be 400 C. The selected residence
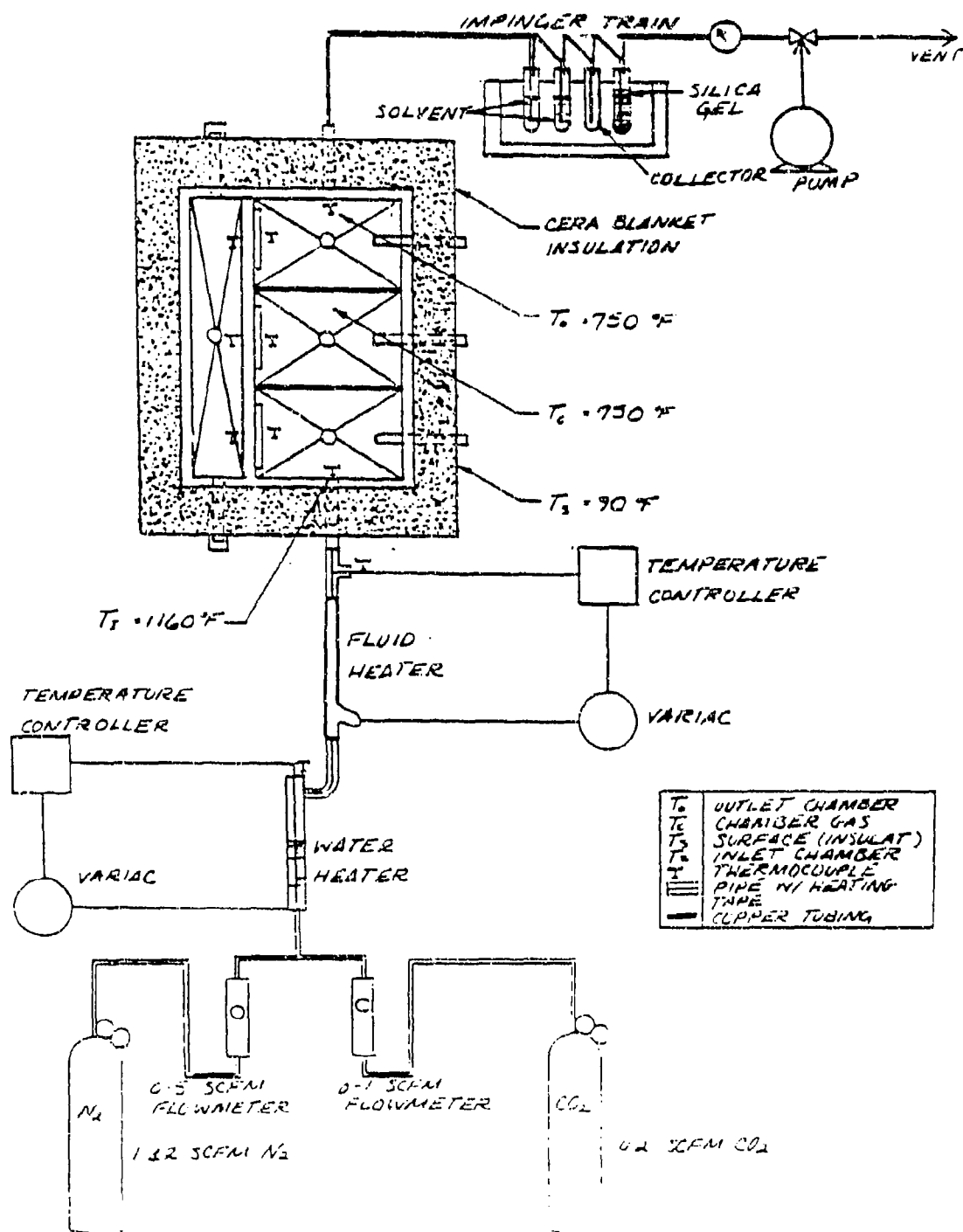
FIGURE 1. HOT GASES EQUIPMENT ARRANGEMENT

times for the testing will be one, four, and eight hours. These times bracket the residence time that kinetic data indicate should produce 99.9 percent decomposition of 2,4-DNT.

Post test analysis of the coupon, the gaseous effluent impinger train, and the chamber washdown for explosives will indicate whether sufficient removal of explosives occurred and if so, was it by decomposition, vaporization, or simply runoff of melted explosives.

4.1.2 Performance Tests

Prior to conducting experiments with explosives, performance tests will be conducted to:

- Assure the apparatus is working properly
- Determine the gas flow conditions required for proper coupon heatup
- Produce sample blanks to provide an analytical baseline
- Determine the thermal gradient which exists across the sample coupons when subjected to test conditions.

4.1.3 Contingency Plans

After conditions sufficient to thermalize HMX and 2,4-DNT have been determined, it is anticipated that only four additional tests will be required to confirm thermalization of the other four explosives. Should the decontamination/removal of any of these explosives prove to be incomplete, testing with this explosive(s) at more severe conditions will be performed.

4.2 Vapor Circulation

4.2.1 Test Description

In this concept, a vaporized solvent is circulated in the building where it condenses on cool surfaces, heating them and

solubilizing any explosive contaminants. Other than the solvent, the operating variables of importance in this concept are the rate and duration of solvent application. The solvent of choice presently appears to be Freon® 113. This solvent, which is the basis of the RADKLEEN® process, has a reasonable boiling point and is not flammable. While these characeristics make it an attractive choice, no data on explosives solubility in Freon® 113 is available. To determine the applicability of this solvent, such data will be generated in the same manner as that used in Subtask 3[6]. If these data indicate solubility is inadequate, consideration will be given to the use of other solvent systems. These data will also be used to order the explosives for testing. Initial condition determinations will be made by testing the least soluble explosive at three conditions. Once sufficient conditions have been determined, removal of the five remaining explosives will be confirmed.

To determine the effectiveness of decontamination of the vapor circulation concept, stainless steel test coupons will be mounted in the test chamber which will be arranged in the configuration represented in Figure 2. The solvent will be vaporized in a boiler and carried into the chamber by a nitrogen purge. The boiler conditions will be adjusted to match the solvent boiling point and the desired application rate (e.g., Freon® 113 - bp: 117.6 F; rate 0.13 lb/min). The inlet plumbing is heated to prevent condensation and allow superheating of the solvent vapors. The chamber is insulated to minimize thermal losses as the vapors condense on the internal surfaces which include the sample coupon. The condensed liquids will drain into and be collected by the chamber sumps. The nitrogen purge gas containing uncondensed solvent and possibly volatilized explosives or decomposition products will be directed through an ice-cooled impinger train that will collect these materials for later analysis.

It is anticipated that one hour will be required to heat the sample coupons from ambient temperature to a steady-state temperature near the solvent boiling point. As a starting point, coupons will be held under this condition an additional hour before being removed and
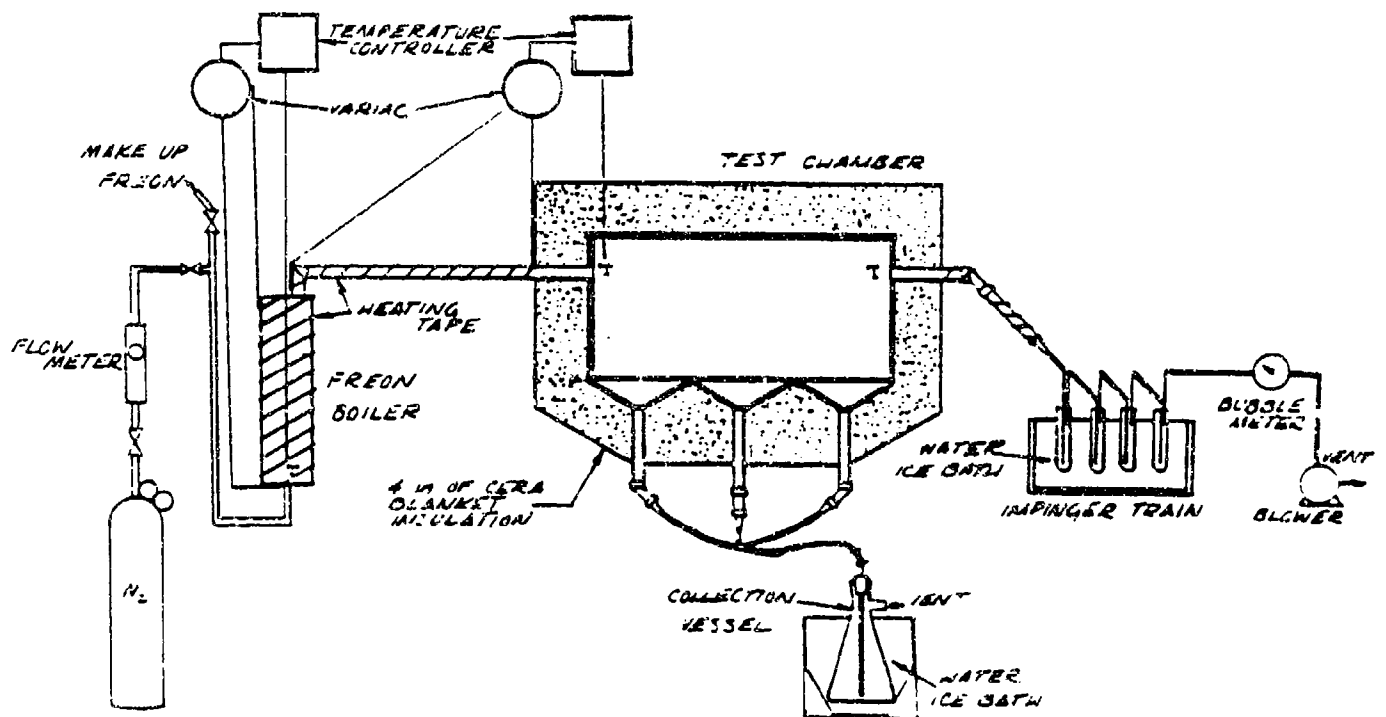
FIGURE 2. VAPOR CIRCULATION/RADKLEEN®
EQUIPMENT ARRANGEMENT

analyzed for residual explosive content. Liquids collected in the sump
and the impinger train will also be analyzed for explosive content.
Though decomposition of explosives by this concept is not anticipated, a
qualitative evaluation of products in these samples will also be made.

4.2.2 Performance Tests

Prior to conducting experiments with explosives, performance
tests will be conducted to:

- Insure all equipment is functioning properly
- Substantiate the solvent flow rates
- Determine the time to reach steady-state conditions and the
  coupon temperature produced
- produce sample blanks to provide an analytical baseline.

During these performance tests, the solvent and nitrogen flow
rates, which produce a rapid coupon heatup to temperatures near the
solvent boiling point, will be determined.

4.2.3 Contingency Plans

The exposure times for subsequent tests with the least soluble
explosive will be based on the results obtained from a 1-hour exposure.
Complete removal of the explosives in one hour will necessitate testing
at shorter exposure times, conceivably shorter than the time required to
reach steady-state conditions. Naturally, incomplete removal will
result in testing longer exposure times (2 to 4 hours). If the con-
firmation tests produce insufficient removal of any of the other five
explosives, additional testing of the explosive(s) will be performed.

As stated previously, if preliminary results from this subtask
indicate that the solubility of explosives in Freon® 113 is insuffi-
cient, the use of alternative solvents will be considered. It is
anticipated that a water-based cosolvent system might be utilized. The
results of Subtask 3, Enhanced Aqueous Solubilization Studies[7], will
be incorporated in this selection process.

4.3 Chemical Concepts

4.3.1 Test Description

Subtask $4^{(7)}$ will prescreen and rank-order the chemical decon-
tamination concepts identified in Task $2^{(1)}$. In this subtask, the three
highest-ranked concepts will be further evaluated.

Sufficient decomposition/removal conditions will be determined
by an approach similar to that used in the prescreening studies.[7] It
will be assumed that the DNT isomers are of such similar chemical nature
that conditions which produce adequate decontamination/removal of
2,4 DNT will suffice for the 2,6 DNT isomer as well. Similarly, it is
assumed that testing RDX will adequately reflect the behavior of HMX and
testing TNT will also adequately reflect tetryl. After determining
the minimal conditions required for each of these three explosives (TNT,
RDX, and 2,4 DNT) the most severe of these conditions will be used to
confirm decontamination/removal of the remaining three explosives. The
operating variables that will be studied are the rate of solvent/reagent
application, the application time, and the spray pattern (droplet size
and distribution). To perform the evaluations, the test chamber will be
equipped with spray nozzles, as shown in Figure 3. Test coupons will be
mounted in the chamber, the appropriate solvent/reagent combination
placed in the reservoir, and the application of solvent started. After
the treatment, both the coupons and the solvent will be quantitatively
analyzed for residual explosives content. In addition, an attempt will
be made to qualitatively confirm the presence of decomposition products
that were identified in the prescreening subtask.

4.3.2 Performance Tests

Performance tests will be conducted prior to conducting tests
with explosive to:

- Assure that all hardware is functioning properly
- Produce sample blanks to provide an analytical baseline.

GENERAL NOTES
1. REMOVABLE PARTITIONS INSTALLED
2. BOTH VIEWS SHOWN WITHOUT CHAMBER LIDS
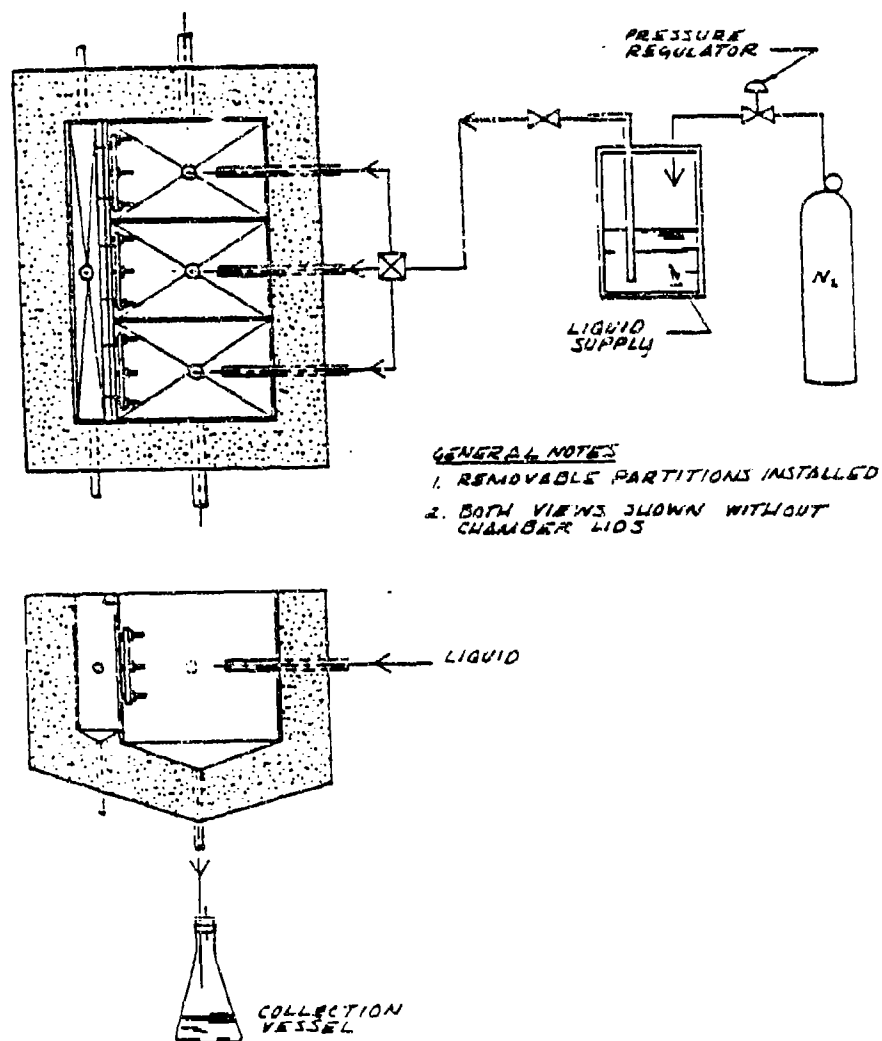
FIGURE 3.   CHEMICAL CONCEPTS EQUIPMENT ARRANGEMENT

During the performance tests, particular attention will be given to the solvent application systems, the only hardware not previously tested. Attempts will be made to determine the distribution of solvent/reagent on the coupons surface that is produced.

4.3.3 Contingency Plans

It is the intent of this subtask to recommend at least one chemical concept for additional study in Subtask 6. If the results indicate that no single or combination of concepts will decontaminate the complete list of explosives, the remaining less highly regarded concepts will be tested.

5.0 MEASUREMENTS

The major objective of this task is to determine the efficiency of destruction/removal of explosives. Therefore, in all experiments, the critical measurement will be the analysis for low concentrations of explosives on the treated coupons. These determinations will be made by High Performance Liquid Chromatography (HPLC) using analytical techniques previously developed and certified in Task 2.[6] To avoid losses, these analyses will be conducted as soon as possible after the test. Decontamination effectiveness will then be calculated by the following equation:

$$\text{Percent Decontamination Effectiveness} = (1 - \frac{\text{residual concentration}}{\text{spiked concentration}}) \times 100$$

Less critical will be the determination of the explosive content of the other process streams. For gaseous process effluents, an impinger train filled with acetonitrile will be used to trap any volatilized explosives. After the test, the impinger contents will be combined, concentrated if necessary, and analyzed for explosives content by HPLC. Acetonitrile is a good solvent for the explosives of

interest and was the solvent used in certifying the HPLC analysis. The chamber wash and the liquid effluents from the chemical methods will also be analyzed by HPLC. As before, using acetonitrile for the wash solvent eliminates concerns of analytical interferences. The use of HPLC has also been done previously and is the basis for the analytical work done in Subtask 3, Enhanced Aqueous Stability Studies.[6]

No previous data exist for analysis of Freon® 113 for explosives content. It is anticipated that samples collected during the performance tests for this method will indicate if interferences with the analytical method exist. However, since the material is extremely volatile, no interference is anticipated.

All of the above samples will be analyzed either by HPLC or GC/MS for the presence of of unreacted explosives and products of decomposition. Such an analysis will be performed qualitatively and in most instances, will be performed only to confirm the products predicted by previous studies.

The final class of measurements is the process variables. These variables are very process-specific and have been discussed in the test descriptions. In general, they fall into the areas of gas flow rate, liquid flow rate, temperature, and time measurements.

6.0 COUPON SPIKING

Stainless steel coupons have been spiked with explosives in previous work[6] and a similar method should suffice for these tests. However, this spiking was done at concentrations near the analytical detection limit. To prove 99.9 percent decontamination/removal, it will be necessary to spike the coupons at levels three orders of magnitude higher. The detection limit for all six explosives on unpainted stainless steel is < 2 mg/m^2. It will therefore be necessary to spike the coupons at 2 gm/m^2. Since the area of exposure is a 4-inch diameter circle or 0.08'1 m^2, it will be necessary to apply 0.0162 grams of explosive to the coupon to produce this concentration. Due to the higher solubilities obtainable, the solvent in the stock solution will be

acetone rather than methanol as used previously. At : C, the solubility of RDX, the least soluble of the six explo /es, in acetone is 7.3 grams/100 grams. If a stock solution of 5 gram./100 grams is used for all explosives, it will be necessary to apply 0.34 grams of solution or less than 0.4 ml. Applying this amount of solution to stainless steel coupons in a uniform manner should not be difficult.

Since the volatility of explosives at ambient conditions is low, no loss of explosive during solvent evaporation is anticipated. To verify that this spiking method is satisfactory, six blank coupons will be prepared in such a manner (one with each explosive), allowed to stand for 4 hours to assure that all solvent is evaporated, and then submitted for analysis. If recoveries similar to those obtained in the certifi-cation testing are achieved, it will be assumed that the spiking method is satisfactory Unsatisfactory recoveries will require reanalysis of the entire spiking procedure.

7.0 SAFETY

At the levels of explosives being used in these tests, no explosion hazard exists. The need to wash out the chamber to recovery residual concentrations, eliminates any possibility that buildup will occur. All handling of explosives, particularly the preparation of the stock solutions, will be performed by experienced personnel. The handling procedures are detailed elsewhere.[5]

8.0 SCHEDULE

The experimental program outlined requires a total of 56 chamber tests conducted. If four additional tests are added for contin-gencies, it is assumed that one test is performed each day and a total of 5 days are allotted for each of the three performance tests, 75 days of testing might be required. With a projected starting date of

November 21, 1983, the tests should be completed by March 9, 1984. This is considerably later than the February 6 date projected on the design plan. However, due to schedule contingencies built into the design plan, no delay in completion of the task is anticipated.

REFERENCES

(1) "Development of Novel Decontaminating and Inerting Techniques for Explosives Contaminated Facilities, Phase I Identification and Evaluation of Concepts", Contract DAAK-11-81-C-0101 to USATHAMA, July 1983, by H. P. Benecke et al.

(2) "Design Plan for Task 4 - Development of Novel Decontaminating and Inerting Techniques for Explosives Contaminated Facilities", Contract DAAK-11-81-C-0101 to USATHAMA, August 29, 1983, by H. P. Benecke and D. R. Hopper.

(3) "Test Plan for Design and Construction of Agent/Explosives Decontamination Concepts Test Chamber", Contract DAAK-11-81-C-0101 to USATHAMA, September 12, 1983, by D. R. Hopper et al.

(4) Letter concerning test chamber design, modification to Andrew P. Roach, COTR, September 22, 1983.

(5) "Custodial Plan for Physical Security of Sensitive Explosives at Battelle Columbus Laboratories", Contract DAAK-11-81-C-0101 to USATHAMA, September 22, 1983, by E. J. Mezey.

(6) "Task 4, Subtask 3 - Test Plan for Enhanced Aqueous Solubilization", Contract DAAK-11-81-C-0101 to USATHAMA, September 12, 1983, by H. P. Benecke.

(7) "Task 4, Subtask 4 - Test Plan for Prescreening of Chemical Decontamination Concepts", Contract DAAK-11-81-C-0101 to USATHAMA, November 1983, by H. P. Benecke.

APPENDIX B

ANALYTICAL PROCEDURES

APPENDIX B

ANALYTICAL PROCEDURES

Coupon Extraction

The procedure used for the recovery of explosives from coupons was identical to that developed by A.D. Little Inc. (Goodwin, 1982) and certified at BCL during Phase I of this program. This recovery involved extraction of the explosive from the coupon with acetonitrile as follows. Following the decontamination test, the treated stainless steel coupons (5"x5") were placed in a 5"x5½" Tupperware box. A 50 ml aliquot of acetonitrile was added to the box which was then covered and the contents sonicated for 15 minutes. The acetonitrile extract from the step was then transferred to a 100 ml volumetric flask and the box and coupon were rinsed with 3-10 ml portions of acetonitrile into the flask. Ten ml of 0.01 phosphoric acid was then added to the flask and the solution diluted to volume with acetonitrile. The resulting solution was then submitted for analysis by high performance liquid chromatography (HPLC).

Effluent Sample Train

After a test, the contents of the impingers and traps were prepared for analysis by HPLC in the following manner. The contents of both impingers were poured into a 250 ml volumetric flask. The impingers and the sample line leading to it were then washed with additional solvent (.etonitrile) which was also transferred to the volumetric flask. The flask contents were then diluted to volume, an aliquot was submitted for analysis of explosives and the remander stored.

The two traps were prepared as an independent sample in a similar manner. In latter runs, when analytical results had established that the impingers and traps were properly collecting the explosives contained in the gas sample, these two sample were combined and diluted as a single sample.

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Chamber Rinse

The acetonitrile used to wash the chamber surfaces and collected in the chamber sump was drained into a volumetric flask (250 or 500 ml). Additional acetonitrile was then used to rinse the sump and drain line. This solvent was also added to the flask and the solution diluted to volume. An aliquot of this sample was submitted for HPLC analysis for explosives. The remainder was stored.

HPLC Analysis

Instrumental analysis of explosives contained in liquid solutions was accomplished by linear gradient high performance liquid chromatography. This procedure which was certified by BCL in Phase I of this program, utilized the following equipment.

- Altex 110 A pump
- LDC Spectromonitor III-UV detector
- Micrometics 725 Autoinjector
- Hewlett Packard 1000 Computer with Computer Inquiry System (CALS) Chromatographic Software.

The operating parameters within which this equipment operated were:

- Column: Spherisorb ODS 5μ, 25cm x 4.6 mm I.D.
- Mobile Phase: methanol/water 50/50, isocratic
- Flow Rate: 1.0 ml/min
- Detector: UV @ 254 nm
- Attenuation: unattenuated output to computer
- Injection volume: 10μl

GC/MS Analysis

Analysis of products was performed on selected liquid samples using a Finnigan 1020 automated GC/MS system that was designed for the analysis of trace organics in water. Chromatography was accomplished on a 30 meter 0.25 mm ID fused silica column coated with a 0.25μ film of Durabond 5. Helium

carrier gas at 20 cm/sec was used. After a 2 minute hold at 50 c, the column temperature was programmed to 250 c at 10 c/min. The analysis was completed by holding the column at 250 C for an additional 5 minutes. During this analysis, the column affluent passed through the mass spectrometer defector operating in the electron Ionization mode at 70 ev.

In order to produce a rough estimate of the quantity of products observed in the ion chromatograms, an analysis of a standard solution of 2,6 DNT was performed. An injection of 2μl of a 200μg/ml, (0.4μg) standard was made and an ion count made of the mass scan representing 2,6 DNT. This ion count (73344) was then used to determine an approximate mass to ion count ratio, 0.4μg/73344 or 5.45×10^{-5} μg/ion count. While not a rigorous calculation, this ratio was adequate for an order of magnitude approximation of the mass of products found in the samples analyzed.

APPENDIX C

EXPERIMENTAL DATA

SAMPLE DATA

The following pages are copies of pages from BCL Record Book 39324.
These pages are typical of all test data that are recorded in this book.

Work Performed by: *Charles Nutter*　Project No. G79.20-0410　Date of Work: 2/20/84

Title or Purpose: *Run #9 HMX 430°F 6Hour Hotgases*　Continued from: *pg. 26*

TIME minutes		THERMOCOUPLE READING °F												Dry Gas Meter ft³	
Clock	Elapsed	1	2	3	4	5	6	7	8	9	10	11	12	READING	Δ
11:45 p.m	0	1909	1659	338	110	65	55	55	66	65	71	72	62	817.54	0
12:15 A.m	30	1767	1146	1475	1268	342	210	230	225	161	341	240	164	823.40	4.86
12:45 A.m	60	1467	808	1364	1287	475	324	353	338	264	423	256	295	828.30	4.9
1:15 A.m	90	1117	364	920	953	471	377	394	390	319	415	261	371	833.71	5.4
1:45 A.m	120	956	392	816	817	469	394	411	396	355	418	265	394	838.42	4.71
2:00 A.m	135	713	339	759	780	482	414	428	414	374	427	272	413	840.9	2.48
2:15 A.m	150	628	354	659	684	463	411	423	413	376	409	260	413	843.42	2.52
2:45 A.m	180	629	475	612	603	442	400	409	402	371	386	251	405	848.5	5.08
3:15 A.m	210	698	542	687	661	458	410	420	412	381	403	257	412	853.35	4.85
3:45 A.m	240	750	547	689	662	456	408	418	410	381	401	265	412	858.10	4.25
4:15 A.m	270	728	554	693	666	456	408	418	410	381	402	265	411	862.8	4.7
4:45 A.m.	300	708	533	691	672	457	408	418	410	381	402	262	411	867.4	4.6
5:15 A.m.	330	700	583	679	660	458	410	421	412	381	402	254	413	871.8	4.4
5:45 A.m.	360	694	528	668	651	459	411	424	413	384	404	269	415	876.3	4.5
6:15 A.m	390	643	538	664	647	459	412	424	414	384	405	270	415	880.9	4.6
6:45 A.m	420	644	547	664	645	459	412	423	415	385	403	264	415	885.3	4.4
7:15 A.m	450	603	560	665	655	536	412	423	414	383	404	268	413	889.6	4.3
7:45 A.m	480	570	538	660	643	458	410	422	413	38x	400	263	412	891.5	1.9
8:00 A.m	495 minutes	565	582	710	655	303	381	385	388	35	317	178	325	893.2	1.7

Shut off Furnace, water, etc. Take off lid

$$\text{Effluent Correction} = \frac{120 \times \frac{495}{60}}{(893.2 - 817.54)} = 1.308$$

Entered by: *Charles Nutter*　Date: 2/20/84　Continued to: 32

Performances of this work observed by:　Disclosed to and understood by me:

J. R. Horne　Date: 2-21-84　Date:

Date:　Date:

PREVIOUS PAGE IS BLANK

0 | 5 | 10 | 15 | 20 | 25 | 30

Work Performed by: Charles Nutter   Project No. G7920-0410   Date of Work: 2/20/84

Title or Purpose: Run #9 HMX (~430°F - 6 Hr) Hot gases   Continued from:

OBJECTIVE   To evaluate the effectiveness of using Hot gases to decontaminate 400 mg of HMX on a 5" dia Circular Area of a Stainless Steel coupon at 400-430°F

Coupon Spiking   26.67 ml. of 1.5% HMX/acetonitrile stock solution was applied via a 100 ml Buret the buret was rinsed with 3 ml of after each amount was applied.

Furnace Power on Friday 2/17/84 5:00 pm

Chamber Heating tapes on 2/19/84 11:45 pm
   "   "   "   off   1:30 Am 2/20/84

39324-26-15	Left Coupon Run #9	
39324-26-16	middle " " "	
39324-26-17	Right " " "	
39324-26-18	Washdown Solution diluted to 250 ml.	
39324-26-19	Impinger " " " 500 ml	
39324-26-20	Trap " " " 500 ml	

Spiking Procedure: To each of 3 individual stainless steel coupons, I applied 26.7 ml of a 1.5% HMX/acetonitrile mixture. These were then refrigerated and placed into the chamber ~ 30 minutes before the start of the run. The 26.7 ml was applied via a 50 ml buret, to each coupon.

Entered by: Charles Nutter   Date: 2/20/84   Continued to: 31

Performances of this work observed by: Disclosed to and understood by me:

R. Hume   Date: 2-21-84   Date:

Date: Date:

TIME/TEMPERATURE .PROFILES

The following graphs are plots of temperature versus time for all of the tests conducted in this subtask.

Run # 1, 2,4 DNT Time–Temperature Profiles
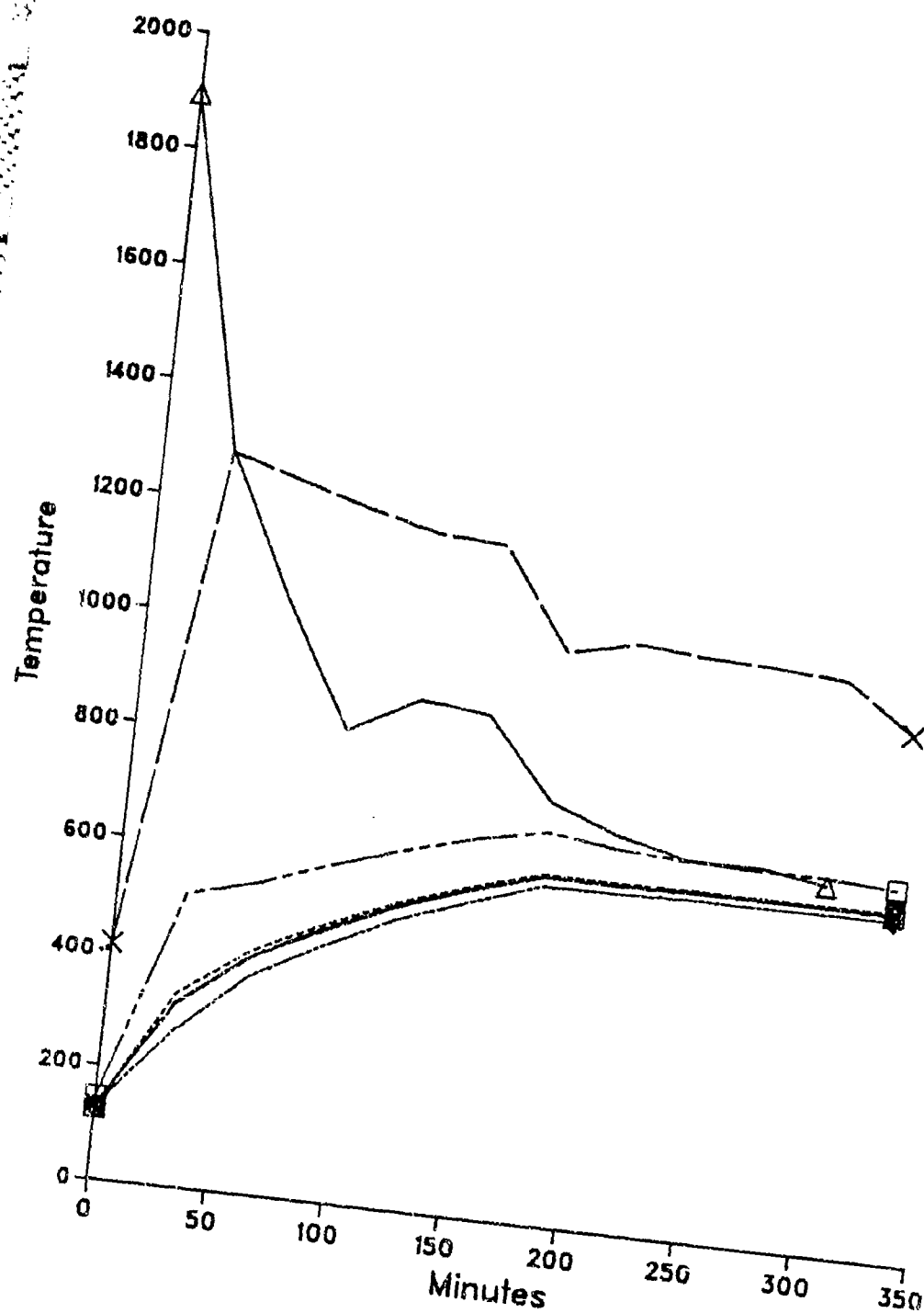
Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmospher
⊠ Entrance Coupon
𝕏 Middle Coupon
✳ Exit Coupon
⊕ Exit Gas

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Run # 2 2,4 DNT Time-Temperature Profiles

Legend
- △ Preheater
- ✕ Inlet Gas
- □ Chamber Atmosph
- ⊠ Entrance Coupon
- ⊠ Middle Coupon
- ✳ Exit Coupon
- ◈ Exit Gas

Run # 3 2,4 DNT Time--Temperature Profiles
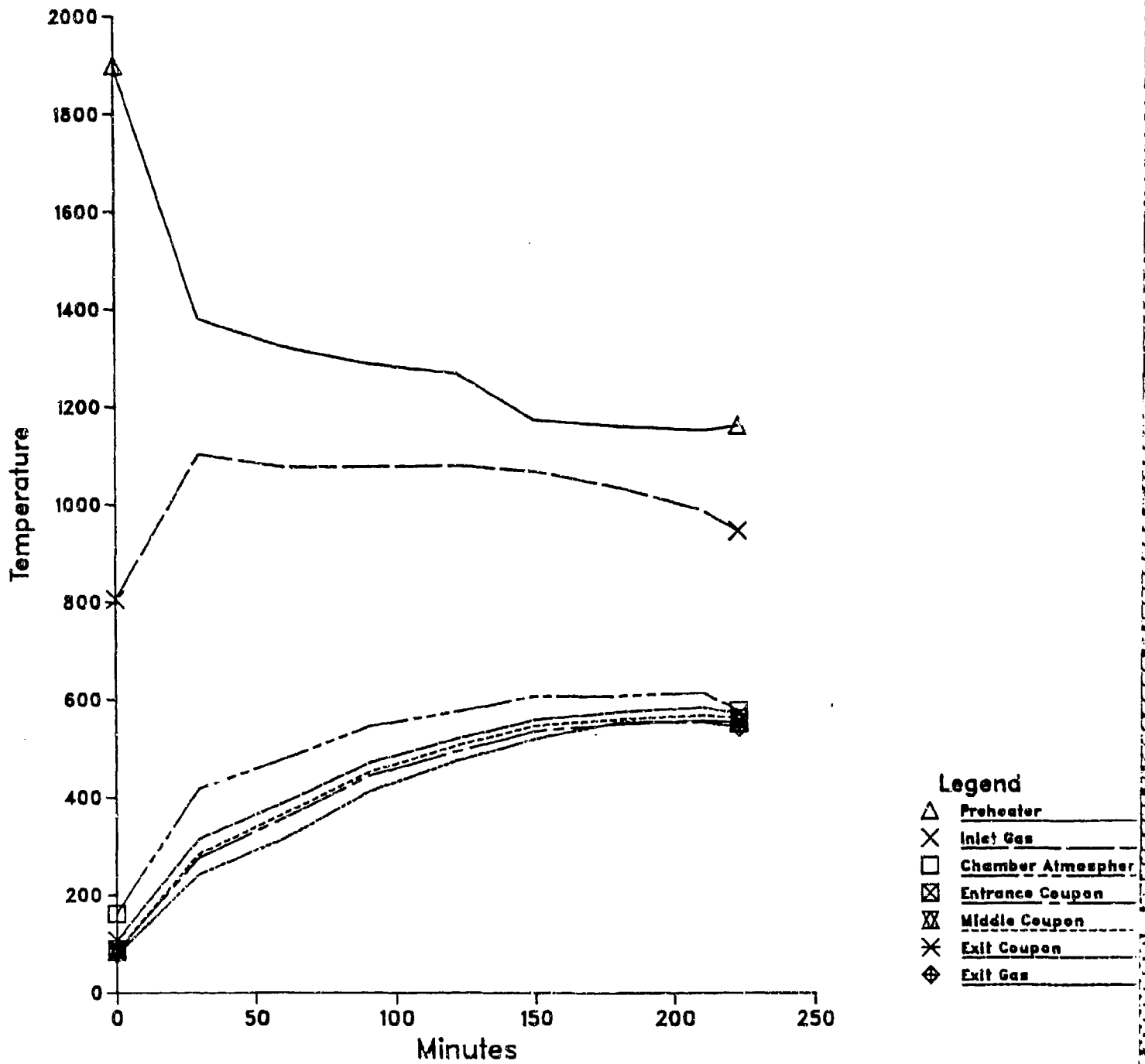
Run # 4 2,4 DNT Time-Temperature Profiles
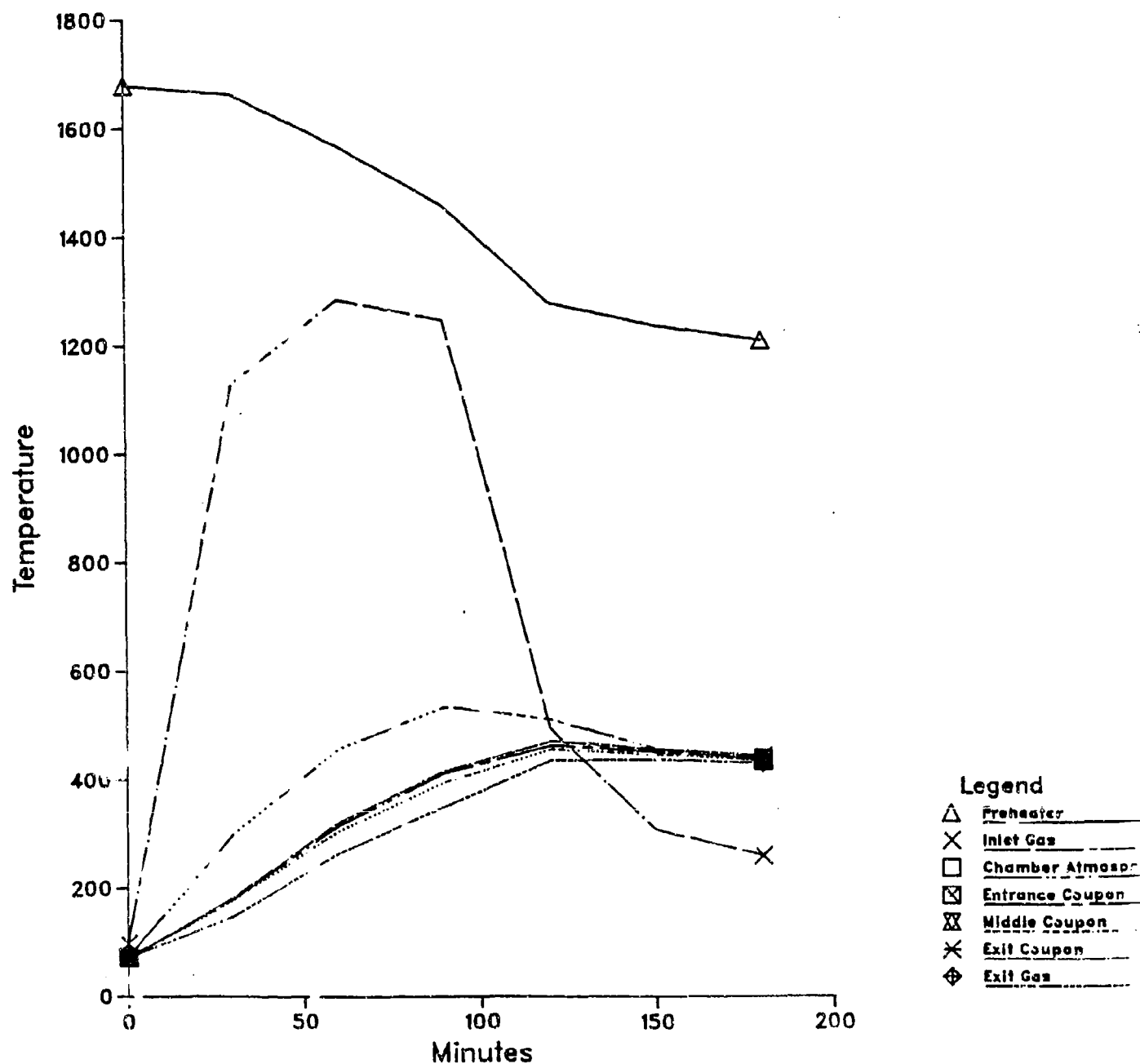
Legend
- △ Preheater
- ✕ Inlet Gas
- □ Chamber Atmosph
- ⊠ Entrance Coupon
- ☒ Middle Coupon
- ✻ Exit Coupon
- ⊕ Exit Gas

Run # 5 2,4 DNT Time–Temperature Profiles

Legend
- △ Preheater
- ✕ Inlet Gas
- ☐ Chamber Atm:
- ⊠ Entrance Coup
- ⧆ Middle Coupor
- ✳ Exit Coupon
- ⬖ Exit Gas

Run # 6 2,4 DNT Time-Temperature Profiles
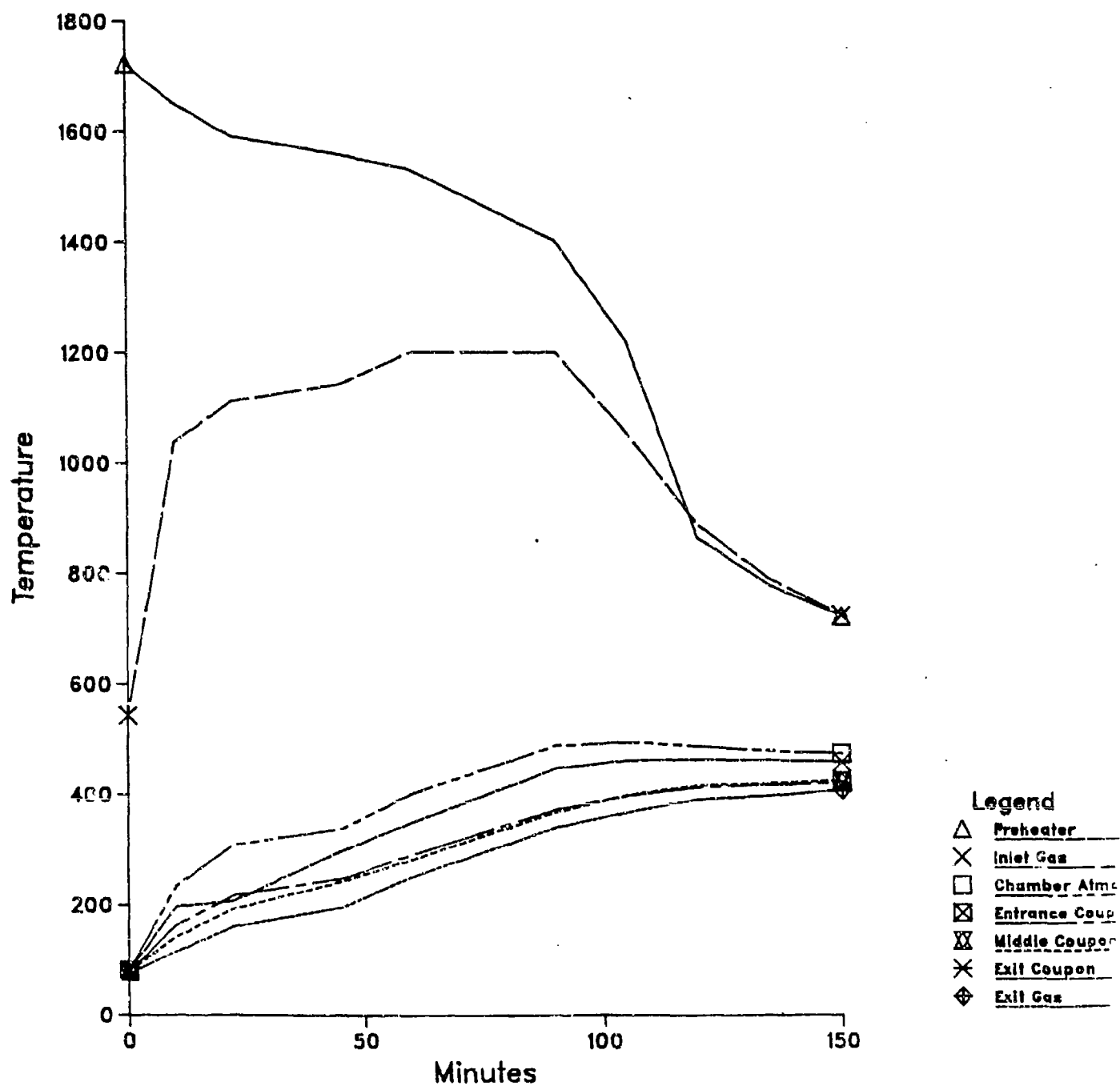
Legend
- △ Preheater
- ✕ Inlet Gas
- ☐ Chamber Atmosphere
- ⊠ Entrance Coupon
- ✕ Middle Coupon
- ✳ Exit Coupon
- ⊕ Exit Gas
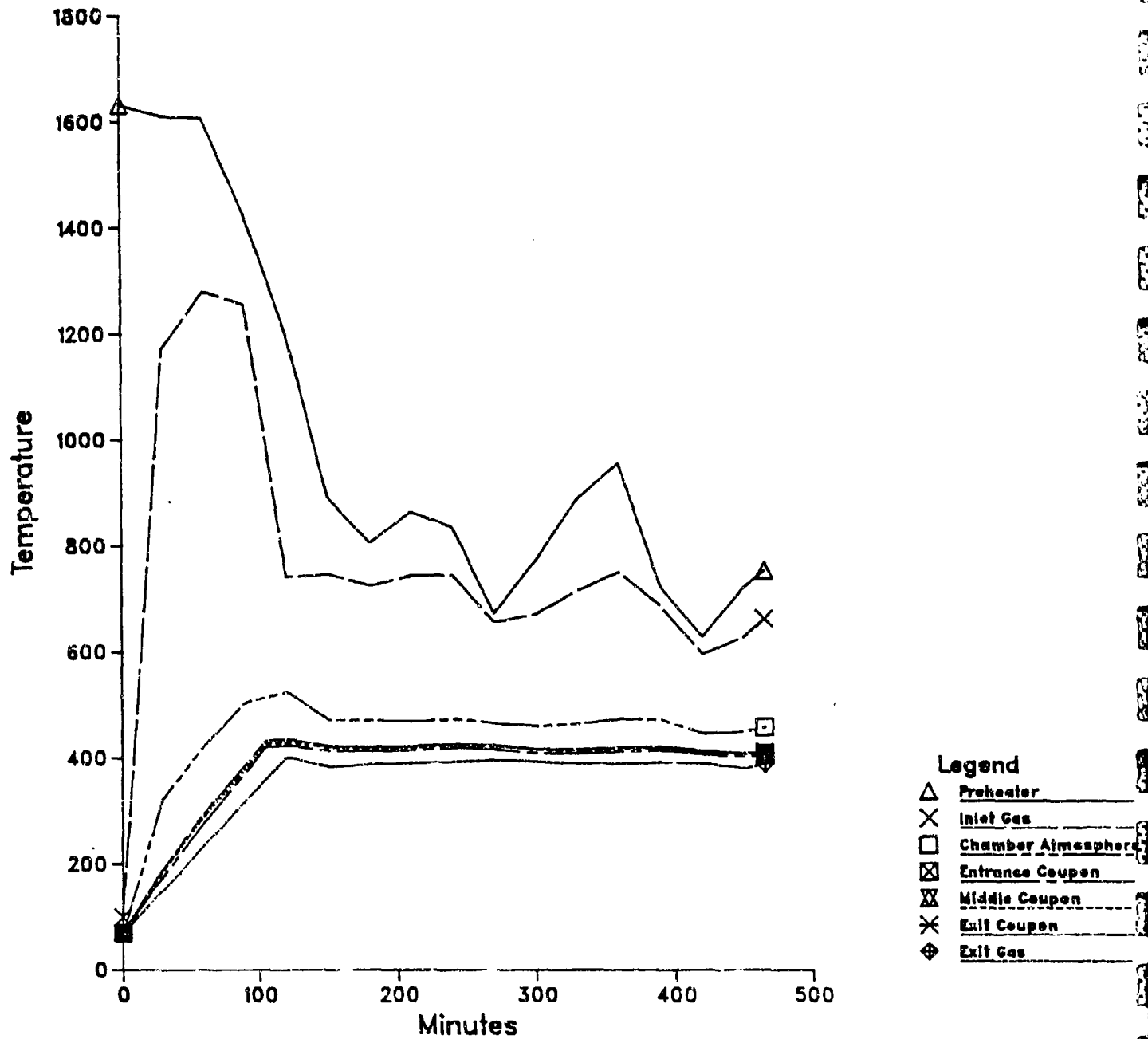
Run # 7 HMX Time–Temperature Profiles

Run # 8 HMX Time–Temperature Profiles

Legend
- △ Preheater
- ✕ Inlet Gas
- ☐ Chamber At—
- ⊠ Entrance Cc.
- ⊠ Middle Couc.
- ✕ Exit Couper
- ◈ Exit Gas

300
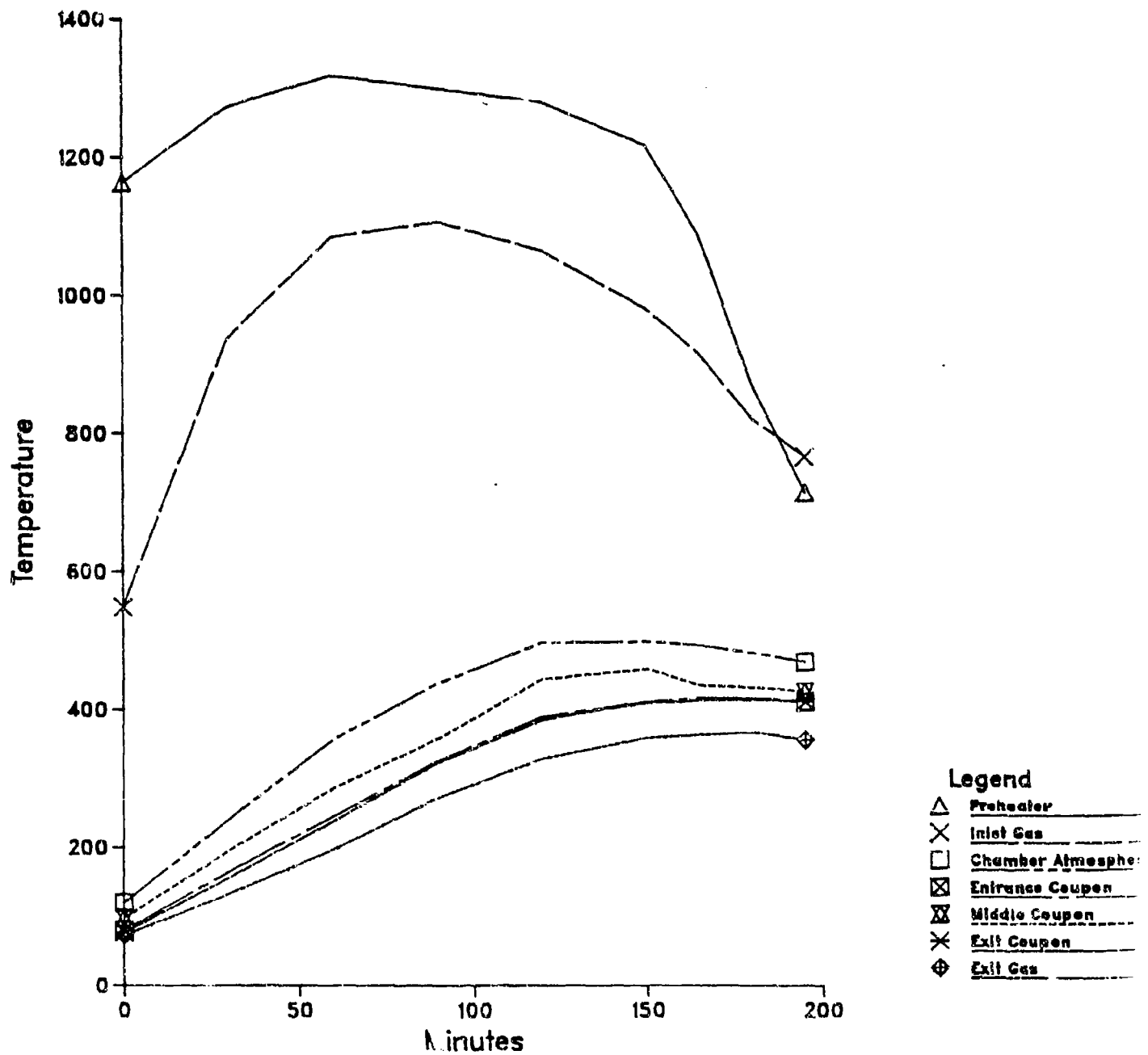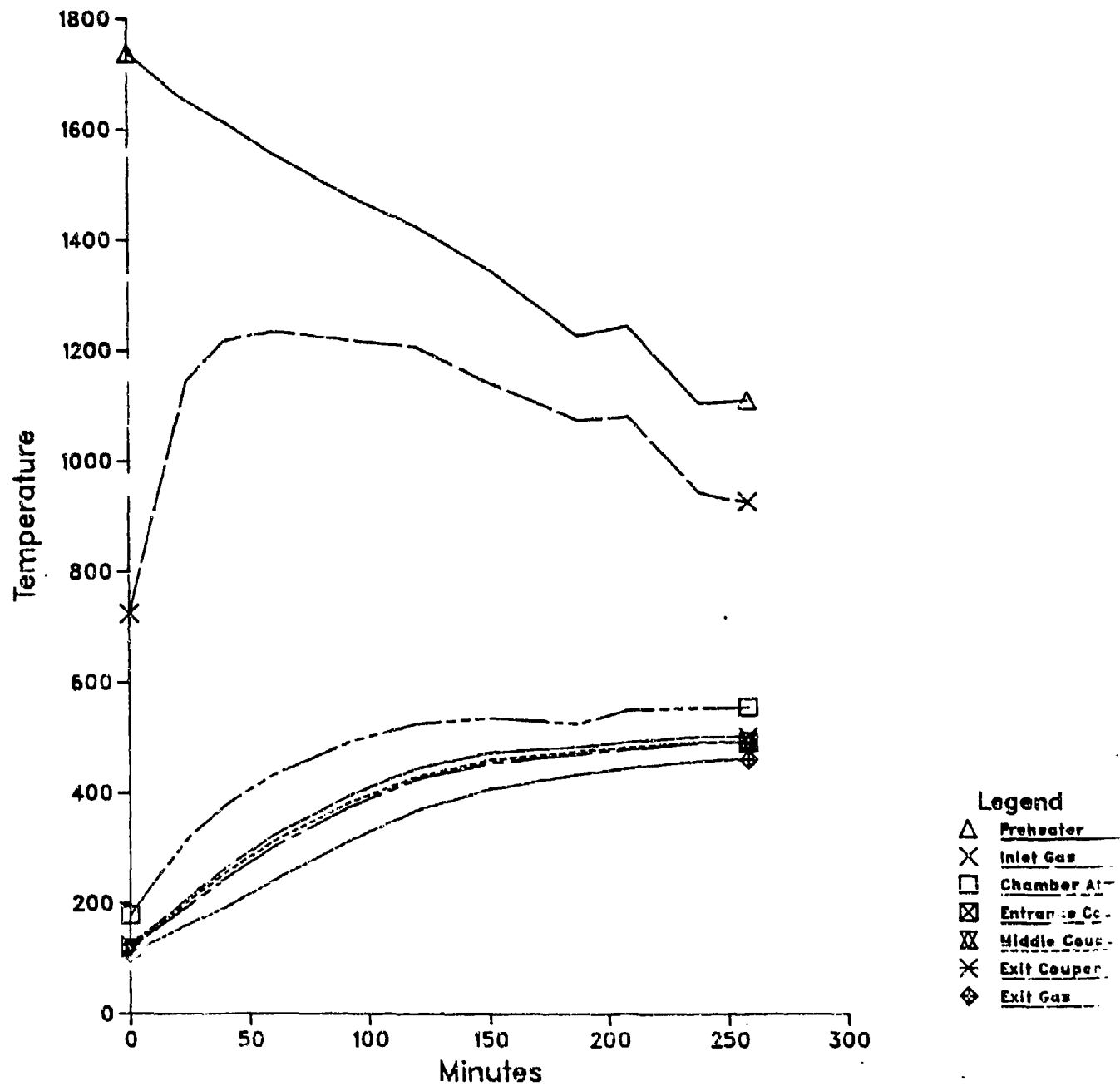
Run # 9   HMX   Time–Temperature Profiles

Run # 10 HMX Time—Temperature Profiles
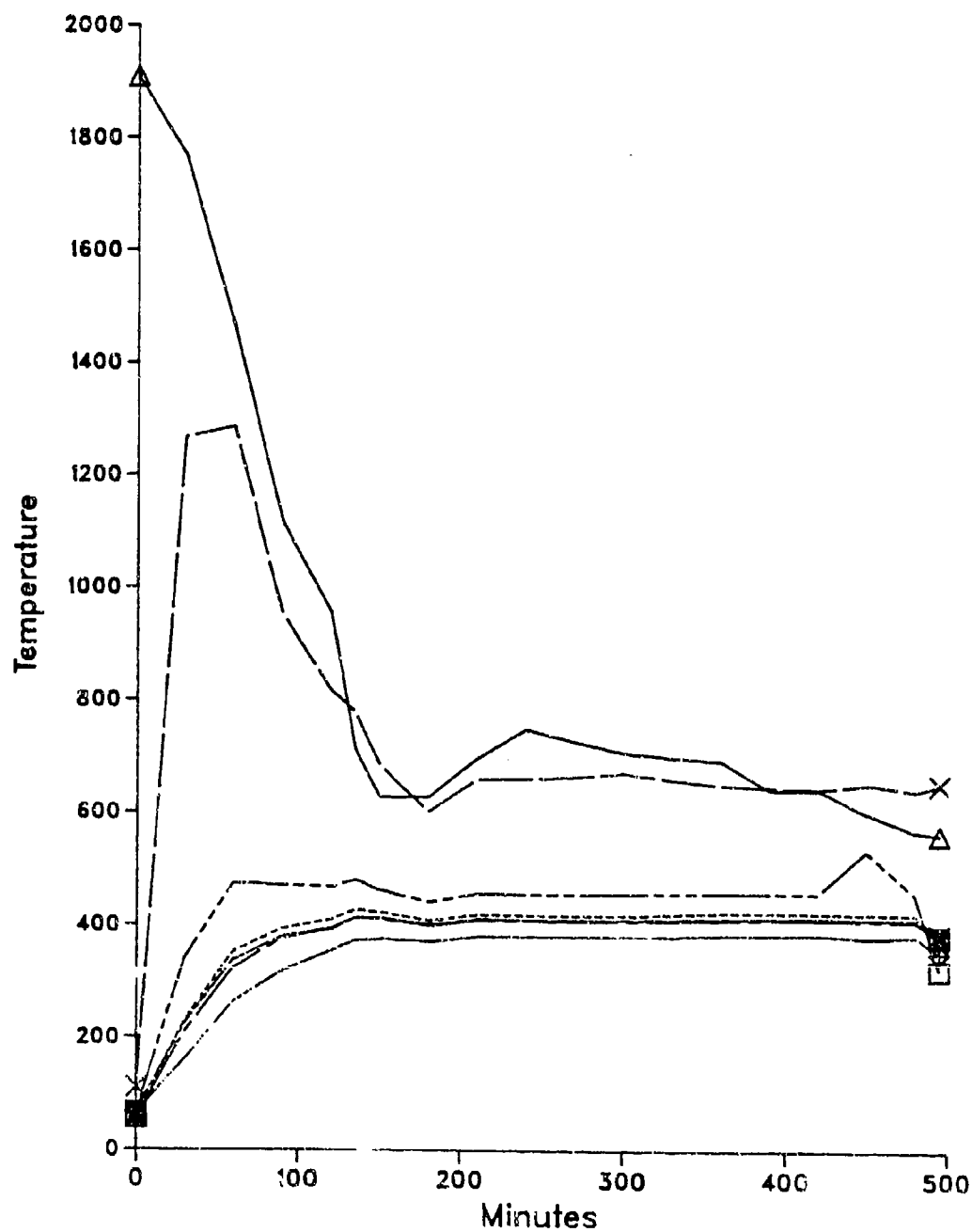
Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmosphere
⊠ Entrance Coupon
⊠ Middle Coupon
✳ Exit Coupon
⬨ Exit Gas

Run # 11 HMX Time-Temperature Profiles
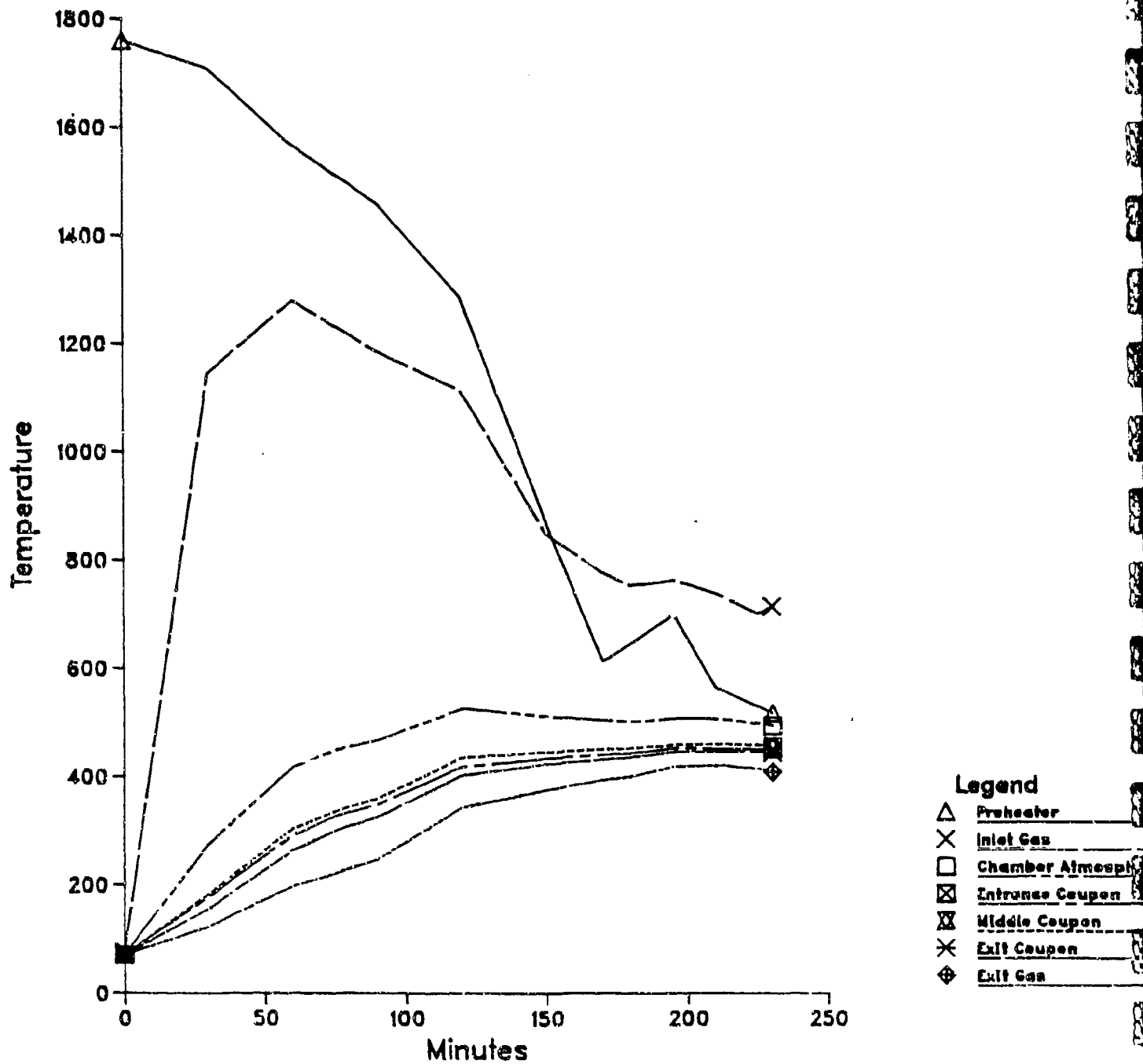
Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmospher
⊠ Entrance Coupon
⊠ Middle Coupon
✳ Exit Coupon
⊕ Ex't Gas
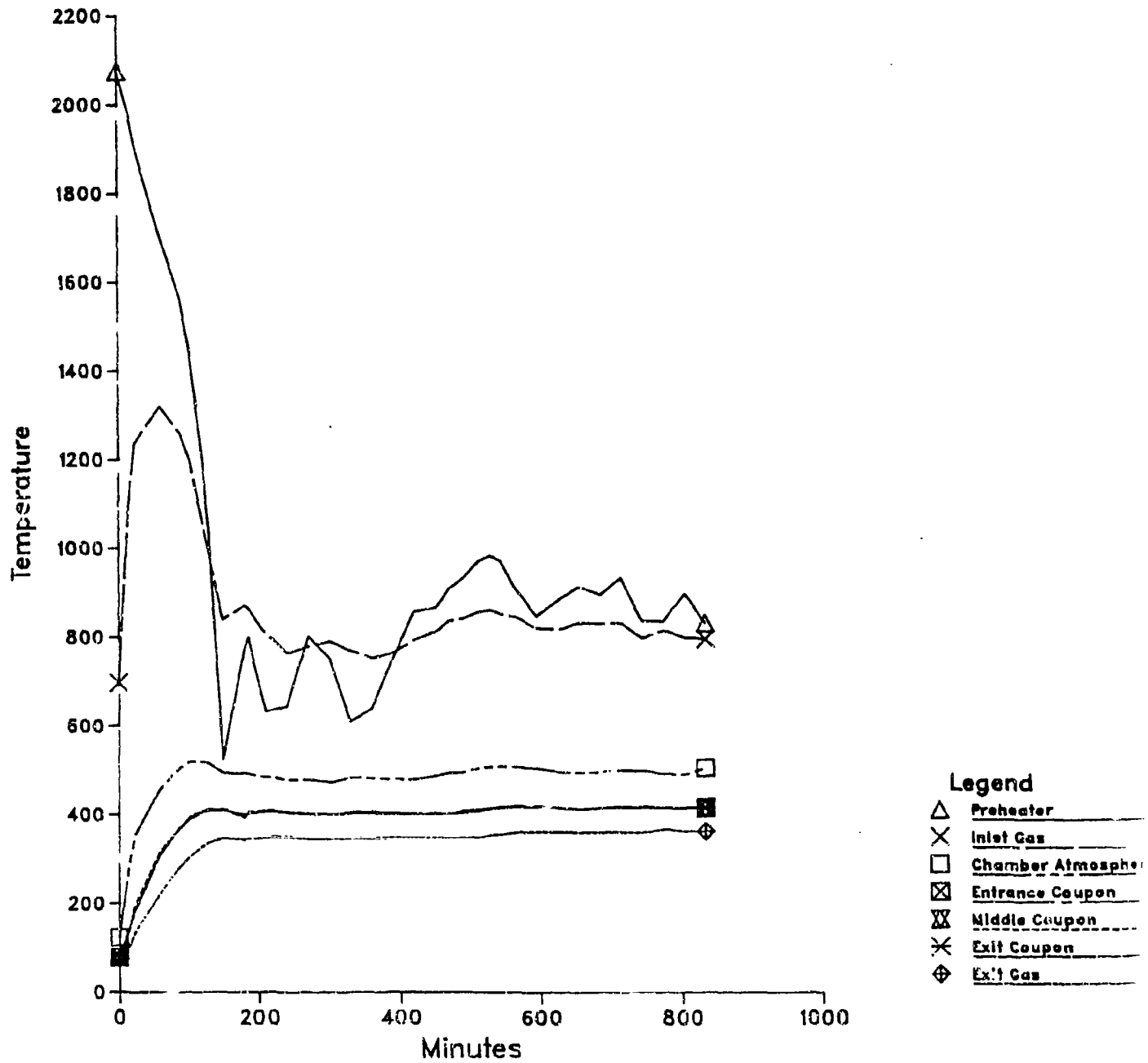
Run # 12 HMX Time—Temperature Profiles
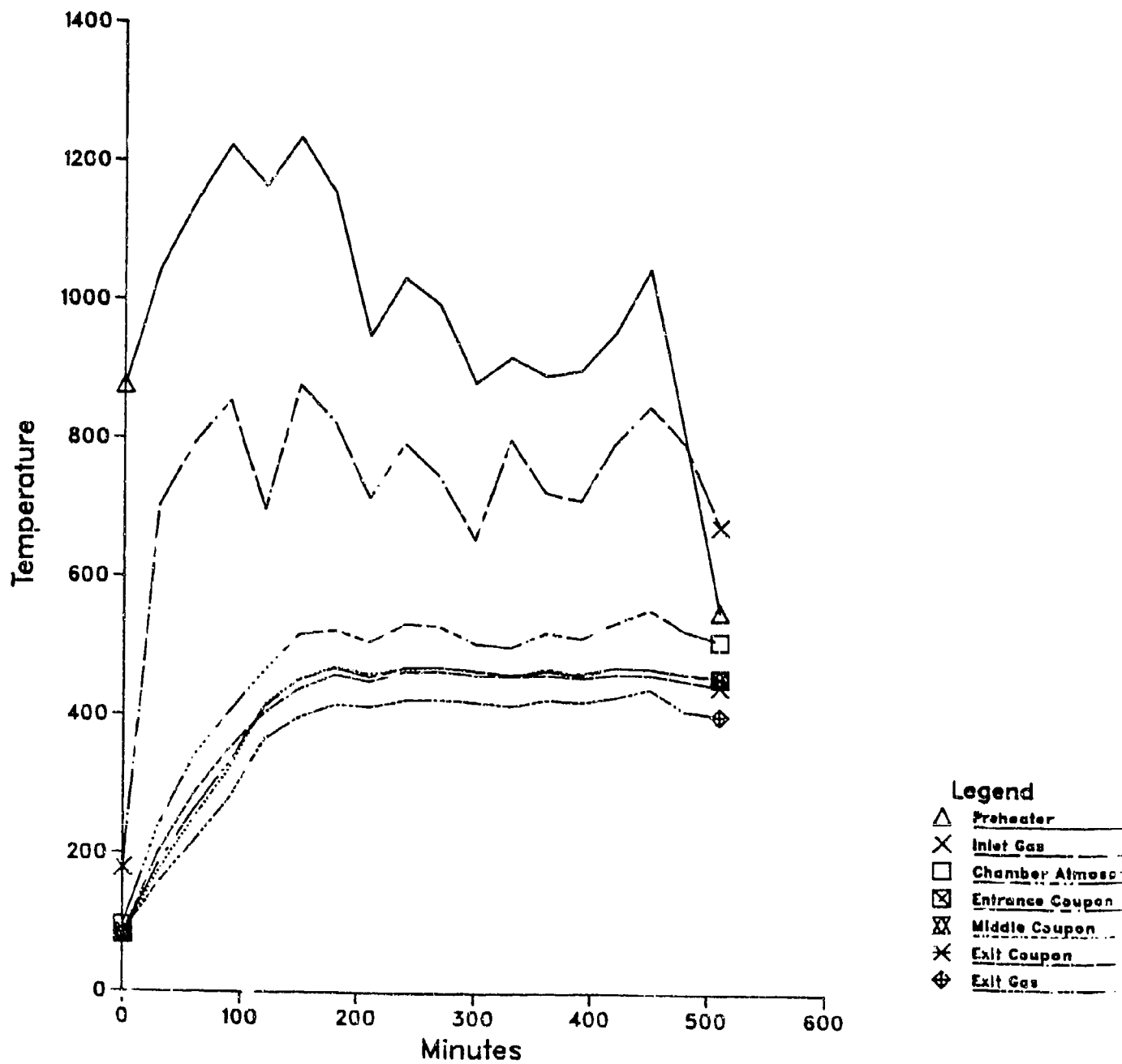
Run # 14 Tetryl Time–Temperature Profiles

Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmosphere
⊠ Entrance Coupon
⊠ Middle Coupon
✳ Exit Coupon
⊕ Exit Gas

305

Run # 15 TNT Time—Temperature Profiles
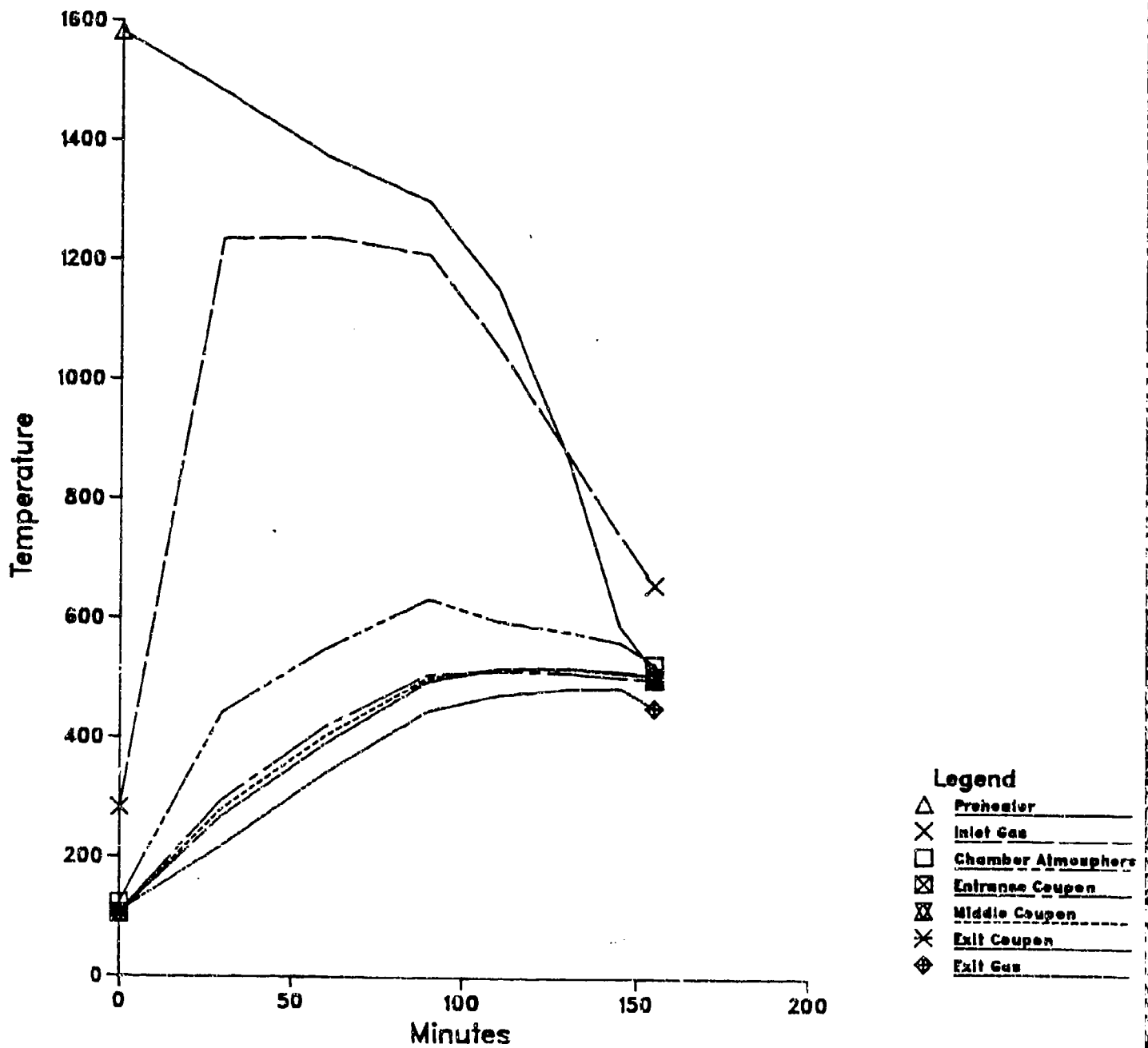
Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmosphere
⊠ Entrance Coupon
⊠ Middle Coupon
✳ Exit Coupon
◈ Exit Gas
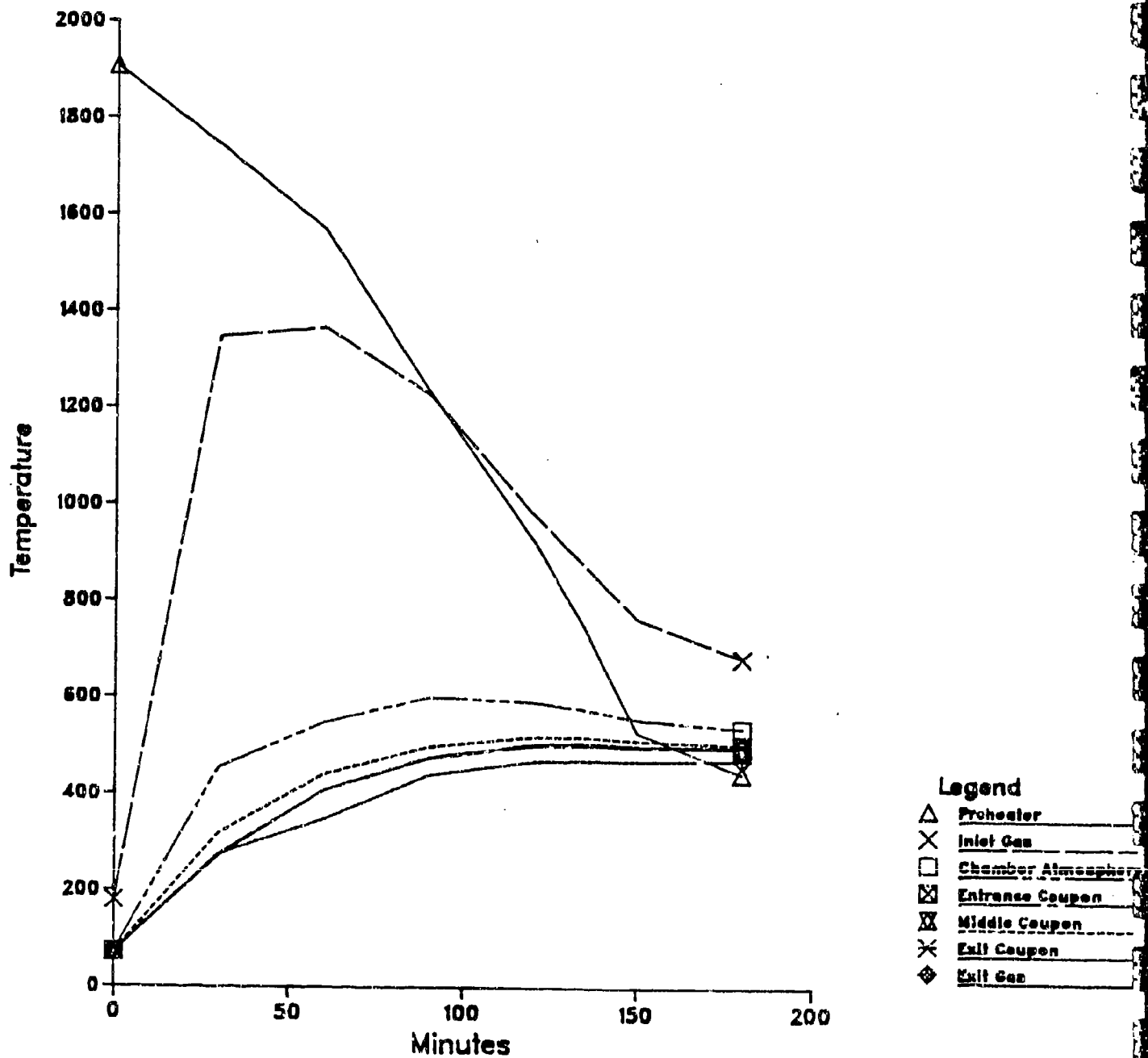
Run # 17 2,6 DNT Time-Temperature Profiles

Run # 18 RDX Time–Temperature Profiles
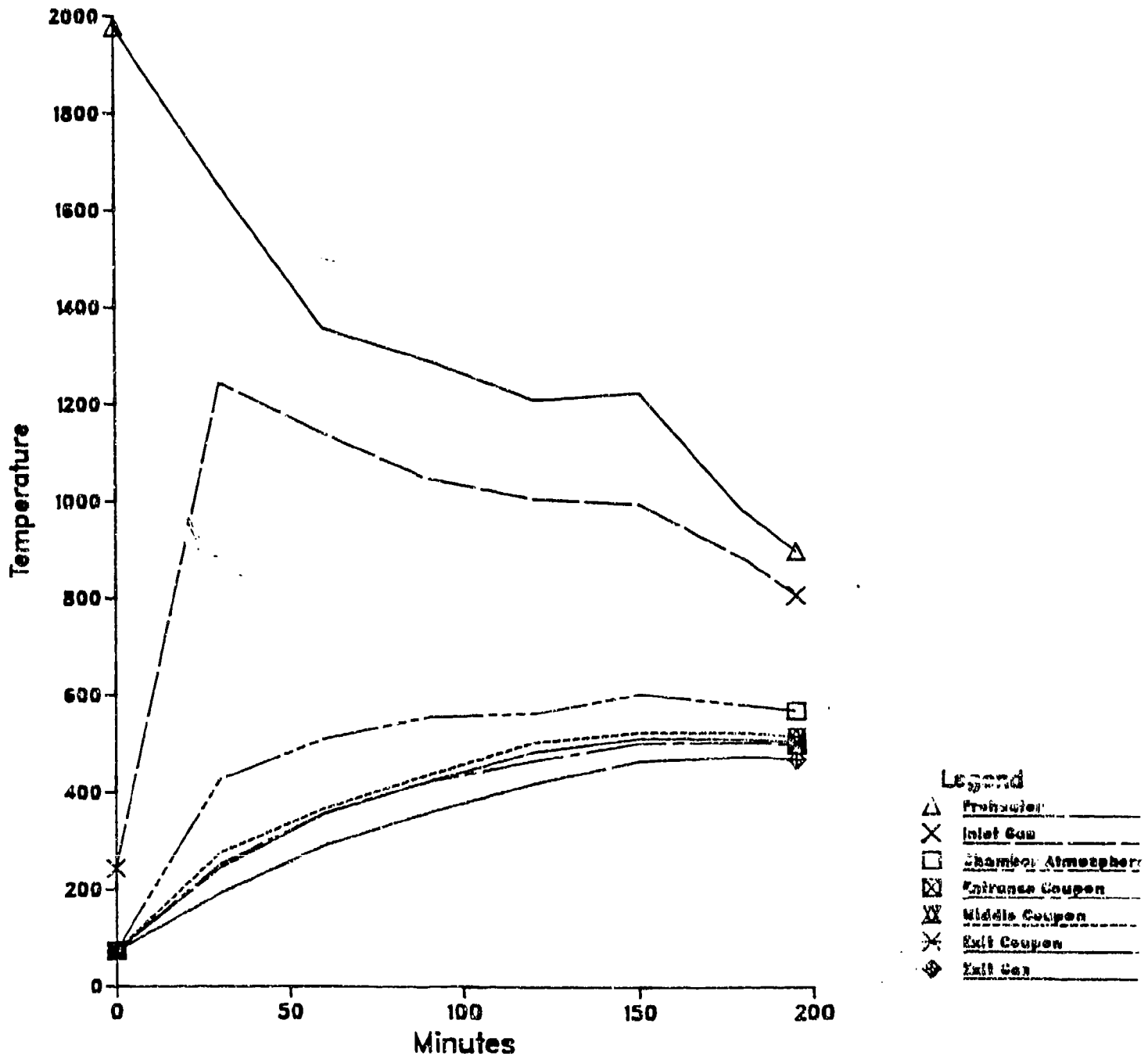
Legend
△ Preheater
✕ Inlet Gas
□ Chamber Atmosphere
⊠ Entrance Coupon
⊠ Middle Coupon
✳ Exit Coupon
⬧ Exit Gas
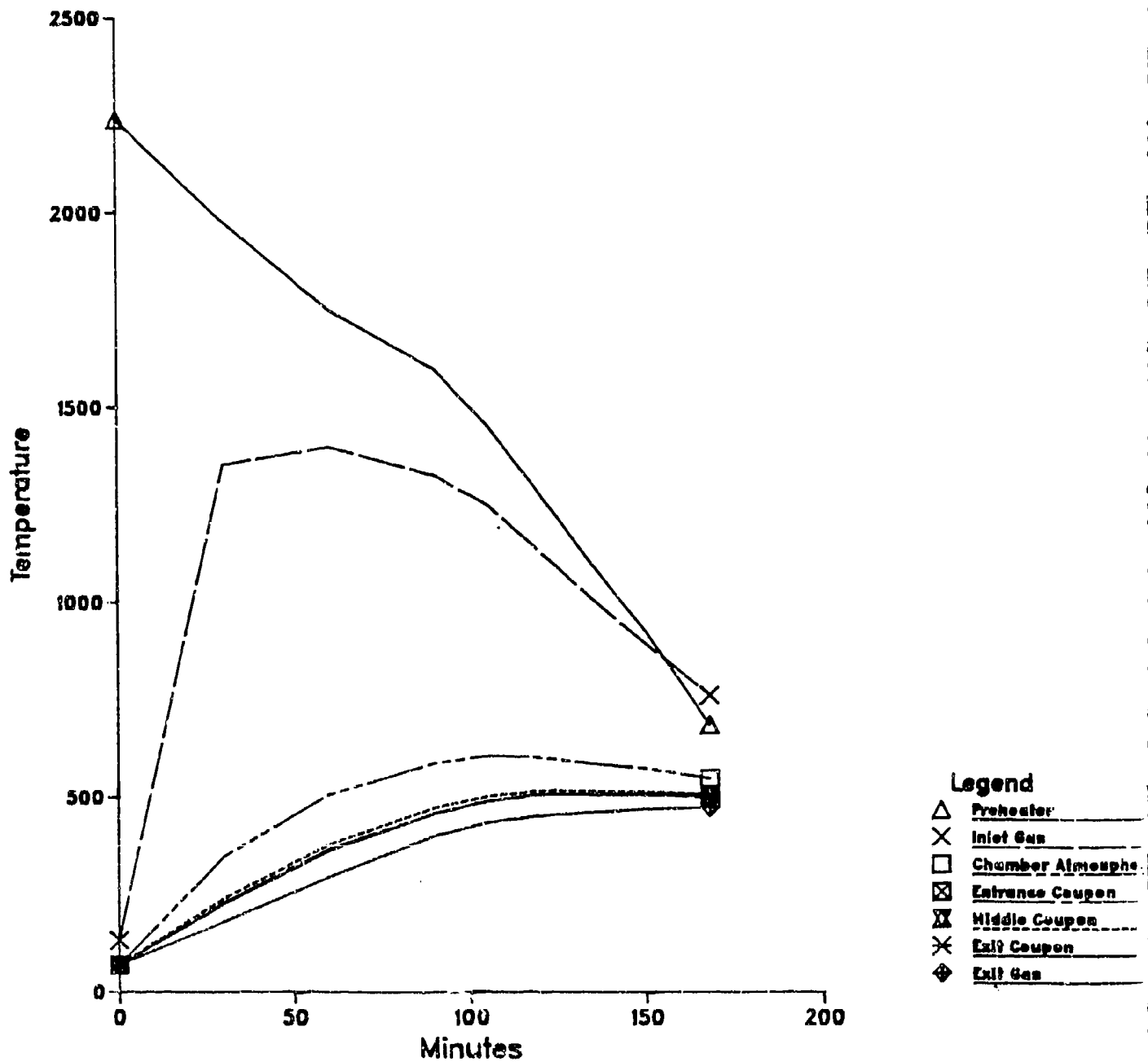
HPLC CHROMATOGRAMS

On the following pages are sets of computer-generated chromatograms and data sheets from the HPLC analysis of the six samples (impinger, trap, sump, and three coupon extracts) typical to these tests. Chromatograms are included for the internal standard containing all six explosives as well. Since inclusion of all the chromatographic data would be too cumbersome, the five runs presented here are from the confirmation tests and are considered typical of all tests.

File:CD400L   EXP 10UG/ML   11:20:59   1/13/1984
E = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

Analytical Standard

HMX   RDX   TETRYL   TNT   2 6 DNT   2 4 DNT
4.52   7.12   10.04   10.73   13.99   16.21

2.0   4.0   6.0   8.0   10.0   12.0   14.0   16.0   18.0   20.0

Max: 250.000 mv   M I N U T E S   X Smx :   250.000
Min: 0.000 mv   X Smn :   0.000

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File: GD400   EXP 10UG/ML   11:20:59   1/13/1984
Mthd: 1310   Inst 8   Chnl 0   Vial #   0 Stnd/Smpl %   100.000

Mthd: 1352   EXP 10UG/ML   15:06:10   1/16/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	0.293	VCV	3.11	1.0000	11.228	1.531
002	0.428	VCB	3.47	1.0000	16.376	1.196
HMX	4.808	BCB	4.52	1.0000	184.008	11.584
RDX	9.828	BCB	7.12	1.0000	376.107	13.682
TETRYL	15.800	BCV	10.04	1.0000	604.683	23.016
TNT	24.791	VCB	10.93	1.0000	948.774	37.499
2 6 DNT	16.597	BCB	13.98	1.0000	635.193	22.800
2 4 DNT	27.454	BCB	16.21	1.0000	1050.683	35.717
	100.000					

Mthd: 1310   Inst 8   Chnl 0   Vial #   0 Stnd/Smpl %   100.000

Area Analysis

File: GD401   EXP 30UG/ML   11:47:54   1/13/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1352   EXP 30UG/ML   15:06:25   1/16/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	0.090	VCV	3.13	1.0000	10.651	1.486
002	0.308	VCV	3.47	1.0000	36.577	3.173
HMX	5.180	VCB	4.51	1.0000	615.246	36.495
RDX	9.800	BCB	7.08	1.0000	1163.985	58.005
TETRYL	15.562	BCV	9.98	1.0000	1848.383	70.677
TNT	24.936	VCB	10.88	1.0000	2961.738	116.917
2 6 DNT	16.570	BCB	13.91	1.0000	1968.141	70.926
2 4 DNT	27.555	BCB	16.13	1.0000	3272.836	111.596
	100.000					

314

File:GD657L   IMPINGER 1/26/84   23:48:28   1/31/1984
FE = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

2 4 DNT
16.03

No. 4

2,4 DNT

Max: 100.000 mv   M I N U T E S   % Smx: 100.000
Min: 0.000 mv   % Smn: 0.000
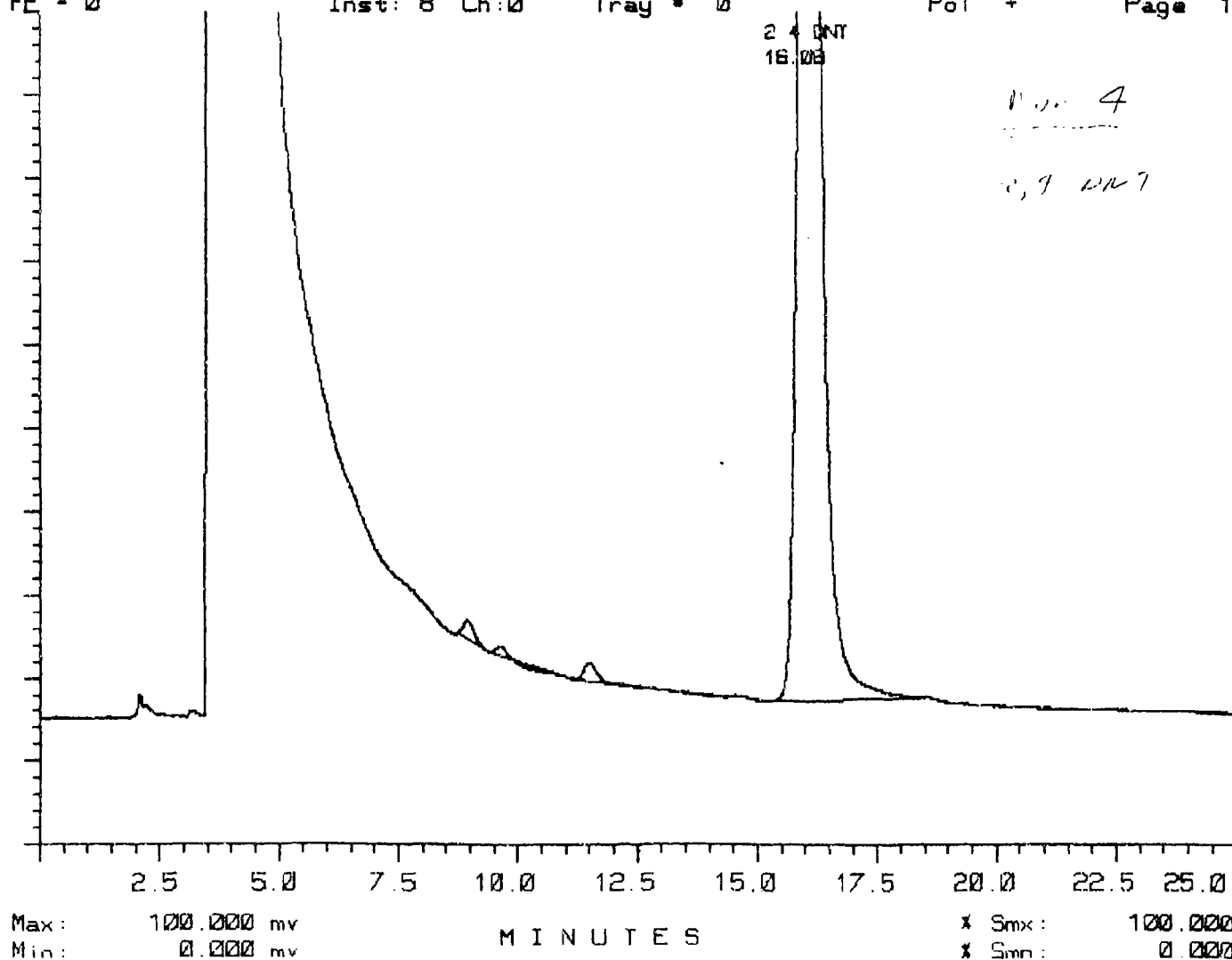
Area Analysis

File: GD657   IMPINGER 1/26/84   23:48:28   1/31/1984
Mthd: 1310   Inst 8   Chnl O   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1390   IMPINGER 1/26/84   09:23:14   2/01/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
RDX			7.10	1.0000		
001	0.554	BCV	8.95	1.0000	33.561	2.272
002	2.706	VCV	9.65	1.0000	163.843	1.049
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
003	0.656	VCB	11.50	1.0000	39.733	2.182
2 6 DNT			14.00	1.0000		
2 4 DNT	96.083	BCB	16.08	1.0000	5816.958	207.200
	100.000					

File:CD65BL
FE = 0

TRAP 1/26/84
Inst: 8 Ch:0   Tray # 0

00:14:30   2/01/1984
Pol +   Page 1

Max: 100.000 mv
Min: 0.000 mv

2.5   5.0   7.5   10.0   12.5   15.0   17.5   20.0   22.5   25.0

M I N U T E S

x Smx: 100.000
x Smn: 0.000

317

Area Analysis

File: GD658   TRAP 1/26/84   00:14:30   2/01/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1390   TRAP 1/26/84   09:23:24   2/01/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
001	100.000	BCB	11.47	1.0000	40.410	1.978
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000		
	100.000					

File:GD659L

SUMP 1/26/84
Inst: 8 Ch:0   Tray # 0

00:40:33   2/01/1984
Pol +   Page 1

FE = 0

2 4 DNT
16.08

2.5	5.0	7.5	10.0	12.5	15.0	17.5	20.0	22.5	25.0

Max: 100.000 mv
Min: 0.000 mv

M I N U T E S

% Smx: 100.000
% Smn: 0.000
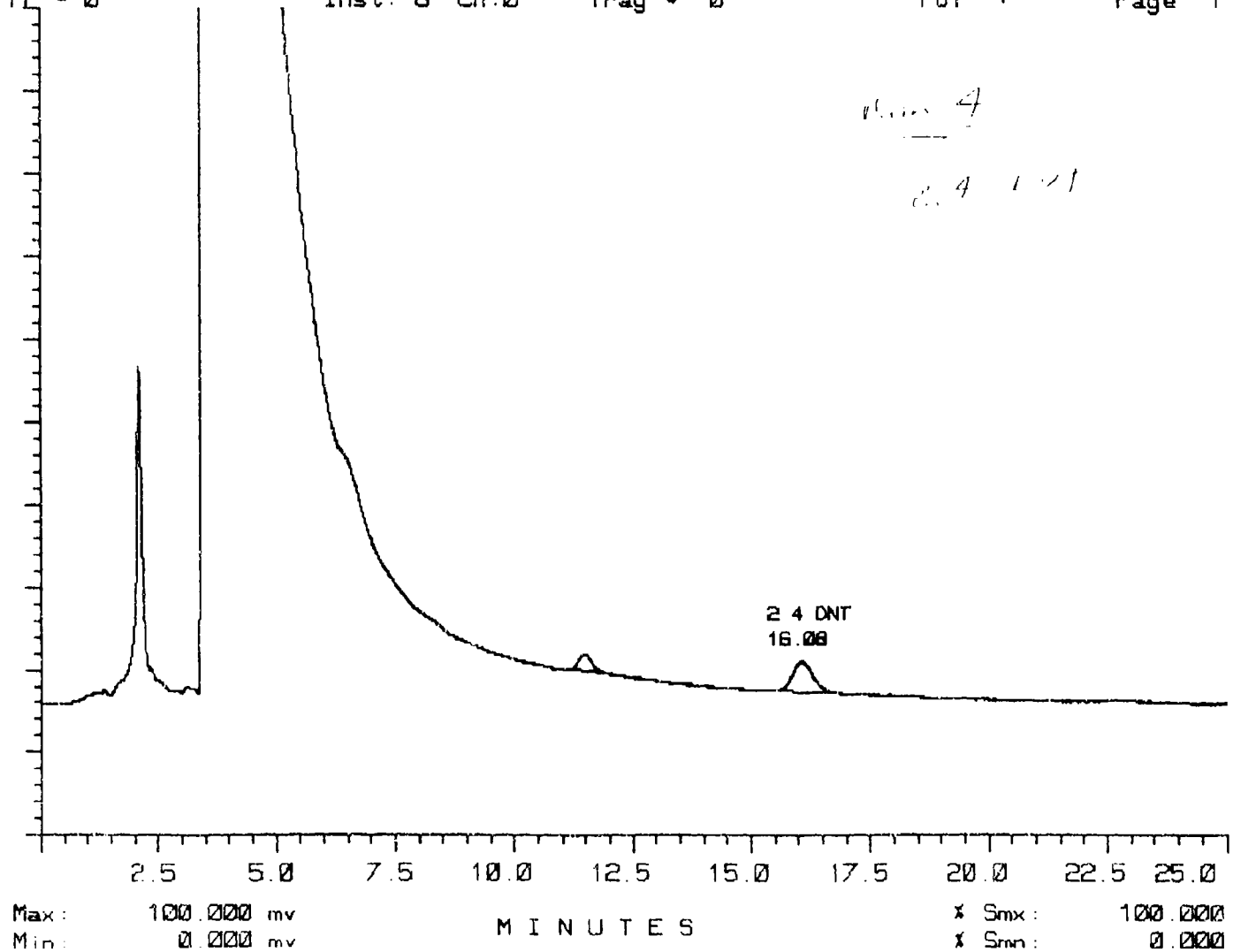
Area Analysis

File: GD659   SUMP 1/26/84   00:40:33   2/01/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1390   SUMP 1/26/84   09:23:33   2/01/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
001	25.387	BCB	11.50	1.0000	38.496	2.065
2 6 DNT			14.00	1.0000		
2 4 DNT	74.613	BCB	16.08	1.0000	113.140	3.714
	100.000					
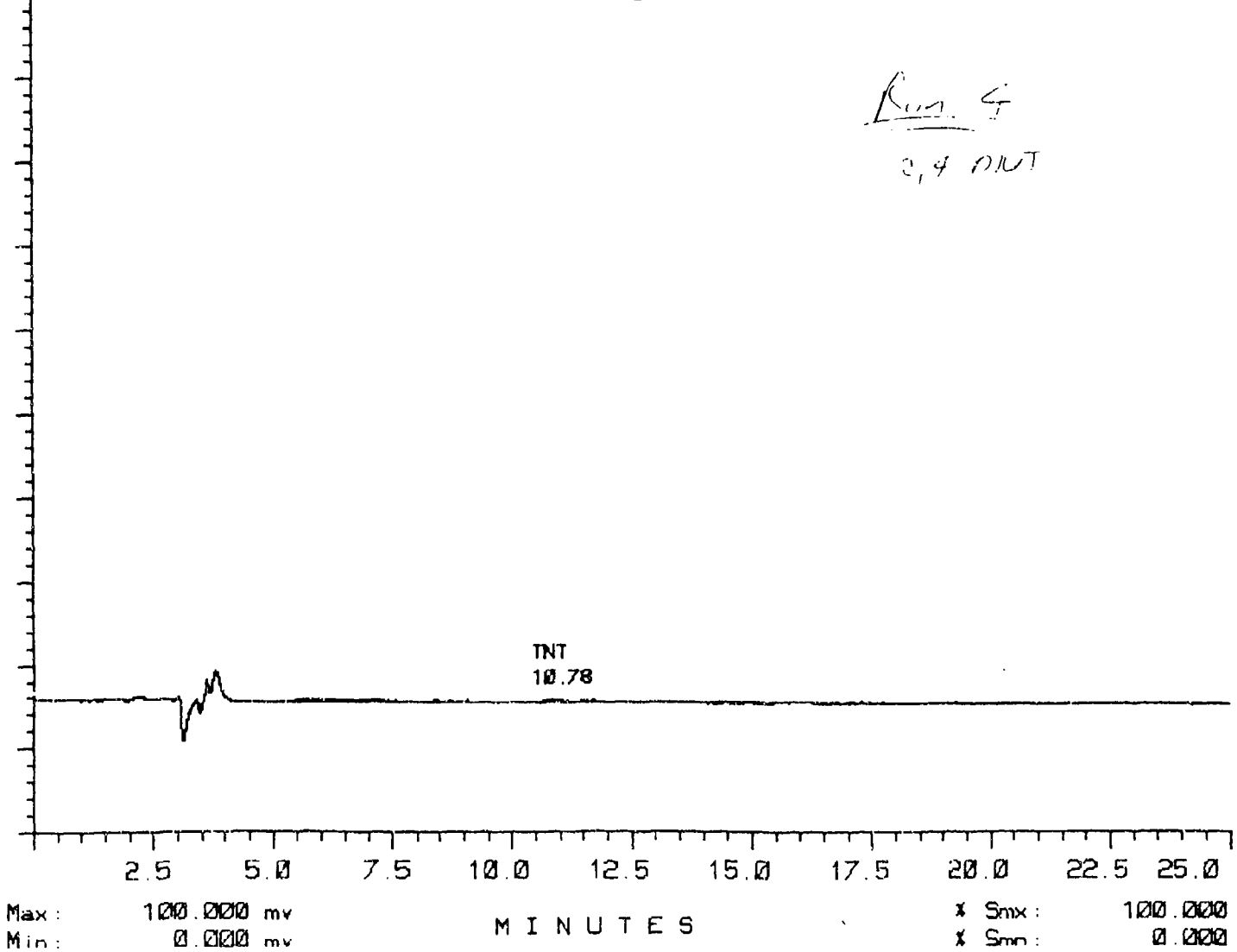
File:GD654L   COUP 1 1/26/84   22:30:22   1/31/1984
E = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

Run 4

3,4 DNT

TNT
10.78

```
  |        |        |        |        |        |        |        |        |        |        |
  2.5      5.0      7.5      10.0     12.5     15.0     17.5     20.0     22.5  25.0
```

Max: 100.000 mv   M I N U T E S   X Smx: 100.000
Min: 0.000 mv   X Smn: 0.000

321

Area Analysis

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
001	83.062	BCB	5.85	1.0000	15.064	0.224
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT	16.938	BCB	10.78	1.0000	3.072	0.243
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000 ·		
	100.000					

File:CD655L   COUP 2 1/26/84   22:56:23   1/31/1984
FE = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

Run 4

: 4 OUT

| 2.5 | 5.0 | 7.5 | 10.0 | 12.5 | 15.0 | 17.5 | 20.0 | 22.5 | 25.0 |

Max: 100.000 mv   M I N U T E S   X Smx :   100.000
Min: 0.000 mv   X Smn :   0.000
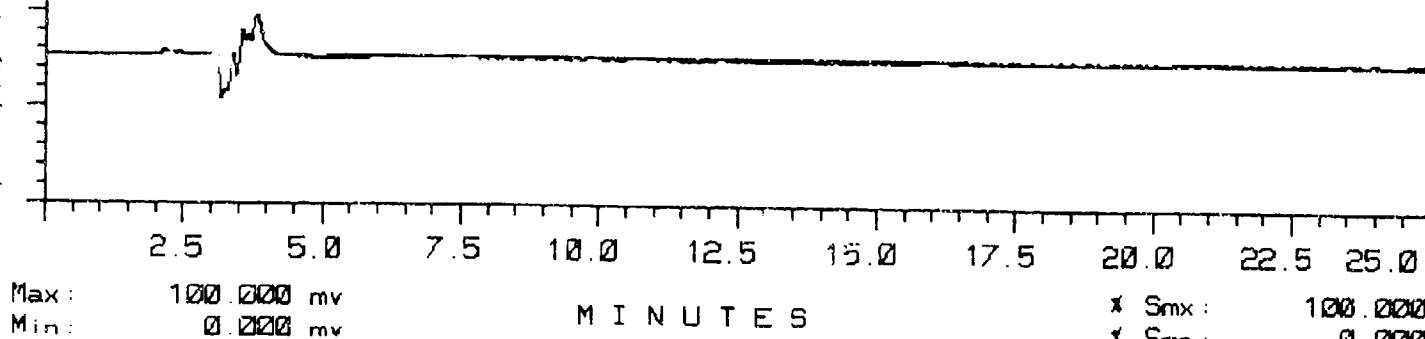
Area Analysis

File: GD655   COUP 2 1/26/84   22:56:23   1/31/1984
Mthd: 1310   Inst 8   Chnl 0   Vial #   0 Stnd/Smpl % 100.000

Mthd: 1390   COUP 2 1/26/84   09:22:55   2/01/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
001	100.000	BCB	5.76	1.0000	12.204	0.264
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000		
	100.000					
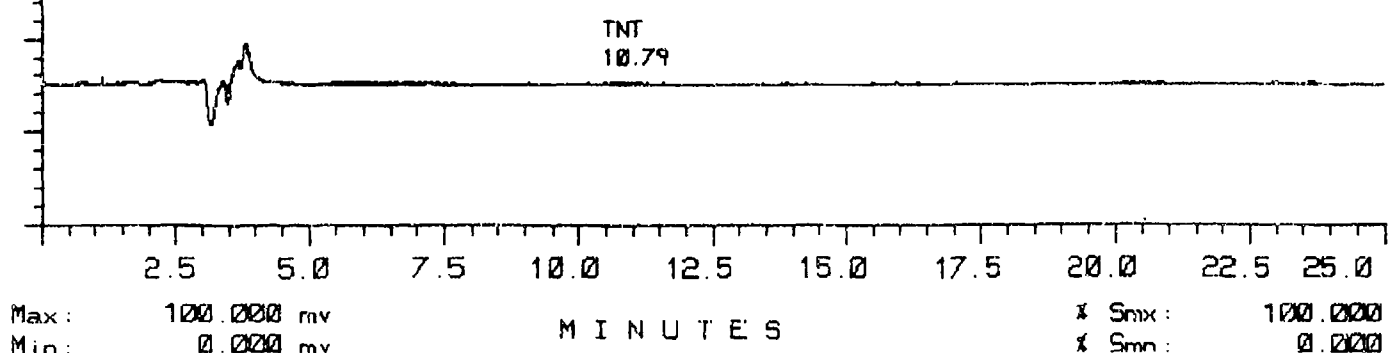
Area Analysis

File: GD656   COUP 3 1/26/84   23:22:26   1/31/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1390   COUP 3 1/26/84   09:23:05   2/01/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
001	44.465	BCB	5.70	1.0000	3.868	0.160
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT	55.535	BCB	10.79	1.0000	4.831	0.234
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000		
	100.000					

File:GD920L   IMPINGER 2/15/84   13:10:08 2/17/1984
FE = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

Run 8


```
        1 0    2 0    3 0    4 0    5 0    6 0    7 0    8.0    9 0  10.0
Max   200 000 mv                M I N U T E S              $ Smx     200.000
Min     5 000 mv                                           $ Smn       5 000
```
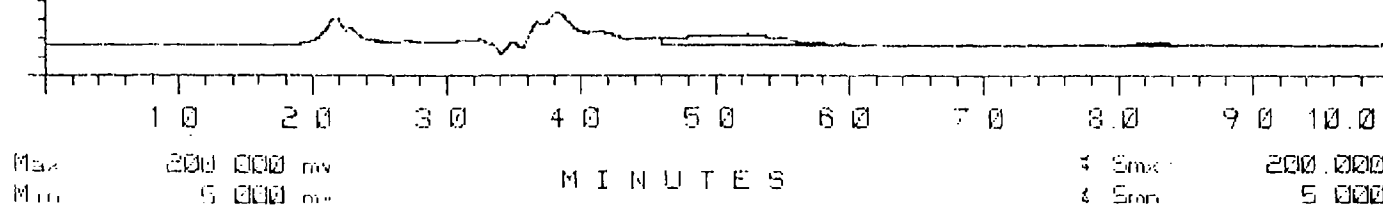
File: GD920   IMPINGER 2/15/84   13:10:08   2/17/1984
Mthd: 1310   Inst 8   Chnl O   Vial #   O Stnd/Smpl % 100.000
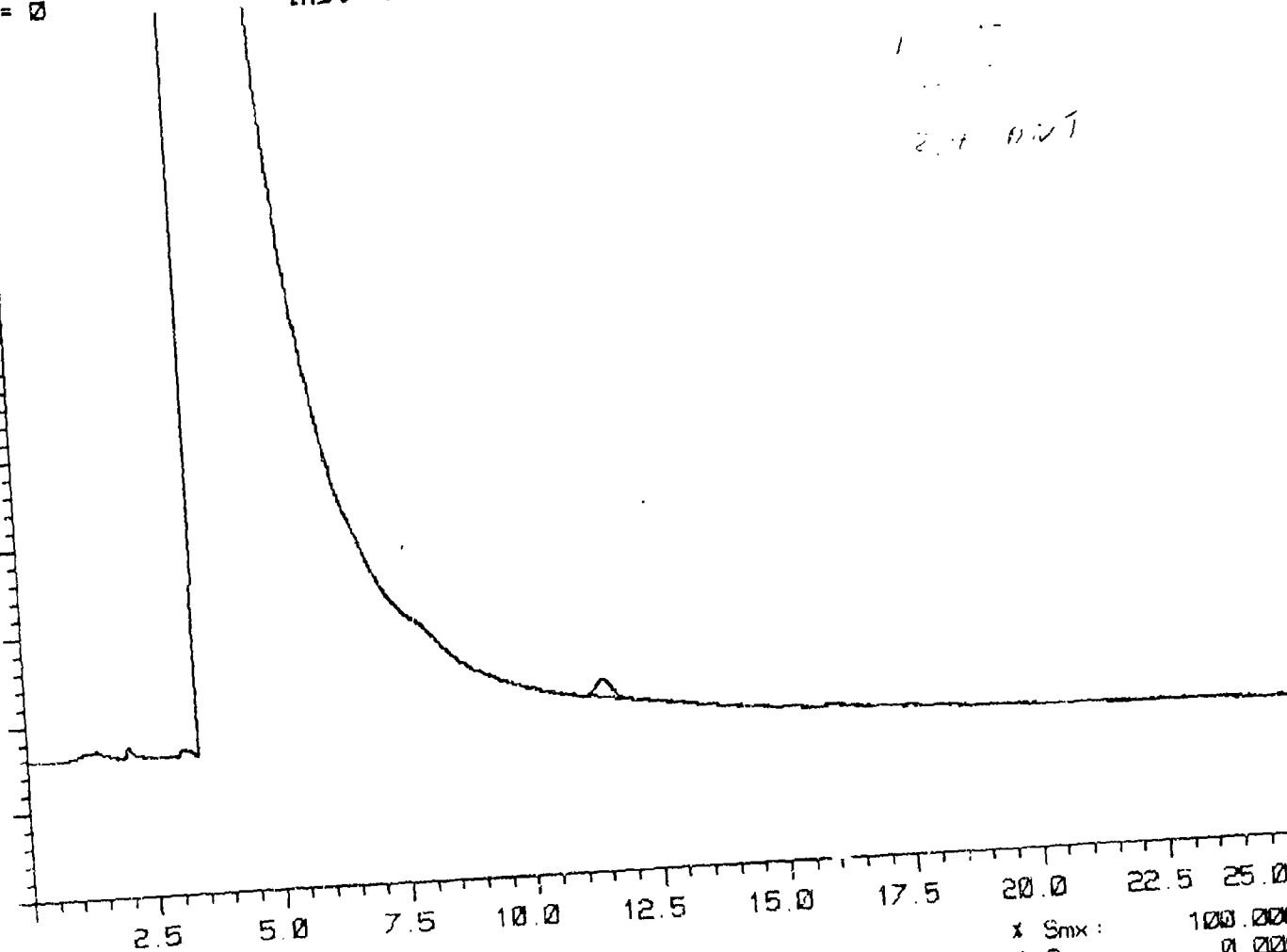
Mthd: 1390   IMPINGER 2/15/84   13:59:53   2/17/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
001	94.852	VCB	5.24	1.0000	104.135	2.069
RDX			7.10	1.0000		
002	5.148	BCB	8.29	1.0000	5.651	0.344
TETRYL			9.85	1.0000		
TNT			10.70	1.0000		
2 6 DNT			13.60	1.0000		
2 4 DNT			15.70	1.0000		
	100.000					

File:CD921L   TRAP 2/15/84   13:21:15   2/17/1984
FE = 0   Inst: 8 Ch.0   Tray # 0   Pol +   Page 1

Run 8

    1.0   2.0   3.0   4.0   5.0   6.0   7.0   8.0   9.0   10.0
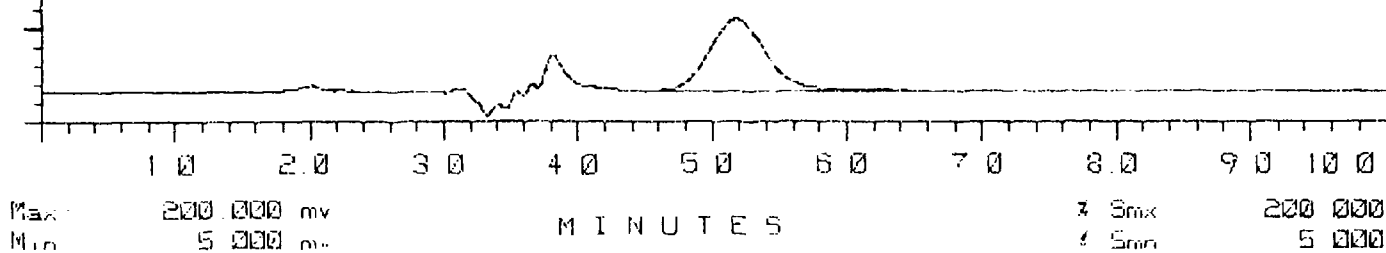
                                M I N U T E S

Max   200.000 mv   % Smx   200 000
Min   5.000 mv   % Smn   5 000

Area Analysis

File: GD921   TRAP 2/15/84   13:21:15   2/17/1984
Mthd: 1310   Inst 8   Chnl O   Vial # O Stnd/Smpl % 100.000

Mthd: 1390   TRAP 2/15/84   13:59.59   2/17/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
001	100.000	VCB	5.18	1.0000	437.316	14.970
RDX			7.10	1.0000		
TETRYL			9.85	1.0000		
TNT			10.70	1.0000		
2 6 DNT			13.60	1.0000		
2 4 DNT			15.70	1.0000		
	100.000					

Run 8

HMX
4 55

1 0   2 0   3 0   4.0   5 0   6.0   7 0   8 0   9 0   10.0

M I N U T E S

Max   200 000 mv   3 Smx   200 000
Min   5 000 mv   1 Smn   5 000

File:GD919   SUMP 2/15/84   12:59:03   2/17/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1390   SUMP 2/15/84   13:59:47   2/17/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	90.051	VCV	4.14	1.0000	588.088	37.546
HMX	9.949	VCB	4.55	1.0000	64.970	4.535
RDX			7.10	1.0000		
TETRYL			9.85	1.0000		
TNT			10.70	1.0000		
2 6 DNT			13.60	1.0000		
2 4 DNT			15.70	1.0000		
	100.000					

File GD918L RIGHT COUP HMX 2/15/84   12:47:55 2/17/1984
FE = 0   Inst: 8 Ch:0 Tray # 0   Pol + Page 1

Run 8

| | 1 0 | 2 0 | 3 0 | 4 0 | 5 0 | 6 0 | 7 0 | 8 0 | 9 0 | 10 0 |

M I N U T E S

Max. 200.000 mv   X Smx 200.000
Min.   5.000 mv   X Smn   5.000
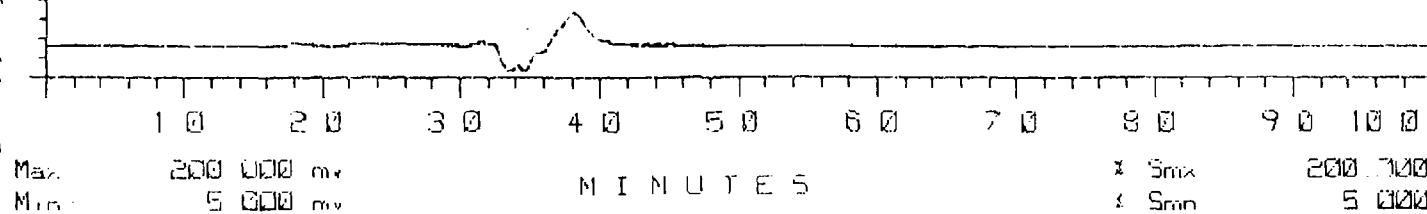
File: GD918   RIGHT COUP HMX 2/15/84   12:47:55   2/17/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1390   RIGHT COUP HMX 2/15/84   13:59:38   2/17/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
001	43.839	BCV	5.83	1.0000	2.635	0.163
002	56.161	VCB	6.13	1.0000	3.375	0.176
RDX			7.10	1.0000		
TETRYL			9.85	1.0000		
TNT			10.70	1.0000		
2 6 DNT			13.60	1.0000		
2 4 DNT			15.70	1.0000		
	100.000					

File:GD417L.   MIDDLE COUP HMX 2/15/84   12:36.49   2/17/1984
FE = 0   Inst. 8 Ch:0   Tray # 0   Pol +   Page 1

Run 8

| | 1.0 | 2.0 | 3.0 | 4.0 | 5.0 | 6.0 | 7.0 | 8.0 | 9.0 | 10.0 |

M I N U T E S

Max   200.000 mv   x Smax   200.000
Min   5.000 mv   x Smin   5.000
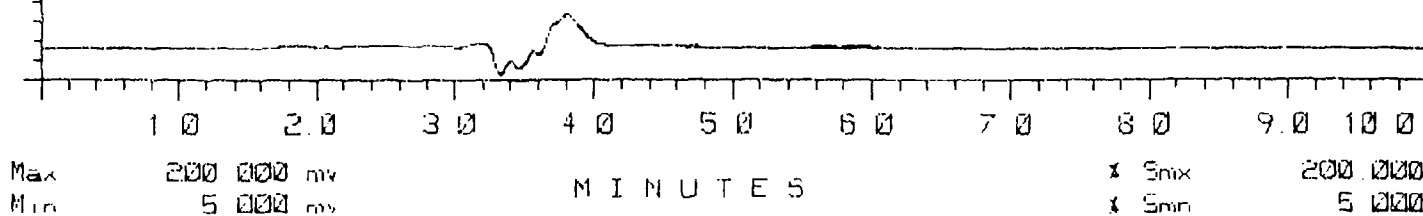
Area Analysis

File: GD917 MIDDLE COUP HMX 2/15/84   12:36:49   2/17/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 1390 MIDDLE COUP HMX 2/15/84   13:59:31   2/17/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
001	100.000	BCB	5.65	1.0000	3.154	0.166
RDX			7.10	1.0000		
TETF 'L			9.85	1.0000		
TNT			10.70	1.0000		
2 6 DNT			13.60	1.0000		
2 4 DNT			15.70	1.0000		
	100.000					

Run 8

1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0
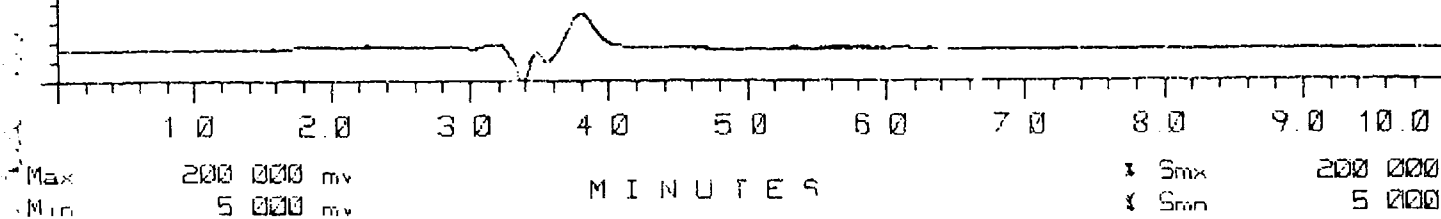
M I N U T E S

Max   200 000 mv   % Smx   200 000
Min   5 000 mv   % Smn   5 000

Area Analysis

File:GD916   LEFT COUP HMX 2/15/84   12:25:45   2/17/1984
Mthd: 1310   Inst 8   Chnl 0   Vial #   0 Stnd/Smpl % 100.000

Mthd: 1390   LEFT COUP HMX 2/15/84   13:59:15   2/17/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX			4.50	1.0000		
RDX			7.10	1.0000		
TETRYL			9.85	1.0000		
TNT			10.70	1.0000		
2 6 DNT			13.60	1.0000		
2 4 DNT			15.70	1.0000		

0.000
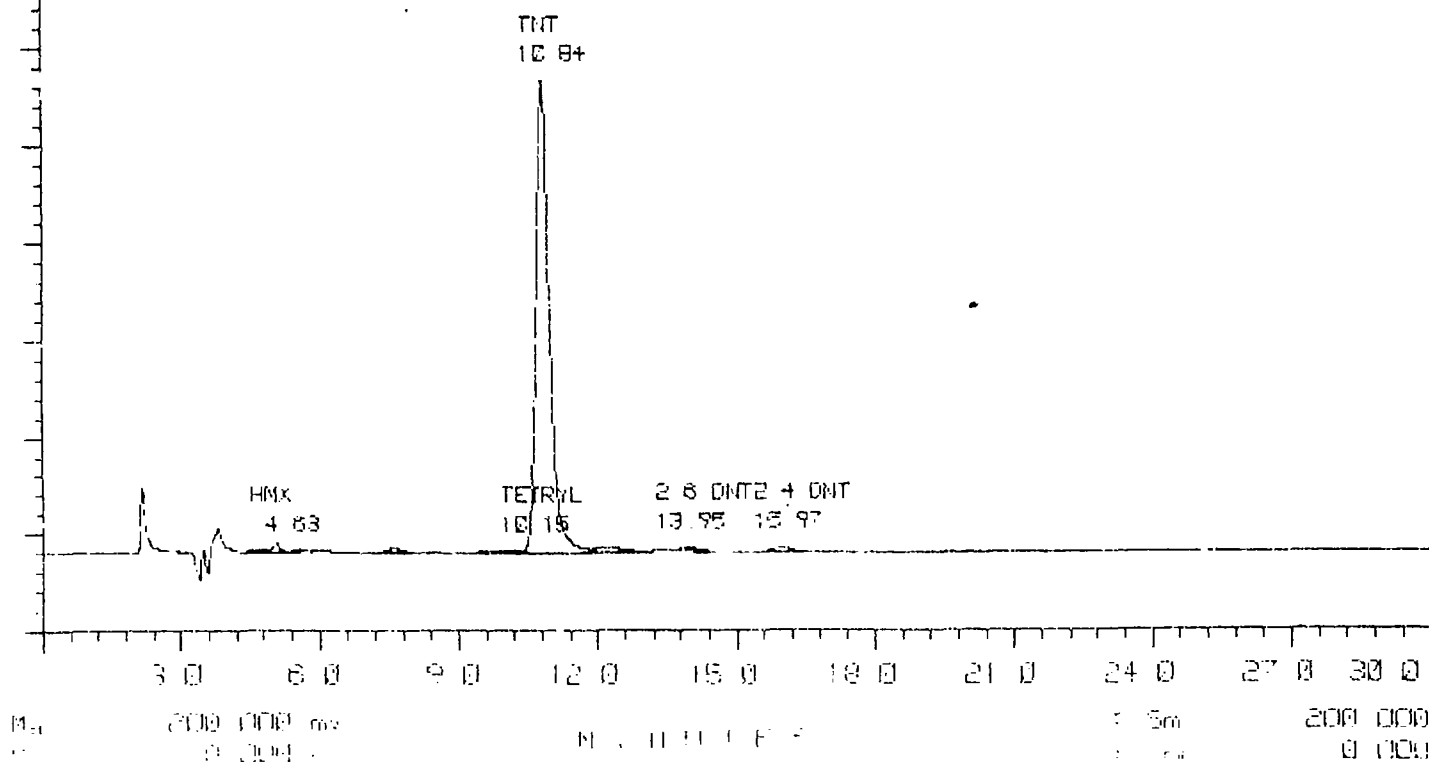
Area Analysis

File: GE227   09324-50-7   02:48:21   24/02/1984
Mthd. 1310   Inst 8   Chnl O   Vial # O Stnd/Smpl % 100.000

Mthd 1391   09324-50-7   10:49:47   23/02/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	0.049	BCV	4.49	1.0000	1.014	0.215
HMX	0.163	VCV	4.63	1.0000	3.399	0.347
002	0.769	VCB	5.08	1.0000	16.063	0.829
003	0.200	BCB	5.62	1.0000	4.178	0.493
RDX			7.10	1.0000		
004	0.714	BCB	7.59	1.0000	14.906	0.922
005	0.348	BCV	9.66	1.0000	7.260	0.370
TETRYL	0.492	VCV	10.15	1.0000	10.275	0.572
TNT	92.280	VCV	10.84	1.0000	1927.857	77.243
006	1.839	VCB	12.29	1.0000	38.426	1.157
2 6 DNT	1.647	BCB	13.95	1.0000	34.405	1.059
2 4 DNT	1.500	BCB	15.97	1.0000	31.346	1.240
	100.000					

Run 15
TNT
Trap

H
4 67

TNT
10.61

3 0   6 0   9 0   12 0   15 0   18 0   21 0   24 0   27 0 30 0

Mx   200 000 ns   200 000
                                                              0 000

Area Analysis

File: GE228   39324-50-8   03:19:26   3/22/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl %   100.000

Mthd: 1391   39324-50-8   10.40:51   3/22/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX	1.621	BCV	4.67	1.0000	0.940	0.130
001	0.642	VCT	4.78	1.0000	0.373	0.091
002	0.435	VCT	4.93	1.0000	0.252	0.096
003	0.818	VCV	5.13	1.0000	0.475	0.129
004	0.961	VCV	5.32	1.0000	0.557	0.082
005	16.709	BCB	5.84	1.0000	9.689	0.244
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT	77.365	BCB	10.81	1.0000	44.861	1.750
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000		
006	1.449	BCB	23.35	1.0000	0.840	0.061
	100.000					

File G2226L   39324-50-6   02 17 19   3/22/1984
FE = 0   Inst 8 Ch.0   Tray # 0   Pol +   Page 1

Run 15
TNT
Sump

HMX
4 76

                    3 0   6 0   9 0   12 0   15 0   18 0   21 0   24 0   27 0   30 0

Ma   .700 000 rc   5 50   200 000
            17 15 %   6 1 4 11 1 1 1   7   8 001

Area Analysis

File: GE226   39324-50-6   02:17:19   3/27/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000
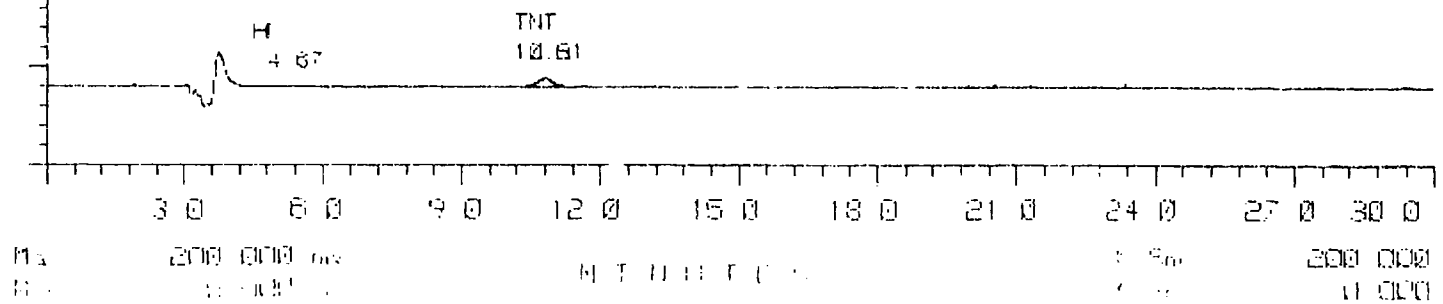
Mthd. 1591   39324-50-6   10:48:34   3/22/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX	4.246	VCB	4.76	1.0000	0.728	0.138
001	0.495	BCV	5.06	1.0000	0.084	0.070
002	95.259	BCB	5.83	1.0000	16.215	3.327
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000		
	100.000					

Run 15   TNT

Left Copen


```
                    HPLC
                    4 39
```

3.0   6.0   9.0   12.0   15.0   18.0   21.0   24.0   27.0   30.0

MINUTES
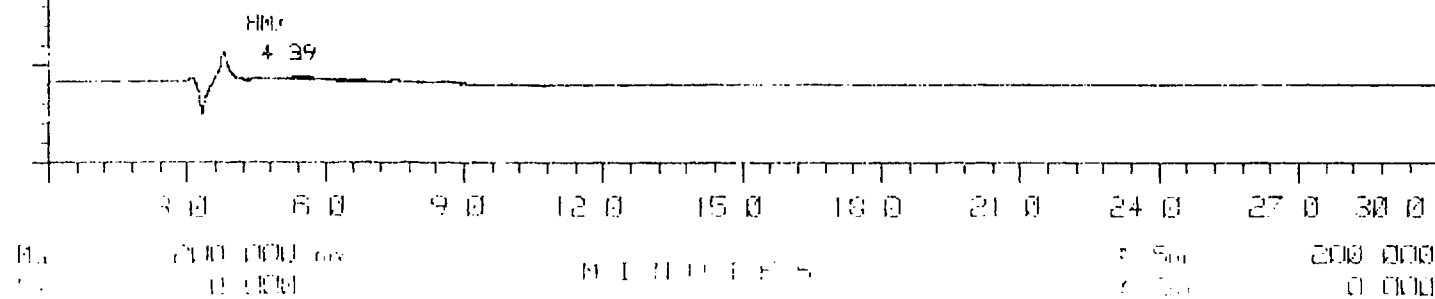
Area Analysis

File: GE223   39324-50-3   00:44:10   3/22/1784
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000
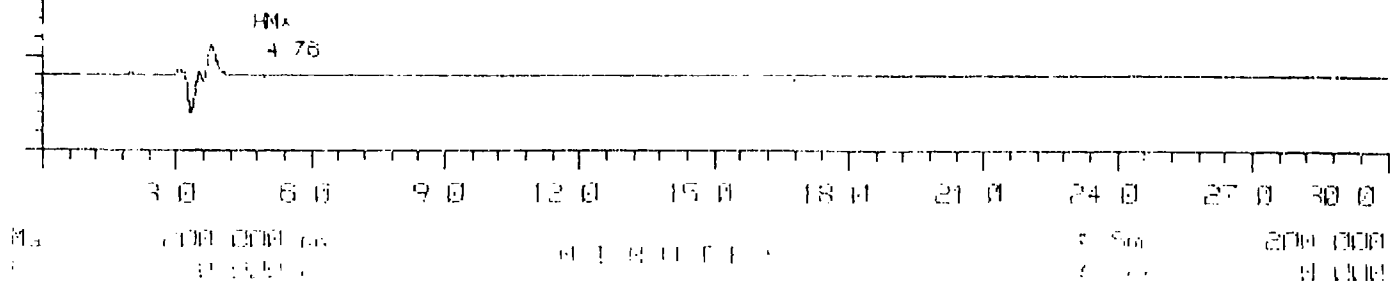
Mthd: 1391   39324-50-3   10:48:07   2/22/1784

Name	WT %	Pk	RRT	RRF	Area	Height
HMX	0.532	BCV	4.39	1.0000	0.075	0.071
001	33.546	VCV	4.82	1.0000	4.709	0.214
002	0.636	VCT	4.94	1.0000	0.089	0.082
003	2.792	VCV	5.14	1.0000	0.392	0.112
004	1.612	VCV	5.16	1.0000	0.226	0.123
005	4.948	VCV	5.32	1.0000	0.574	0.111
006	2.211	VCV	5.40	1.0000	0.310	0.074
007	53.722	BCB	5.49	1.0000	7.541	0.112
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000		

100.000

File: GE224L   39324-60-4   01 15 13   3/22/1984
FE = 0   Inst. 0   Ch 0   Tray # 0   Pol +   Page 1

Run 15   TNT
Middle Coupon
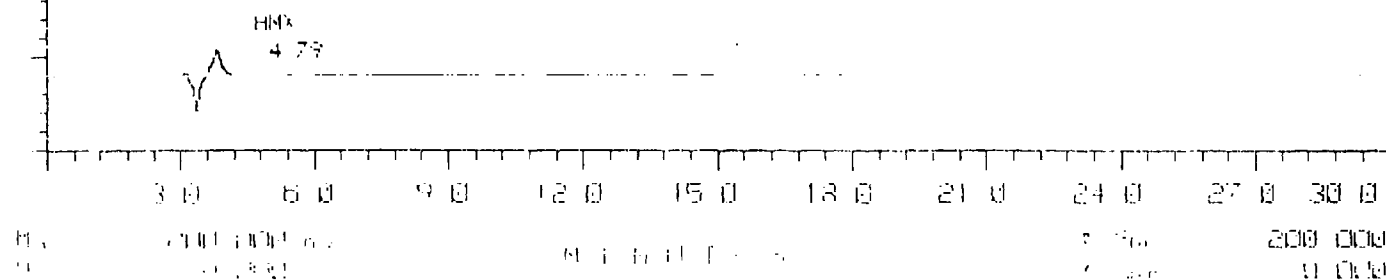
HMX
4.79

3.0   6.0   9.0   12.0   15.0   18.0   21.0   24.0   27.0   30.0

Area Analysis

Name	WT %	Pk	RRT	RRF	Area	Height
001	3.656	VCV	4.42	1.0000	0.391	0.065
002	7.224	VCV	4.64	1.0000	0.774	0.129
003	1.903	VCV	4.71	1.0000	0.204	0.141
HMX	9.575	VCV	4.79	1.0000	1.025	0.160
004	6.047	VCT	4.91	1.0000	0.648	0.115
005	10.223	VCV	5.36	1.0000	1.095	0.115
006	61.373	BCB	5.69	1.0000	6.572	0.245
RDX			7.10	1.0000		
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
2 6 DNT			14.00	1.0000		
2 4 DNT			16.10	1.0000		

100.000

Run 15   TNT

Rignt Corpon

Area Analysis

File: GE225   39324-50-5   01:46:16   2   1984
Mthd: 1310   Inst 8   Chnl O   Vial #   O   Stnd/Smpl x   100.000

Mthd: 1391   39324-50-5   10:43.25   1784

Name	WT %	Pk	RRT	RRF	Area	Height
HMX	3.993	VCV	4.52	1.0000	0.989	0.175
001	0.925	VCV	4.64	1.0000	0.205	0.117
002	2.642	VCB	4.74	1.0000	0.585	0.105
003	0.451	BCV	5.05	1.0000	0.100	0.027
004	2.963	VCV	5.24	1.0000	0.660	0.078
005	45.802	BCB	5.97	1.0000	10.175	0.244
RDX			7.10	1.0000		
006	43.223	BCB	7.57	1.0000	9.621	0.031
TETRYL			10.00	1.0000		
TNT			10.90	1.0000		
2 6 DNT			14.00	1.0000		
2 4 DNT			15.10	1.0000		
	100.000					

File: GE225   39324-50-5   01:46:16   2   1984
Mthd: 1310   Inst 8   Chnl O   Vial #   O   Stnd/Smpl x   100.000
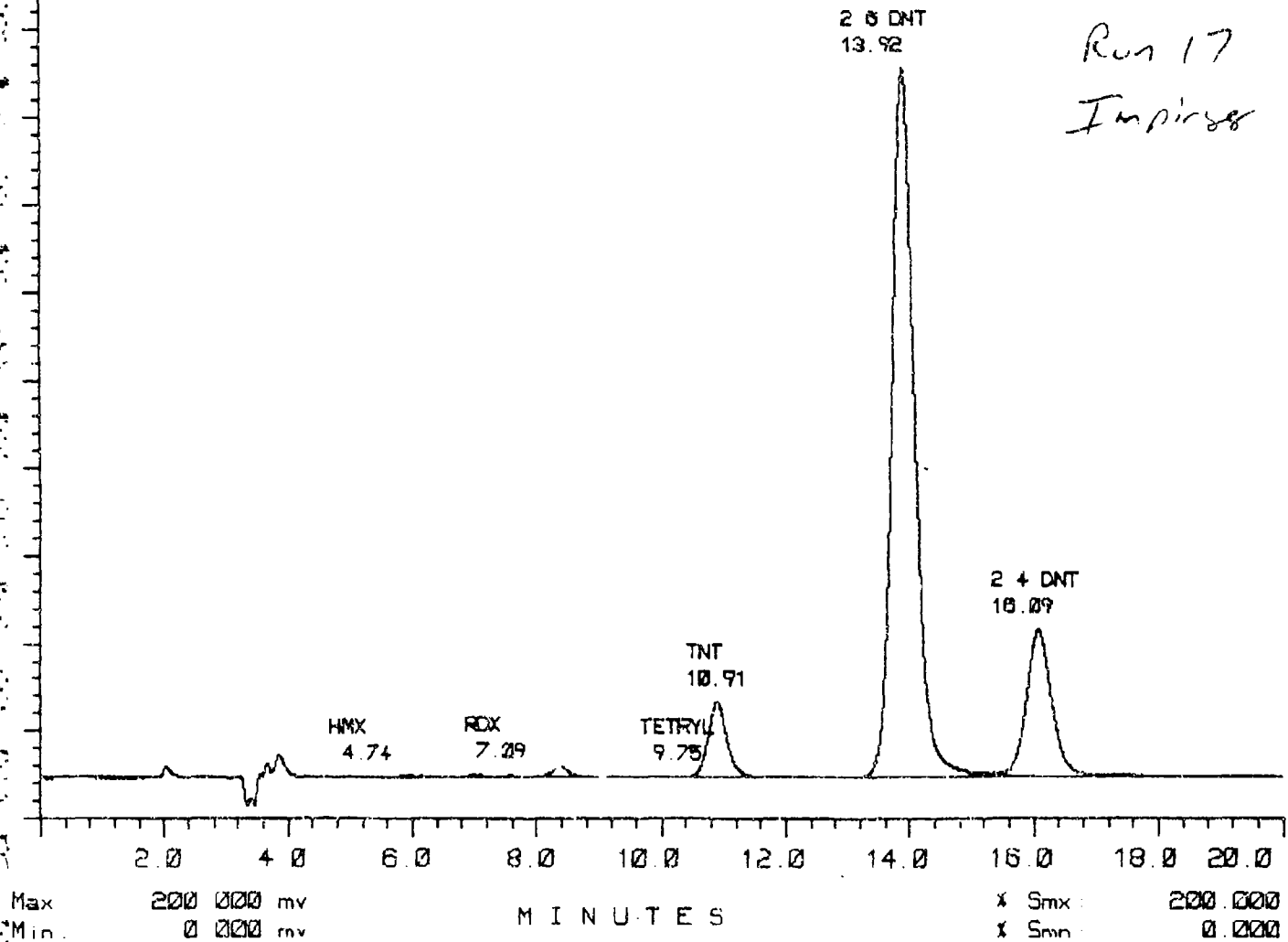
Area Analysis

Name	WT %	Pk	RRT	RRF	Area	Height
001	1.279	VCV	4.22	1.0000	0.055	0.061
002	4.165	VCV	4.26	1.0000	0.179	0.059
003	9.605	VCV	4.40	1.0000	0.413	0.113
004	9.307	VCV	4.46	1.0000	0.400	0.091
005	1.922	VCV	4.55	1.0000	0.083	0.091
006	9.203	VCV	4.60	1.0000	0.396	0.131
007	7.973	VCV	4.64	1.0000	0.343	0.144
008	0.643	VCT	4.67	1.0000	0.028	0.051
HMX	16.140	VCV	4.74	1.0000	0.694	0.161
009	11.251	VCV	4.83	1.0000	0.484	0.133
010	1.637	VCT	4.88	1.0000	0.070	0.056
011	7.172	VCV	4.95	1.0000	0.308	0.141
012	1.740	VCV	5.00	1.0000	0.075	0.053
013	2.883	VCV	5.07	1.0000	0.124	0.064
014	5.152	VCV	5.15	1.0000	0.222	0.087
015	8.929	VCV	5.22	1.0000	0.384	0.106
016	6.047	VCV	5.26	1.0000	0.260	0.113
017	4.916	VCV	5.34	1.0000	0.211	0.086
018	5.614	VCV	5.43	1.0000	0.241	0.100
019	128.641	BCV	5.92	1.0000	5.532	0.314
020	155.634	VCV	6.17	1.0000	6.693	0.256
021	8.442	VCT	6.49	1.0000	0.363	0.128
022	38.319	VCV	6.83	1.0000	1.648	0.269
RDX	237.486	VCV	7.09	1.0000	10.213	0.404
023	101.788	VCT	7.62	1.0000	4.377	0.311
024	1105.068	VCB	8.39	1.0000	47.524	2.518
025	7.693	BCV	9.31	1.0000	0.331	0.127
026	80.947	VCV	9.62	1.0000	3.481	0.329
TETRYL	213.875	VCV	9.75	1.0000	9.198	0.357
027	29.302	VCT	10.26	1.0000	1.260	0.160
TNT	8830.908	VCV	10.91	1.0000	379.774	17.385
028	17.392	VCV	11.94	1.0000	0.748	0.192
029	53.228	VCV	12.11	1.0000	2.289	0.195
030	107.737	VCV	12.31	1.0000	4.633	0.216
2 6 DNT	$271.374	VCV	13.92	1.0000	4140.165	162.013
2 4 DNT	$417.792	VCB	16.09	1.0000	1007.025	33.676
031	23.299	VCT	17.23	1.0000	1.002	0.161
032	12.182	VCT	17.91	1.0000	0.524	0.136
033	43.361	VCT	18.34	1.0000	1.865	0.176
	$1000.046					

Run 17   2,6 DNT

Trap

HMX	ROX	TETRYL	2 5 DNT	2 4 DNT
4.58	7.38	10.283	13.82	16.57

2.0   4.0   6.0   8.0   10.0   12.0   14.0   16.0   18.0   20.0

Max: 200.000 mv   M I N U T E S   Smx :   200.000
Min   0.000 mv   Smn :   0.000

353
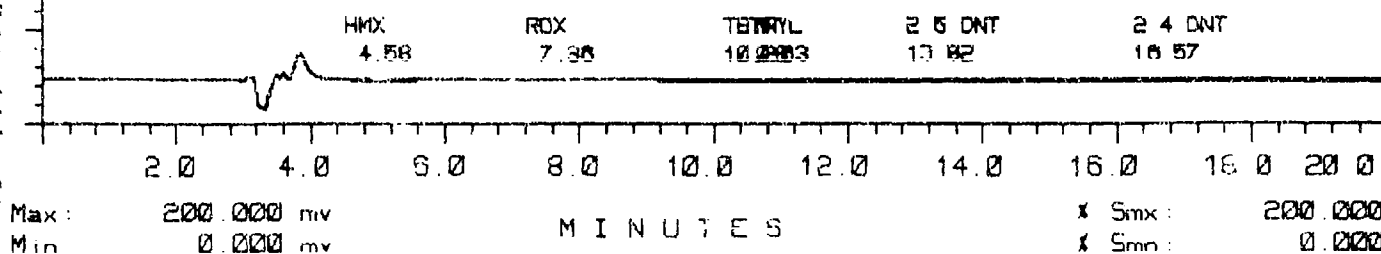
Area Analysis

Name	Wt %	Pk	RRT	RRF	Area	Height
001	0.228	VCV	4.27	1.0000	0.242	0.077
002	0.317	VCV	4.36	1.0000	0.336	0.132
003	0.493	VCV	4.44	1.0000	0.522	0.177
004	0.436	VCV	4.46	1.0000	0.462	0.157
005	0.056	VCT	4.50	1.0000	0.060	0.056
HMX	0.863	VCV	4.58	1.0000	0.915	0.169
006	0.113	VCT	4.62	1.0000	0.120	0.082
007	0.329	VCV	4.71	1.0000	0.349	0.164
008	0.352	VCV	4.75	1.0000	0.373	0.196
009	0.482	VCV	4.82	1.0000	0.511	0.157
010	0.374	VCV	4.88	1.0000	0.396	0.178
011	0.344	VCV	4.90	1.0000	0.364	0.197
012	0.277	VCV	4.96	1.0000	0.293	0.109
013	0.749	VCV	5.12	1.0000	0.794	0.145
014	0.480	VCV	5.20	1.0000	0.509	0.174
015	0.470	VCV	5.28	1.0000	0.498	0.183
016	0.683	VCV	5.36	1.0000	0.724	0.225
017	0.225	VCV	5.40	1.0000	0.239	0.158
018	4.367	VCV	5.65	1.0000	4.628	0.347
019	3.296	VCB	5.87	1.0000	3.493	0.274
020	0.991	BCV	6.21	1.0000	1.050	0.138
021	0.465	VCV	6.35	1.0000	0.493	0.120
022	1.129	VCV	6.49	1.0000	1.197	0.146
023	0.599	VCV	6.80	1.0000	0.635	0.143
024	1.481	VCV	7.00	1.0000	1.569	0.107
RDX	1.541	VCV	7.34	1.0000	1.633	0.141
025	1.055	VCV	7.76	1.0000	1.118	0.152
026	0.939	VCV	7.96	1.0000	0.995	0.163
027	1.719	VCV	8.18	1.0000	1.822	0.200
028	1.133	VCV	8.31	1.0000	1.201	0.185
029	2.411	VCV	8.49	1.0000	2.555	0.204
030	0.383	BCV	8.96	1.0000	0.406	0.106
031	0.552	VCV	9.15	1.0000	0.585	0.129
032	2.833	VCV	9.44	1.0000	3.002	0.170
033	1.342	VCT	9.75	1.0000	1.422	0.150
TETRYL	1.788	VCV	10.26	1.0000	1.895	0.148
TNT	3.744	VCV	10.63	1.0000	3.968	0.189
034	0.765	VCT	10.83	1.0000	0.811	0.135
035	0.463	VCV	11.28	1.0000	0.490	0.137
036	0.829	VCV	11.39	1.0000	0.878	0.133
037	1.073	VCV	11.73	1.0000	1.137	0.161
038	1.224	VCV	11.82	1.0000	1.297	0.174
039	2.981	VCV	12.07	1.0000	3.159	0.202
040	1.435	VCT	12.27	1.0000	1.521	0.141
041	1.246	VCV	12.69	1.0000	1.321	0.149
2 6 DNT	17.277	VCV	13.82	1.0000	18.310	0.519
042	2.604	VCV	14.65	1.0000	2.760	0.182
043	3.109	VCV	15.19	1.0000	3.295	0.146
044	1.490	VCV	15.64	1.0000	1.579	0.182
045	2.206	VCV	15.98	1.0000	2.338	0.186
046	1.340	VCV	16.15	1.0000	1.420	0.202
2 4 DNT	2.923	VCV	16.57	1.0000	2.991	0.195

File: IA029	39324-53-28				21:31:49	3/26/1984
Mthd: 900	39324-53-28				14:07:56	3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
047	2.096	VCV	16.89	1.0000	2.221	0.191
048	1.246	VCV	17.09	1.0000	1.321	0.151
049	1.922	VCV	17.37	1.0000	2.037	0.172
050	2.129	VCV	17.69	1.0000	2.257	0.195
051	1.407	VCV	17.98	1.0000	1.491	0.169
052	0.529	VCV	18.18	1.0000	0.561	0.159
053	2.340	VCV	18.49	1.0000	2.480	0.174
054	2.966	VCV	18.74	1.0000	3.143	0.174
055	1.154	VCT	19.04	1.0000	1.223	0.131
056	1.917	VCV	19.42	1.0000	2.032	0.178
057	0.994	VCV	19.71	1.0000	1.053	0.159
058	1.398	VCV	19.86	1.0000	1.481	0.199
	100.000					

File:IA027L   39324-53-26   20:49:41   3/26/1984
FE * 0   Inst: 8 Ch:0   Tray * 0   Pol +   Page 1

Run 17   2,6 DN

Sump

```
        HMX         RDX        TETRYL      2 6 DNT    2 4 DNT
        4.28        7.25       9.9 10.82   13.93      16.21
```

```
  2.0    4.0    6.0    8.0   10.0   12.0   14.0   16.0   18.0  20.0
```

                         M I N U T E S

Max: 200.000 mv   % Smx :   200.000
Min: 0.000 mv   % Smn :   0.000
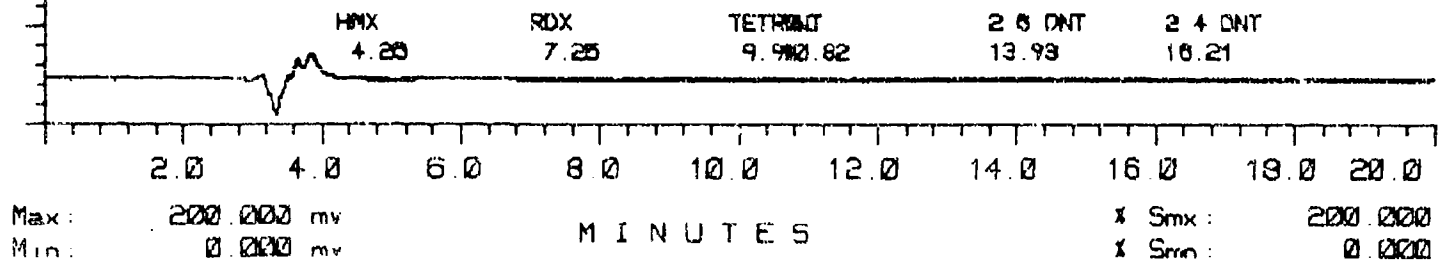
Area Analysis

File: IA027 39324-53-26   20:49:41   3/26/1984
Mthd: 1310   Inst 8   Chnl O   Vial # 0 Stnd/Smpl % 100.000

Mthd: 900 39324-53-26   14:07:20   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
HMX	0.855	VCV	4.26	1.0000	0.957	0.072
001	0.506	VCV	4.40	1.0000	0.567	0.208
002	0.707	VCV	4.45	1.0000	0.792	0.218
003	0.587	VCV	4.57	1.0000	0.657	0.184
004	0.588	VCV	4.60	1.0000	0.657	0.188
005	0.367	VCV	4.71	1.0000	0.411	0.158
006	0.119	VCV	4.75	1.0000	0.133	0.096
007	0.225	VCV	4.80	1.0000	0.251	0.101
008	0.669	VCV	4.86	1.0000	0.749	0.168
009	0.245	VCT	4.88	1.0000	0.274	0.105
010	0.051	VCV	4.99	1.0000	0.057	0.080
011	0.099	VCV	5.01	1.0000	0.111	0.080
012	0.173	VCV	5.07	1.0000	0.193	0.084
013	0.238	VCV	5.17	1.0000	0.266	0.133
014	0.650	VCV	5.24	1.0000	0.728	0.156
015	0.677	VCV	5.34	1.0000	0.758	0.151
016	0.118	VCV	5.44	1.0000	0.132	0.067
017	4.556	BCV	5.98	1.0000	5.098	0.258
018	3.416	VCV	6.17	1.0000	3.822	0.268
019	2.042	VCV	6.77	1.0000	2.285	0.156
RDX	2.021	VCV	7.25	1.000C	2.261	0.183
020	2.482	VCV	7.46	1.0000	2.777	0.176
021	1.014	VCV	7.88	1.0000	1.135	0.198
022	1.438	VCV	8.04	1.0000	1.609	0.156
023	1.115	VCV	8.32	1.0000	1.247	0.177
024	0.632	VCV	8.50	1.0000	0.708	0.112
025	0.813	VCV	8.77	1.0000	0.909	0.139
026	0.737	BCV	8.97	1.0000	0.824	0.149
027	1.152	VCV	9.12	1.0000	1.289	0.139
028	2.529	VCV	9.41	1.0000	2.829	0.143
TETRYL	0.573	VCV	9.91	1.0000	0.641	0.167
029	3.141	VCV	10.36	1.0000	3.515	0.212
030	1.123	VCV	10.59	1.0000	1.256	0.198
TNT	4.832	VCV	10.82	1.0000	5.407	0.228
031	0.475	VCT	11.16	1.0000	0.532	0.134
032	1.954	VCV	11.91	1.0000	2.186	0.145
033	1.360	VCV	12.11	1.000C	1.522	0.147
034	2.612	VCV	12.50	1.0000	2.923	0.178
035	2.053	VCV	12.88	1.0000	2.297	0.255
036	3.650	VCV	13.07	1.0000	4.084	0.264
037	1.199	VCV	13.37	1.C000	1.342	0.251
038	2.252	VCV	13.55	1.0000	2.519	0.274
2 6 DNT	10.889	VCV	13.93	1.0000	12.184	0.461
039	0.080	VCT	14.18	1.0000	0.090	0.163
040	0.990	VCV	14.47	1.0000	1.108	0.134
041	1.515	VCV	14.76	1.0000	1.695	0.172
042	0.666	VCV	14.91	1.0000	0.745	0.144
043	1.415	VCV	15.27	1.0000	1.583	0.150
044	0.861	VCV	15.39	1.0000	0.963	0.187
045	0.974	VCV	15.56	1.0000	1.090	0.184
046	2.505	VCV	15.74	1.0000	2.803	0.265
047	1.697	VCV	16.04	1.0000	1.898	0.226

File: IA027 39324-53-26   20:49:41   3/26/1984

File: IA027   39324-53-26   20:49:41   3/26/1984
Mthd: 900   39324-53-26   14:07:20   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
2 4 DNT	2.935	VCV	16.21	1.0000	3.284	0.234
048	1.665	VCV	16.67	1.0000	1.863	0.188
049	2.644	VCV	17.21	1.0000	2.958	0.176
050	0.344	VCV	17.47	1.0000	0.385	0.132
051	0.937	VCV	17.65	1.0000	1.048	0.161
052	2.312	VCV	17.81	1.0000	2.587	0.186
053	4.999	VCV	18.42	1.0000	5.593	0.213
054	2.633	VCV	19.12	1.0000	2.946	0.218
055	3.895	VCV	19.50	1.0000	4.358	0.211
	100.000					
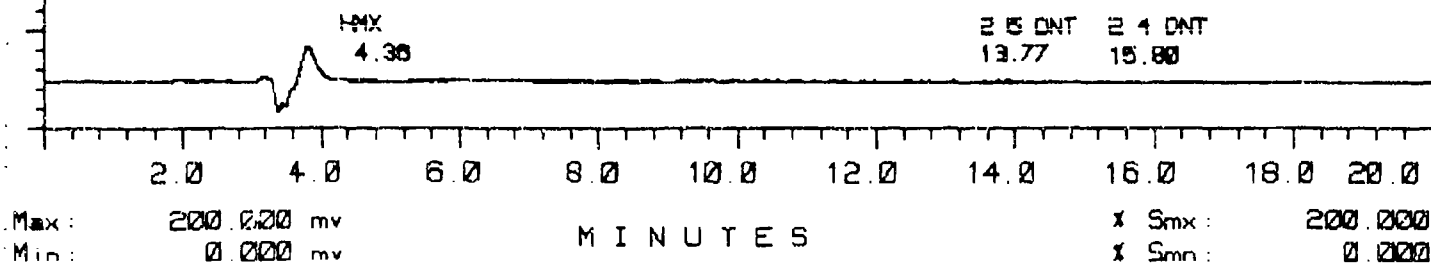
File: IA011L   39324-53-23   15:12:43   3/26/1984
E = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

Run 17   2,6 DN
Rt. Coupon

HMX
4.36

2 6 DNT   2 4 DNT
13.77   15.80

    2.0   4.0   6.0   8.0   10.0   12.0   14.0   16.0   18.0 20.0

Max: 200.000 mv   M I N U T E S   X Smx: 200.000
Min: 0.000 mv   X Smn: 0.000

Area Analysis

Name	WT %	Pk	RRT	RRF	Area	Height
001	0. 245	VCV	4. 32	1. 0000	0. 138	0. 126
HMX	1. 944	VCV	4. 36	1. 0000	1. 094	0. 200
002	0. 215	VCT	4. 44	1. 0000	0. 121	0. 078
003	1. 139	VCV	4. 55	1. 0000	0. 641	0. 160
004	1. 341	VCV	4. 61	1. 0000	0. 754	0. 160
005	1. 809	VCV	4. 75	1. 0000	1. 018	0. 184
006	0. 629	VCV	4. 81	1. 0000	0. 354	0. 146
007	0. 768	VCV	4. 87	1. 0000	0. 432	0. 133
008	0. 126	VCT	4. 92	1. 0000	0. 071	0. 067
009	0. 309	VCV	5. 01	1. 0000	0. 174	0. 076
010	0. 341	VCV	5. 08	1. 0000	0. 192	0. 112
011	0. 379	VCV	5. 10	1. 0000	0. 213	0. 138
012	0. 738	VCV	5. 17	1. 0000	0. 415	0. 149
013	0. 735	VCV	5. 20	1. 0000	0. 414	0. 140
014	0. 352	VCV	5. 25	1. 0000	0. 198	0. 131
015	1. 578	VCV	5. 31	1. 0000	0. 888	0. 228
016	2. 686	VCV	5. 44	1. 0000	1. 511	0. 232
017	13. 683	VCB	5. 80	1. 0000	7. 698	0. 330
018	3. 486	BCV	6. 36	1. 0000	1. 961	0. 146
019	8. 030	VCV	6. 74	1. 0000	4. 518	0. 165
RDX			7. 10	1. 0000		
020	3. 893	VCV	7. 67	1. 0000	2. 190	0. 173
021	8. 838	VCB	8. 45	1. 0000	4. 972	0. 163
TETRYL			10. 00	1. 0000		
TNT			10. 90	1. 0000		
2 6 DNT	3. 364	BCV	13. 77	1. 0000	1. 893	0. 139
022	1. 534	VCT	13. 88	1. 0000	0. 863	0. 118
023	1. 630	VCV	14. 64	1. 0000	0. 917	0. 121
024	4. 579	VCV	14. 97	1. 0000	2. 576	0. 122
2 4 DNT	9. 134	VCV	15. 80	1. 0000	5. 138	0. 180
025	2. 397	VCV	16. 17	1. 0000	1. 348	0. 188
026	1. 555	VCV	16. 35	1. 0000	0. 875	0. 199
027	1. 317	VCV	16. 51	1. 0000	0. 741	0. 137
028	3. 498	VCV	16. 78	1. 0000	1. 968	0. 173
029	1. 268	VCV	16. 97	1. 0000	0. 714	0. 134
030	3. 265	VCV	17. 27	1. 0000	1. 837	0. 190
031	5. 685	VCV	17. 35	1. 0000	3. 198	0. 188
032	0. 645	VCT	17. 60	1. 0000	0. 363	0. 135
033	0. 971	VCV	17. 94	1. 0000	0. 546	0. 121
034	4. 517	VCB	18. 10	1. 0000	2. 541	0. 176
035	1. 375	BCV	19. 82	1. 0000	0. 774	0. 125
	100. 000					

File: IA012L   39324-53-24   15:33:47   3/26/1984
FE = 0   Inst: 8 Ch:0   Tray = 0   Pol +   Page 1

Run 17 2,6 DNT

M - Comm

HMX   RDX   TETRYL   2 6 DNT DNT
4.55   5.84   10.07.18   14.595.51

| | | | | | | | | | |
2.0   4.0   6.0   8.0   10.0   12.0   14.0   16.0   18.0   20.0

Max: 200.0000 mv   M I N U T E S   % Smx: 200.0000
Min: 0.0000 mv   % Smn: 0.0000

File: IA012   39324-53-24   15:33:47   3/26/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl %   100.000

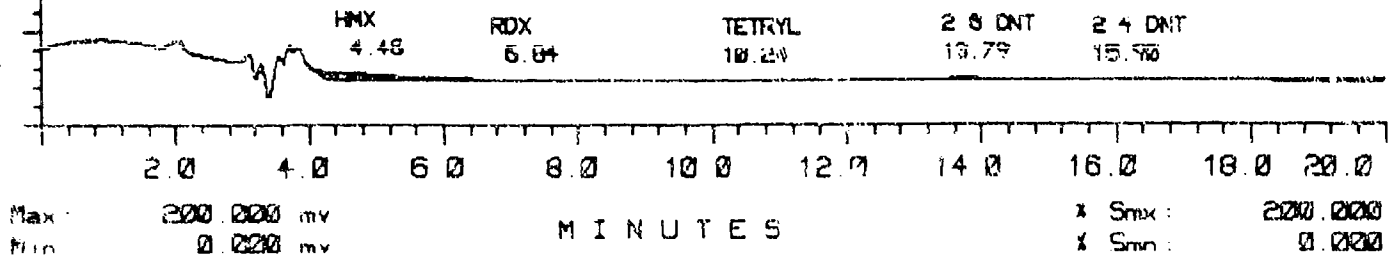Mthd: 900 39324-53-24   14:03:13   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	0.330	VCV	4.45	1.0000	0.315	0.096
002	0.161	VCV	4.56	1.0000	0.153	0.084
003	0.107	VCV	4.60	1.0000	0.102	0.104
HMX	1.022	VCV	4.65	1.0000	0.976	0.131
004	0.228	VCV	4.77	1.0000	0.218	0.156
005	1.177	VCV	4.82	1.0000	1.124	0.186
006	0.472	VCT	4.90	1.0000	0.451	0.104
007	0.083	VCV	5.04	1.0000	0.079	0.055
008	0.082	VCV	5.08	1.0000	0.078	0.064
009	0.755	VCV	5.16	1.0000	0.721	0.148
010	0.319	VCV	5.24	1.0000	0.305	0.157
011	0.442	VCV	5.27	1.0000	0.422	0.154
012	0.307	VCV	5.31	1.0000	0.293	0.134
013	0.168	VCV	5.36	1.0000	0.161	0.124
014	0.667	VCV	5.44	1.0000	0.637	0.160
015	1.556	VCV	5.61	1.0000	1.486	0.171
016	1.460	VCV	5.90	1.0000	1.394	0.122
017	2.210	BCV	6.02	1.0000	2.110	0.146
RDX	7.069	VCV	6.84	1.0000	6.752	0.243
018	1.174	VCT	7.43	1.0000	1.121	0.136
019	1.997	VCV	7.74	1.0000	1.907	0.156
020	0.910	VCT	8.04	1.0000	0.869	0.131
021	2.327	VCV	8.52	1.0000	2.223	0.167
022	1.420	VCB	8.87	1.0000	1.356	0.130
023	2.181	BCV	9.17	1.0000	2.083	0.147
024	0.610	VCV	9.42	1.0000	0.583	0.134
025	1.644	VCV	9.62	1.0000	1.570	0.221
026	0.599	VCV	9.81	1.0000	0.572	0.118
TETRYL	2.131	VCV	10.07	1.0000	2.035	0 196
027	2.011	VCV	10.20	1.0000	1.921	0.195
TNT	9.899	VCV	11.18	1.0000	9.455	0.223
028	0.856	VCT	11.40	1.0000	0.817	0.145
029	9.770	VCV	12.43	1.0000	9.332	0.234
030	0.583	VCT	12.57	1.0000	0.557	0.156
031	3.168	VCV	13.20	1.0000	3.026	0.218
032	0.632	VCT	13.36	1.0000	0.604	0.116
033	0.811	VCV	13.72	1.0000	0.775	0.154
034	2.149	VCV	14.04	1.0000	2.053	0.163
035	1.501	VCV	14.27	1.0000	1.434	0.182
2 6 DNT	2.709	VCV	14.59	1.0000	2.587	0.214
036	4.860	VCV	14.77	1.0000	4.642	0.221
2 4 DNT	3.987	VCV	15.51	1.0000	3.808	0.218
037	1.372	VCV	15.88	1.0000	1.310	0.207
038	2.458	VCV	16.03	1.0000	2.348	0.172
039	2.043	VCV	16.67	1.0000	1.951	0.166
040	1.259	VCV	17.00	1.0000	1.203	0.172
041	1.903	VCV	17.29	1.0000	1.817	0.181
042	0.811	VCV	17.40	1.0000	0.775	0.149
043	2.966	VCV	17.72	1.0000	2.833	0.193
044	1.473	VCV	18.18	1.0000	1.407	0.169
045	1.316	VCV	18.34	1.0000	1.257	0.179
046	0.577	VCV	18.45	1.0000	0.551	0.123

File: IA012   39324-53-24   15:33:47   3/26/1984
Mthd: 900   39324-53-24   14:03:13   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
047	2.565	VCV	18.96	1.0000	2.450	0.154
048	1.343	VCV	19.12	1.0000	1.283	0.159
049	1.061	VCV	19.34	1.0000	1.014	0.159
050	0.712	VCV	19.58	1.0000	0.680	0.154
051	1.598	VCV	19.88	1.0000	1.526	0.161
	100.000					

File:IA010L   39324-53-25   14:51:39 3/26/1984
FE = 0   Inst: 8 Ch:0   Tray = 0   Pol +   Page 1

Run 17   2,6 DNT

Left Coupon

HMX	RDX	TETRYL	TNT	2 6 DNT	2 4 DNT
4.39	6 82	10 82	11 28	13 01	15 55

2 0   4 0   6 0   8 0   10 0   12 0   14 0   16 0   18 0   20 0

Max: 200 0000 mv   M I N U T E S   % Smx   200 0000
Min: 0 0000 mv   % Smn   0 0000
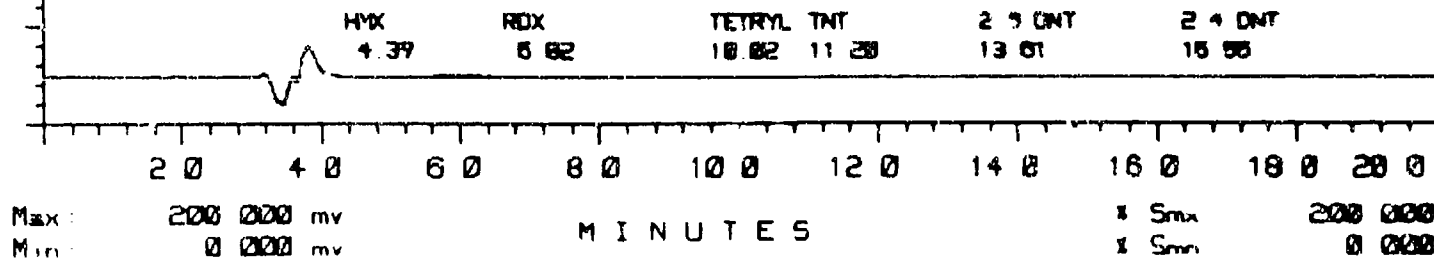
Area Analysis

File: IAO10   39324-53-25   14:51:39   3/26/1984
Mthd. 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 930   39324-53-25   14:02:43   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	0.081	VCV	4.32	1.0000	0.068	0.051
HMX	1.482	VCV	4.39	1.0000	1.256	0.119
002	0.133	VCV	4.68	1.0000	0.113	0.094
003	0.829	VCV	4.74	1.0000	0.702	0.148
004	0.399	VCV	4.80	1.0000	0.338	0.173
005	0.139	VCV	4.84	1.0000	0.118	0.145
006	0.715	VCV	4.89	1.0000	0.606	0.158
007	0.160	VCV	5.02	1.0000	0.136	0.056
008	0.225	VCV	5.07	1.0000	0.191	0.100
009	0.138	VCV	5.11	1.0000	0.117	0.066
010	0.426	VCV	5.18	1.0000	0.370	0.120
011	0.548	VCV	5.28	1.0000	0.465	0.169
012	0.440	VCV	5.32	1.0000	0.373	0.181
013	0.888	VCV	5.42	1.0000	0.752	0.192
014	0.460	VCV	5.47	1.0000	0.390	0.157
015	4.392	VCV	5.68	1.0000	3.722	0.261
016	2.279	BCV	5.98	1.0000	1.931	0.119
017	1.674	VCV	6.42	1.0000	1.418	0.169
RDX	3.840	VCV	6.82	1.0000	3.254	0.158
018	1.595	VCV	7.52	1.0000	1.352	0.159
019	0.977	VCV	7.68	1.0000	0.828	0.152
020	2.963	VCV	7.98	1.0000	2.511	0.156
021	1.697	VCV	8.51	1.0000	1.433	0.155
022	0.319	VCT	8.60	1.0000	0.270	0.110
023	2.202	BCV	8.94	1.0000	1.866	0.157
024	2.338	VCV	9.20	1.0000	1.981	0.150
025	0.490	VCT	9.40	1.0000	0.415	0.124
TETRYL	4.455	VCV	10.02	1.0000	3.775	0.158
026	2.796	VCV	10.35	1.0000	2.369	0.163
027	0.561	VCT	10.63	1.0000	0.476	0.122
DNT	2.626	VCV	11.20	1.0000	2.226	0.176
028	1.685	VCT	11.56	1.0000	1.423	0.160
029	0.720	VCV	11.88	1.0000	0.610	0.129
030	2.335	VCV	12.00	1.0000	1.979	0.165
031	3.370	VCV	12.80	1.0000	2.856	0.170
032	2.035	VCV	13.15	1.0000	1.733	0.139
033	1.208	VCV	13.32	1.0000	1.023	0.173
2 6 DNT	2.708	VCV	13.61	1.0000	2.295	0.136
034	1.480	VCV	14.07	1.0000	1.255	0.135
035	0.859	VCV	14.43	1.0000	0.728	0.119
036	2.668	VCV	14.81	1.0000	2.261	0.169
037	1.662	VCV	15.__	1.0000	1.409	0.139
038	2.863	VCV	15.	1.0000	2.426	0.156
039	2.149	VCV	15.__	1.0000	1.822	0.157
040	1.970	VCV	16.25	1.0000	1.669	0.139
2 4 DNT	5.301	VCV	16.55	1.0000	4.492	0.221
041	1.841	VCV	17.14	1.0000	1.561	0.173
042	6.909	VCV	17.33	1.0000	5.855	0 196
043	3.321	VCV	18.12	1.0000	2.815	0.201
044	3.496	VCV	18.43	1.0000	2.962	0.243
045	3.430	VCV	18.75	1.0000	2.907	0.209
046	1.898	VCV	19.08	1.0000	1.609	0.201

File: IA010   39324-53-25   14:51:39   3/26/1984
Mthd: 900   39324-53-25   14:02:43   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
047	3.805	VCV	19.35	1.0000	3.225	0.213
	100.000					

File:IA022L　　　　39324-55-18　　　　　　　　　19:04:24　3/26/1984
E = 0　　　　　　　Inst: 8　Ch:0　　Tray = 0　　　Pol　+　　　Page　1

Run 18 RDX
Inninser

2 6 DNT
13.84

2 4 DNT
15.99

TNT
10.95

HMX
4.32

RDX
7 11

TETRYL
10.21

2.0　4.0　6.0　8.0　10.0　12.0　14.0　16.0　18.0　20.0

Max:　200.000 mv　　　　　　　　　　　　　　% Smx:　200.000
Min:　0.000 mv　　　M I N U T E S　　　　　% Smn:　0.000
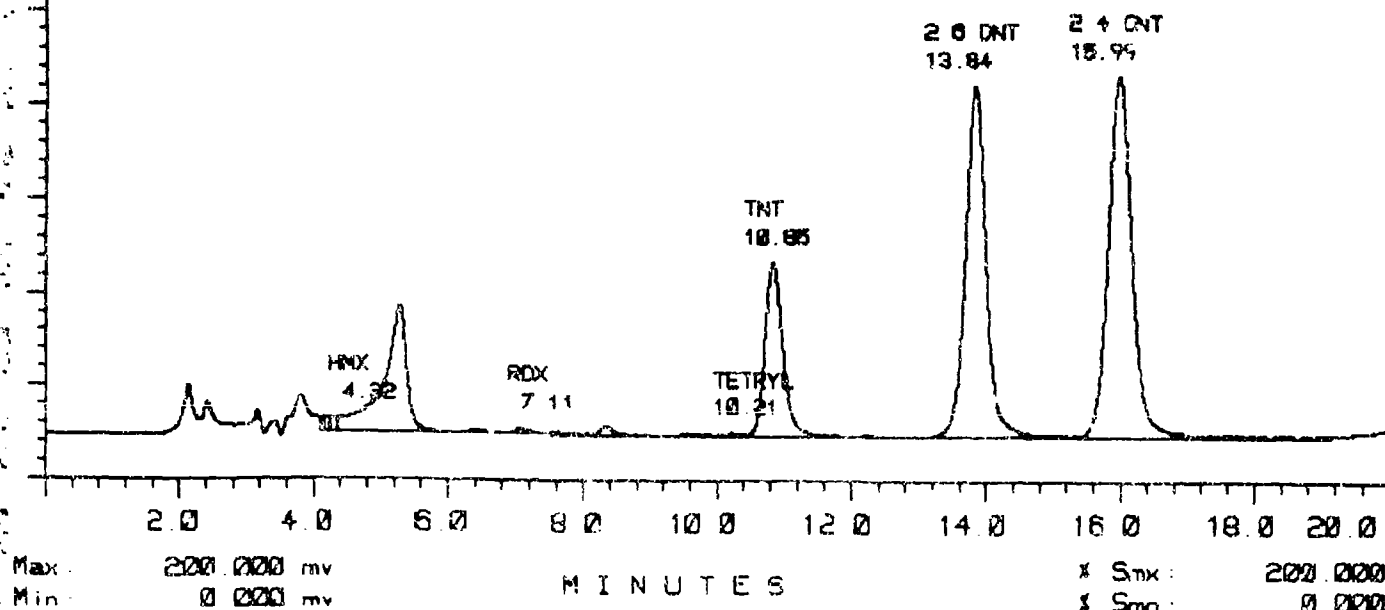
Area Analysis

File: IA022   39324-55-18   19:04:24   3/26/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stn1/Smpl %   100.000

Mthd: 900   39324-55-18   14:05:56   3/27/1984

Name	WT %		RRT	RRF	Area	Height
001	0.124	VCV	4.13	1.0000	6.330	3.000
002	0.371	VCV	4.18	1.0000	18.897	3.020
HM	0.410	VCV	4.32	1.0000	20.893	2.960
003	13.335	VCV	5.31	1.0000	679.266	27.214
004	0.183	BCV	6.45	1.0000	9.297	0.647
RDX	0.377	VCV	7.11	1.0000	19.219	1.060
005	0.112	VCT	7.60	1.0000	5.730	0.511
006	0.497	VCV	8.34	1.0000	25.323	1.736
007	0.060	BCV	8.99	1.0000	3.045	0.205
008	0.229	VCV	9.81	1.0000	11.642	0.482
TETRYL	0.258	VCV	10.21	1.0000	13.166	0.773
TNT	13.591	VCV	10.85	1.0000	692.288	37.030
009	0.179	VCV	12.24	1.0000	9.107	0.369
010	0.009	VCT	12.84	1.0000	0.442	0.124
2 6 DNT	32.186	VCV	13.84	1.0000	1639.458	75.303
2 4 DNT	37.828	VCV	15.99	1.0000	1926.873	77.468
011	0.021	VCT	17.42	1.0000	1.066	0.167
012	0.021	VCT	18.01	1.0000	1.048	0.130
013	0.072	VCV	18.76	1.0000	3.688	0.385
014	0.136	VCV	19.06	1.0000	6.951	0.468
	100.000					

File:IA023L   39234-55-19   19:25:28 3/26/1984
FE = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

Run 18   ROX

Trap

HMX	RDX	TETRYL	2 6 DNT	2 4 DNT
4.48	5.84	10.24	13.79	15.58

2.0   4.0   6.0   8.0   10.0   12.0   14.0   16.0   18.0 20.0

Max   200.000 mv   M I N U T E S   X Smx: 200.000
Min   0.000 mv   X Smn: 0.000

Area Analysis

Name	WT %	Pk	RRT	RRF	Area	Height
001	15.990	VCV	4.00	1.0000	32.688	0.210
002	1.969	VCV	4.30	1.0000	4.026	1.401
003	4.343	VCV	4.34	1.0000	8.877	1.405
HMX	5.982	VCV	4.48	1.0000	12.230	1.334
004	3.167	VCV	4.60	1.0000	6.473	1.193
005	3.118	VCV	4.70	1.0000	6.374	1.231
006	14.654	VCV	4.80	1.0000	29.957	1.212
007	0.100	VCT	4.98	1.0000	0.204	0.085
008	0.016	VCT	5.08	1.0000	0.032	0.049
009	0.060	VCT	5.25	1.0000	0.123	0.094
010	9.182	VCB	5.36	1.0000	18.771	0.598
011	0.088	VCT	5.41	1.0000	0.179	0.114
012	0.512	VCT	5.51	1.0000	1.047	0.140
013	0.457	BCV	6.63	1.0000	0.934	0.143
RDX	1.120	VCV	6.84	1.0000	2.290	0.168
014	0.789	VCT	7.00	1.0000	1.613	0.147
015	0.383	VCV	7.60	1.0000	0.784	0.150
016	0.407	VCV	7.82	1.0000	0.833	0.141
017	0.831	VCV	7.91	1.0000	1.698	0.177
018	0.421	VCV	8.30	1.0000	0.861	0.145
019	0.528	VCV	8.53	1.0000	1.080	0.191
020	0.374	VCV	8.69	1.0000	0.764	0.118
021	1.068	BCV	9.02	1.0000	2.182	0.212
022	1.179	VCV	9.36	1.0000	2.409	0.199
023	0.682	VCV	9.69	1.0000	1.394	0.151
024	0.272	VCV	9.99	1.0000	0.556	0.127
025	0.301	VCV	10.03	1.0000	0.616	0.133
TETRYL	1.333	VCB	10.24	1.0000	2.725	0.175
TNT			10.90	1.0000		
026	0.385	BCV	12.76	1.0000	0.787	0.173
027	0.813	VCV	13.05	1.0000	1.662	0.184
028	0.519	VCV	13.16	1.0000	1.061	0.187
029	0.874	VCV	13.40	1.0000	1.786	0.262
2 6 DNT	9.118	VCV	13.79	1.0000	18.640	0.663
030	0.379	VCT	14.36	1.0000	0.776	0.124
031	1.166	VCV	14.92	1.0000	2.384	0.205
032	0.530	VCV	15.22	1.0000	1.082	0.197
033	1.346	VCV	15.66	1.0000	2.752	0.232
2 4 DNT	4.758	VCV	15.90	1.0000	9.727	0.373
034	0.395	VCT	16.05	1.0000	0.807	0.142
035	0.384	VCV	16.71	1.0000	0.785	0.128
036	2.767	VCV	16.92	1.0000	5.656	0.200
037	0.616	VCT	17.06	1.0000	1.260	0.142
038	0.521	VCT	17.67	1.0000	1.065	0.155
039	0.536	VCV	18.07	1.0000	1.097	0.171
040	0.783	VCV	18.18	1.0000	1.601	0.142
041	0.728	VCV	18.61	1.0000	1.488	0.159
042	0.231	VCV	18.79	1.0000	0.473	0.129
043	0.614	VCV	18.94	1.0000	1.255	0.200
044	0.835	VCV	19.25	1.0000	1.706	0.149
045	0.812	VCV	19.40	1.0000	1.659	0.187
046	1.073	VCV	19.58	1.0000	2.193	0.199

File: IA023   39324-55-19   19:25:28   3/26/1984
Mthd: 900   39324-55-19   14:06:06   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
047	0.489	VCV	19.85	1.0000	0.999	0.191
	100.000					

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File: IA023   39324-55-19   19:25:28   3/26/1984
Mthd: 900

File: IA024L   39324-55-20   19:46:32   3/26/1984
FE : 0   Inst: 8   Ch:0   Tray # 0   Pol +   Page 1

Run 18   RDX

Sump

HMX	RDX		TETRYL		2 5 DNT	2 4 DNT
4 57	7 07		1 0 0 0 0 0		14 51	18 38

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   2.0      4 0      6 0      8 0      10 0     12 0     14 0     16 0     18.0   20.0
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M I N U T E S

Max: 200.0000 mv   % Smx: 200.0000
Min: 0.0000 mv   % Smn: 0.0000
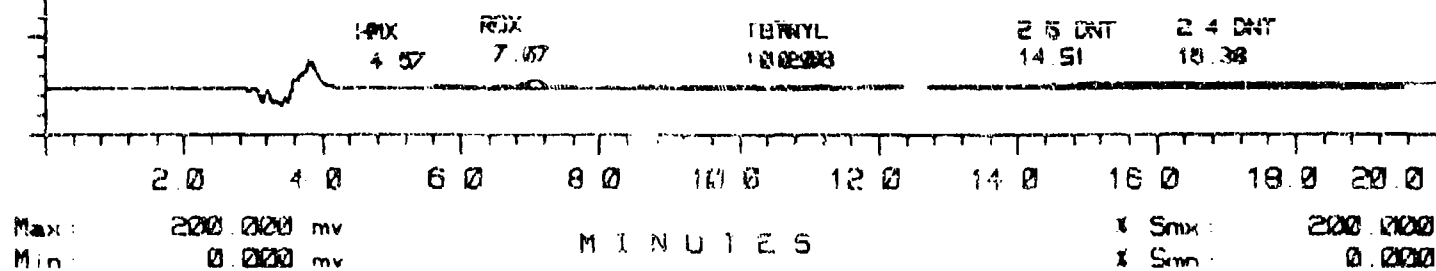
Area Analysis

File: IA024 39324-55-20   19:46:32 3/26/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 900 39324-55-20   14:06:26 3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	0.029	VCV	4.25	1.0000	0.107	0.052
002	0.016	VCV	4.33	1.0000	0.058	0.059
003	0.033	VCV	4.38	1.0000	0.122	0.058
004	0.067	VCV	4.44	1.0000	0.245	0.092
005	0.086	VCV	4.50	1.0000	0.315	0.148
HMX	0.705	VCV	4.57	1.0000	2.572	0.213
006	0.021	VCT	4.65	1.0000	0.076	0.056
007	0.029	VCT	4.75	1.0000	0.106	0.078
008	0.091	VCT	4.85	1.0000	0.333	0.083
009	0.023	VCV	4.99	1.0000	0.082	0.059
010	0.014	VCV	5.01	1.0000	0.050	0.052
011	0.047	VCV	5.07	1.0000	0.173	0.072
012	0.284	VCV	5.21	1.0000	1.037	0.175
013	0.007	VCT	5.25	1.0000	0.026	0.052
014	0.238	VCV	5.39	1.0000	0.870	0.184
015	0.066	VCV	5.42	1.0000	0.242	0.170
016	0.112	VCV	5.47	1.0000	0.409	0.163
017	1.491	VCV	5.78	1.0000	5.438	0.315
018	0.270	VCB	5.94	1.0000	0.986	0.212
019	0.424	BCV	6.12	1.0000	1.545	0.190
RDX	10.880	VCV	7.07	1.0000	39.673	1.473
020	0.158	VCT	7.63	1.0000	0.577	0.155
021	0.762	VCV	8.11	1.0000	1.319	0.157
022	0.372	VCV	8.28	1.0000	1.356	0.165
023	0.393	VCV	8.62	1.0000	1.431	0.168
024	0.260	VCV	8.76	1.0000	0.950	0.170
025	0.182	VCB	8.90	1.0000	0.664	0.150
026	0.714	BCV	9.45	1.0000	2.603	0.191
027	0.170	VCV	9.54	1.0000	0.619	0.181
028	0.464	VCV	9.73	1.0000	1.691	0.185
029	0.218	VCV	9.86	1.0000	0.794	0.187
030	0.520	VCV	9.98	1.0000	1.897	0.242
TETRYL	0.788	VCV	10.20	1.0000	2.874	0.222
TNT	0.779	VCV	10.58	1.0000	2.841	0.206
031	5.917	VCV	11.88	1.0000	21.576	0.430
032	0.690	VCV	12.04	1.0000	2.516	0.402
033	3.357	VCV	12.48	1.0000	12.242	0.451
034	1.890	VCV	12.81	1.0000	6.891	0.511
035	3.602	VCV	13.30	1.0000	13.134	0.492
036	4.290	VCV	13.83	1.0000	15.643	0.551
2 6 DNT	4.849	VCV	14.51	1.0000	17.682	0.613
037	2.196	VCV	14.79	1.0000	8.009	0.597
038	3.780	VCV	15.19	1.0000	13.784	0.674
039	2.751	VCV	15.27	1.0000	10.031	0.712
040	2.237	VCV	15.51	1.0000	8.156	0.707
041	4.392	VCV	15.92	1.0000	16.016	0.694
042	1.796	VCV	16.19	1.0000	6.550	0.695
2 4 DNT	5.301	VCV	16.38	1.0000	19.331	0.765
043	3.076	VCV	16.95	1.0000	11.216	0.737
044	3.736	VCV	17.32	1.0000	13.622	0.786
045	10.570	VCV	18.14	1.0000	38.542	0.796
046	5.247	VCV	18.44	1.0000	22.780	0.807

Name	WT %	Pk	RRT	RRF	Area	Height
047	9.007	VCT	19.42	1.0000	32.842	0.826
	100.000					

File: IA009L   39324-55-14   14:30:35   3/26/1984
FE = 0   Inst: 8   Ch:0   Tray # 0   Pol +   Page 1

Run 18   RDX

Left Curon

	HMX	RDX	TETRYL TNT	2 6 DNT	2 4 DNT
	4 42	7 38	9 92 11 04	13 64	15 68

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2 0      4 0      6 0      8 0     10 0     12 0     14 0    16 0    18 0   20 0
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Min   0 0000 mv   % Smm   0 0000
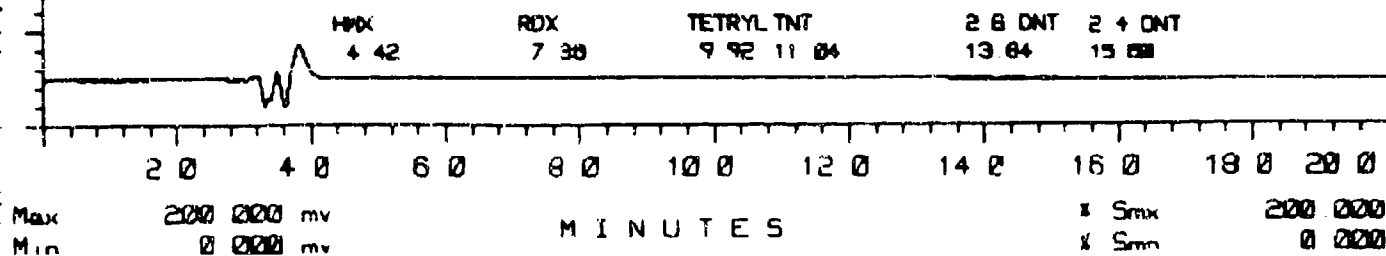
Area Analysis

File: IA009 39324-55-14   14:30:35   3/26/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 900   39324-55-14   14:02:27   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	0.026	VCV	4.24	1.0000	0.024	0.047
002	0.349	VCV	4.28	1.0000	0.329	0.119
HMX	0.681	VCV	4.42	1.0000	0.642	0.147
003	0.215	VCV	4.50	1.0000	0.203	0.140
004	0.172	VCV	4.55	1.0000	0.162	0.097
005	0.275	VCV	4.60	1.0000	0.259	0.112
006	0.180	VCV	4.68	1.0000	0.170	0.082
007	0.165	VCV	4.71	1.0000	0.155	0.095
008	0.147	VCV	4.74	1.0000	0.138	0.089
009	0.557	VCV	4.81	1.0000	0.525	0.134
010	0.651	VCV	4.89	1.0000	0.613	0.150
011	0.099	VCT	4.92	1.0000	0.093	0.086
012	0.060	TCT	4.99	1.0000	0.057	0.062
013	1.042	VCV	5.21	1.0000	0.982	0.152
014	0.941	VCV	5.33	1.0000	0.886	0.185
015	0.577	VCV	5.38	1.0000	0.544	0.176
016	0.252	VCV	5.42	1.0000	0.237	0.140
017	0.115	VCV	5.46	1.0000	0.108	0.105
018	8.570	VCB	5.82	1.0000	8.073	0.321
019	2.653	BCV	6.66	1.0000	2.500	0.165
RDX	1.954	VCV	7.36	1.0000	1.841	0.150
020	2.276	VCV	7.68	1.0000	2.145	0.160
021	1.573	VCV	8.17	1.0000	1.482	0.184
022	2.053	VCV	8.59	1.0000	1.934	0.165
023	3.209	BCV	8.96	1.0000	3.023	0.166
024	1.518	VCV	9.53	1.0000	1.431	0.164
TETRYL	3.379	VCV	9.92	1.0000	3.184	0.234
025	2.735	VCV	10.10	1.0000	2.577	0.220
026	4.720	VCV	10.34	1.0000	4.446	0.234
027	1.474	VCV	10.88	1.0000	1.388	0.249
TNT	5.520	VCV	11.04	1.0000	5.201	0.249
028	1.016	VCV	11.46	1.0000	0.957	0.197
029	8.923	VCV	11.59	1.0000	8.406	0.249
030	1.220	VCT	11.92	1.0000	1.150	0.124
031	14.195	VCV	12.64	1.0000	13.373	0.241
032	0.547	VCT	12.99	1.0000	0.516	0.123
033	0.864	VCT	13.31	1.0000	0.814	0.138
2 6 DNT	2.112	VCT	13.84	1.0000	1.989	0.159
034	1.028	VCV	14.55	1.0000	0.968	0.148
035	1.808	VCV	14.84	1.0000	1.703	0.159
036	1.614	VCV	15.08	1.0000	1.520	0.149
037	1.864	VCV	15.40	1.0000	1.756	0.159
2 4 DNT	6.086	VCV	15.80	1.0000	5.734	0.211
038	0.808	VCT	16.08	1.0000	0.761	0.121
039	0.795	VCV	16.83	1.0000	0.749	0.136
040	2.288	VCV	16.94	1.0000	2.156	0.152
041	3.307	VCV	17.50	1.0000	3.115	0.185
042	0.966	VCB	18.00	1.0000	0.910	0.160
043	0.783	BCV	19.36	1.0000	0.738	0.136
044	1.636	VCV	19.58	1.0000	1.542	0.137

                              100.000

File:IA008L   39324-55-15   14:09.30   3/26/1984
FE = 0   Inst: 8 Ch:0   Tray # 0   Pol +   Page 1

Run 18   RDX

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         |
         |                    HMX              TETRYL TNT       2 6 DNT2 4 DNT
         |                    4 37             9 75 11 14        14 66  16 79
         |_____/\/_____
         |___|___|___|___|___|___|___|___|___|___|___|___|___|___|___|___|___|___|___|___|___|
           2 0     4 0     6 0     8 0    10 0    12 0    14 0    16 0    18 0    20 0
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Min   0 0000 mv   # Smn   0 0000
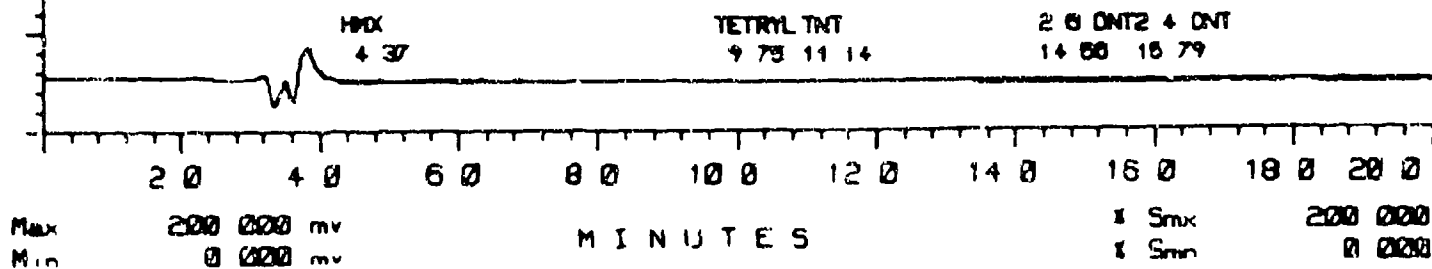
Area Analysis

File: IA008   39324-55-15   14:09:30   3/26/1984
Mthd: 1310   Inst 8   Chnl 0   Vial # 0 Stnd/Smpl % 100.000

Mthd: 900   39324-55-15   14:02:11   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
001	1.290	VCV	4.31	1.0000	1.070	0.413
HMX	2.972	VCV	4.37	1.0000	2.466	0.377
002	0.788	VCV	4.50	1.0000	0.654	0.230
003	1.121	VCV	4.60	1.0000	0.931	0.236
004	1.277	VCV	4.67	1.0000	1.059	0.250
005	0.378	VCV	4.73	1.0000	0.314	0.223
006	0.864	VCV	4.76	1.0000	0.717	0.267
007	2.084	VCV	4.82	1.0000	1.729	0.264
008	0.295	VCT	4.89	1.0000	0.245	0.089
009	0.078	VCV	5.03	1.0000	0.065	0.087
010	0.122	VCV	5.05	1.0000	0.101	0.090
011	0.108	VCV	5.08	1.0000	0.090	0.084
012	0.140	VCV	5.11	1.0000	0.116	0.079
013	0.217	VCV	5.19	1.0000	0.180	0.081
014	0.293	VCV	5.23	1.0000	0.243	0.119
015	0.287	VCV	5.28	1.0000	0.238	0.112
016	0.516	VCV	5.41	1.0000	0.428	0.107
017	0.086	VCV	5.46	1.0000	0.071	0.066
018	7.839	BCV	5.86	1.0000	6.505	0.222
019	8.903	VCV	6.32	1.0000	7.388	0.175
020	0.814	VCT	6.47	1.0000	0.675	0.125
RDX			7.10	1.0000		
021	2.556	VCV	7.72	1.0000	2.121	0.163
022	1.224	VCV	7.95	1.0000	1.016	0.150
023	1.136	VCV	8.19	1.0000	0.942	0.135
024	1.710	VCV	8.52	1.0000	1.419	0.131
025	3.273	BCV	9.01	1.0000	2.716	0.188
026	4.077	VCV	9.63	1.0000	3.383	0.189
TETRYL	2.699	VCV	9.75	1.0000	2.239	0.200
027	1.517	VCV	10.14	1.0000	1.258	0.151
028	0.388	VCT	10.22	1.0000	0.322	0.115
029	1.982	VCV	10.66	1.0000	1.645	0.156
030	1.567	VCV	10.85	1.0000	1.300	0.166
TNT	3.178	VCV	11.14	1.0000	2.637	0.193
031	7.487	VCV	11.82	1.0000	6.213	0.215
032	0.371	VCT	12.01	1.0000	0.308	0.117
033	1.740	VCV	12.48	1.0000	1.444	0.151
034	2.149	VCV	12.85	1.0000	1.784	0.171
035	1.717	VCV	13.08	1.0000	1.425	0.143
036	2.050	VCV	13.40	1.0000	1.701	0.143
037	0.880	VCV	13.71	1.0000	0.730	0.166
038	1.958	VCV	13.86	1.0000	1.625	0.157
039	2.129	VCV	14.30	1.0000	1.766	0.214
2 6 DNT	3.035	VCV	14.56	1.0000	2.518	0.173
040	3.328	VCV	14.79	1.0000	2.761	0.174
041	1.930	VCV	15.38	1.0000	1.602	0.150
042	2.257	VCV	15.67	1.0000	1.873	0.218
2 4 DNT	3.329	VCB	15.79	1.0000	4.422	0.174
043	0.993	BCV	18.38	1.0000	0.824	0.133
044	4.866	VCV	18.72	1.0000	4.038	0.160
045	0.563	VCT	19.30	1.0000	0.467	0.116
046	1.443	VCV	19.7	1.0000	1.197	0.196

378

File: IA008   39324-55-15   14:09:30   3/26/1984
Mthd: 900   39324-55 .5   14:02:11   3/27/1984

Name	WT %	Pk	RRT	RRF	Area	Height
	100.000					

Name	WT %	Pk	RRT	RRF	Area	Height

DATA SHEETS

The following section contains the data sheets summarizing the analytical results from all the tests conducted in this subtask.

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Project Number G7920-0430   memo[

✻ Battelle
Columbus Laboratories

Internal Distribution

AJ Killmeyer
BC Garrett
G7920 Files
E J Mezey

Date   1/25/84

To   H Benecke ▓▓▓▓ BCG

From   AJ Killmeyer/BC Garrett

Subject   Analytical Results of Subtask 5

→ Recovery Baseline

● **EXPERIMENTAL CONDITIONS**

LABORATORY RECORD BOOK 38759
NUMBER/PAGES: pp 49-50

Date of trial   1/13/54
Explosive: ( )2,4 DNT ( )2,6 DNT (X)TNT ( )Tetryl ( )RDX ( )HMX
N/A ~~Decon Technique:~~ ( )Hot Gases   ( )Chemical 1
                         ( )Freon/RADKLEEN   ( )Chemical 2
                                             ( )Chemical 3

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _1_ coupons): 343
2. Explosive in rinse solution (mg): ——
3. Explosive in atmosphere in chamber (mg): ——

I. Calculation of Residual Explosive on Coupons

   A. Surface area per coupon: 81 cm^2 (8.1 × 10^{-3} m^2)
   B. Contamination density: _4.94_ mg/cm^2
   C. Extraction volume per coupon: _100_ mL
   D. Recovery efficiency: _85.75_ %
   E. Extraction concentration factor: _500_
   F. Analysis results for extracts:

      1. Coupon 1   _6.86_ ug/mL
      2. Coupon 2   —— ug/mL
      3. Coupon 3   —— ug/mL

   G. Residual agent per coupon [(F×E×C×10^{-3})/D]:

      1. Coupon 1   _343_ mg → VS 400
      2. Coupon 2   —— mg
      3. Coupon 3   —— mg

$$\frac{343}{400}(100) = 85.75\,\%$$

II. Calculation of Explosive in Rinse Solution

   N/A
   A. Volume rinse solution: _____ mL
   B. Extraction concentration factor: _____
   C. Analytical results: _____ ug/mL
   D. Mass in rinse solution (A×B×C×10^{-3}): _____ mg

III. Calculation of Explosive in Atmosphere in Chamber

   N/A
   A. Impinger volume (total of four): _____ mL
   B. Analysis results: _____ ug/mL
   C. Mass in impingers (A×B×10^{-3}): _____ mg
   D. Trap volume: _____ mL
   E. Extraction concentration factor: _____
   F. Analysis result: _____ ug/mL
   G. Mass in trap (F×D×E×10^{-3}): _____ mg
   H. Total in atmosphere (C+G): _____ mg

❄️ **Battelle**
Columbus Laboratories

Date   1/25/84

To   H Benecke,

From   AJ Killmeyer/BC Garrett

Subject   Analytical Results of Subtask 5

→ Recovery Baseline

- **EXPERIMENTAL CONDITIONS**

  Date of trial 1/13/84

  Explosive: ( )2,4 DNT (X)2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX

  N/A Decon Technique: ( )Hot Gases   ( )Chemical 1
                         ( )Freon/RADKLEEN   ( )Chemical 2
                                               ( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: 49-50

- **SUMMARY OF RESULTS**

  1. Residual explosive per coupon (mg)(average of 1 coupons): 396 mg/g
  2. Explosive in rinse solution (mg): —
  3. Explosive in atmosphere in chamber (mg): —

I. **Calculation of Residual Explosive on Coupons**

   A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
   B. Contamination density: 4,94 mg/cm^2
   C. Extraction volume per coupon: 100 mL
   D. Recovery efficiency: 99,0 %
   E. Extraction concentration factor: 500
   F. Analysis results for extracts:

      1. Coupon 1 7,93 ug/mL
      2. Coupon 2 — ug/mL
      3. Coupon 3 — ug/mL

   G. Residual agent per coupon $[(F \times E \times C \times 10^{-3})/D]$:

      1. Coupon 1 396 mg → VS 400
      2. Coupon 2 — mg
      3. Coupon 3 — mg

$$\frac{396}{400}(100) = 99.0\%$$

II. **Calculation of Explosive in Rinse Solution**

   A. Volume rinse solution: _____ mL
   B. Extraction concentration factor: _____
   N/A C. Analytical results: _____ ug/mL
   D. Mass in rinse solution $(A \times B \times C \times 10^{-3})$: _____ mg

III. **Calculation of Explosive in Atmosphere in Chamber**

   A. Impinger volume (total of four): _____ mL
   B. Analysis results: _____ ug/mL
   C. Mass in impingers $(A \times B \times 10^{-3})$: _____ mg
   D. Trap volume: _____ mL
   N/A E. Extraction concentration factor: _____
   F. Analysis result: _____ ug/mL
   G. Mass in trap $(F \times D \times E \times 10^{-3})$: _____ mg
   H. Total in atmosphere (C+G): _____ mg

Battelle
Columbus Laboratories

Date   1/25/84

To   H Benecke/███████

From   AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5

→ Recovery Baseline

- **EXPERIMENTAL CONDITIONS**

  Date of trial 1/13/84

  Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT (X)Tetryl ( )RDX ( )HMX

  N/A Decon Technique: ( )Hot Gases   ( )Chemical 1
                        ( )Freon/RADKLEEN   ( )Chemical 2
                                            ( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: pp 49-50

- **SUMMARY OF RESULTS**

  1. Residual explosive per coupon (mg)(average of $\underline{1}$ coupons): $\underline{507}$
  2. Explosive in rinse solution (mg): _____
  3. Explosive in atmosphere in chamber (mg): _____

  ### I. Calculation of Residual Explosive on Coupons

  A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
  B. Contamination density: $\underline{4.94}$ mg/cm^2
  C. Extraction volume per coupon: $\underline{100}$ mL
  D. Recovery efficiency: $\underline{126.8\%}$
  E. Extraction concentration factor: $\underline{500}$
  F. Analysis results for extracts:

      1. Coupon 1 $\underline{10.14}$ µg/mL
      2. Coupon 2 ____ µg/mL
      3. Coupon 3 ____ µg/mL

  G. Residual agent per coupon [(FxExCx10^{-3})/D]:

      1. Coupon 1 $\underline{507}$ mg → vs 400
      2. Coupon 2 ____ mg
      3. Coupon 3 ____ mg

  $$\frac{507}{400}(100) = 126.8\%$$

  ### II. Calculation of Explosive in Rinse Solution

  A. Volume rinse solution: _____ mL
  B. Extraction concentration factor: _____
  N/A C. Analytical results: _____ ug/mL
  D. Mass in rinse solution (AxBxCx10^{-3}): _____ mg

  ### III. Calculation of Explosive in Atmosphere in Chamber

  A. Impinger volume (total of four): _____ mL
  B. Analysis results: _____ µg/mL
  C. Mass in impingers (AxBx10^{-3}): _____ mg
  N/A D. Trap volume: _____ mL
  E. Extraction concentration factor: _____
  F. Analysis result: _____ µg/mL
  G. Mass in trap (FxDxEx10^{-3}): _____ mg
  H. Total in atmosphere (C+G): _____ mg

❋ Battelle
Columbus Laboratories

Internal Distribution

AJ Killmeyer
BC Garrett
G7920 Files

E.J. Mezey

Date   1/25/84

To   H Benecke/

From   AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5

● **EXPERIMENTAL CONDITIONS**

Date of trial ___1/5/84___

~~Explosive~~: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX

blank

Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                       ( )Chemical 3

LABORATORY RECORD BOOK 3895-9
NUMBER/PAGES: pp 51,53

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _2_ coupons): _<0.05 mg_
2. Explosive in rinse solution (mg): _____
3. Explosive in atmosphere in chamber (mg): _____

I. Calculation of Residual Explosive on Coupons

   A. Surface area per coupon: 81 cm^2 ($8.1 \times 10^{-3} \text{ m}^2$)
   B. Contamination density: ___0___ mg/cm^2
   C. Extraction volume per coupon: _100_ mL
   D. Recovery efficiency: _____
   E. Extraction concentration factor: _____
   F. Analysis results for extracts:

      1. Coupon 1 _____ µg/mL
      2. Coupon 2 ___<0.5___ µg/mL
      3. Coupon 3 ___<0.5___ µg/mL

   G. Residual agent per coupon $[(F \times E \times C \times 10^{-3})/D]$:

      1. Coupon 1 _____ mg
      2. Coupon 2 ___<0.05___ mg
      3. Coupon 3 ___<0.05___ mg

II. Calculation of Explosive in Rinse Solution

    N/A
    A. Volume rinse solution: _____ mL
    B. Extraction concentration factor: _____
    C. Analytical results: _____ µg/mL
    D. Mass in rinse solution ($A \times B \times C \times 10^{-3}$): _____ mg

III. Calculation of Explosive in Atmosphere in Chamber

     N/A
     A. Impinger volume (total of four): _____ mL
     B. Analysis results: _____ µg/mL
     C. Mass in impingers ($A \times B \times 10^{-3}$): _____ mg
     D. Trap volume: _____ mL
     E. Extraction concentration factor: _____
     F. Analysis result: _____ µg/mL
     G. Mass in trap ($F \times D \times E \times 10^{-3}$): _____ mg
     H. Total in atmosphere ($C + G$): _____ mg

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E J Mezey

Date   1/25/84

To   H Benecke

From   AJ Killmeyer/BC Garrett

Subject   Analytical Results of Subtask 5   Run 1

● **EXPERIMENTAL CONDITIONS**

Date of trial 1/10/84
Explosive: (X)2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX
Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                        ( )Chemical 3

LABORATORY RECORD BOOK
NUMBER/PAGES: 38953
55

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of 3 coupons): <0.05
2. Explosive in rinse solution (mg): 0.16
3. Explosive in atmosphere in chamber (mg): —

I. **Calculation of Residual Explosive on Coupons**

   A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
   B. Contamination density: _____4.94_____ mg/cm^2
   C. Extraction volume per coupon: _____100_____ mL
   D. Recovery efficiency: _____100%_____
   E. Extraction concentration factor: ___—___
   F. Analysis results for extracts:

      1. Coupon 1   <0.5   µg/mL
      2. Coupon 2   <0.5   µg/mL
      3. Coupon 3   <0.5   µg/mL

   G. Residual agent per coupon [(FxExCx10^{-3})/D]:

      1. Coupon 1   <0.05   mg
      2. Coupon 2   <0.05   mg
      3. Coupon 3   <0.05   mg

II. **Calculation of Explosive in Rinse Solution**

   A. Volume rinse solution: _____200_____ mL
   B. Extraction concentration factor: ___—___
   C. Analytical results: _____0.29_____ µg/mL
   D. Mass in rinse solution (AxBxCx10^{-3}): _____0.16_____ mg

III. **Calculation of Explosive in Atmosphere in Chamber**

   A. Impinger volume (total of four): ___—___ mL
   B. Analysis results: _____2.48_____ µg/mL
   C. Mass in impingers (AxBx10^{-3}): ___—___ mg
   D. Trap volume: ___—___ mL
   E. Extraction concentration factor: _____
   F. Analysis result: _____1.47_____ µg/mL
   G. Mass in trap (FxDxEx10^{-3}): ___—___ mg
   H. Total in atmosphere (C+G): ___—___ mg

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To   H Benecke/~~████████~~ _BCG_

From   AJ Killmeyer/BC Garrett

Subject   Analytical Results of Subtask 5

● **EXPERIMENTAL CONDITIONS**

Date of trial 1/16/84
Explosive: (X)2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX
Decon Technique: (X)Hot Gases   ( )Chemical 1
                 ( )Freon/RADKLEEN   ( )Chemical 2
                                     ( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: ___ 52 55

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _3_ coupons): <0.05 mg
2. Explosive in rinse solution (mg): <0.125 mg
3. Explosive in atmosphere in chamber (mg): 3.86 mg × 0.04 = 1.5

I. **Calculation of Residual Explosive on Coupons**

A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
B. Contamination density: 4.94 mg/cm^2
C. Extraction volume per coupon: 100 mL
D. Recovery efficiency: 100%
E. Extraction concentration factor: —
F. Analysis results for extracts:

   1. Coupon 1   <0.5 ug/mL
   2. Coupon 2   <0.5 ug/mL
   3. Coupon 3   <0.5 ug/mL

G. Residual agent per coupon [(FxExCx10^{-3})/D]:

   1. Coupon 1   <0.05 mg
   2. Coupon 2   <0.05 mg
   3. Coupon 3   <0.05 mg

II. **Calculation of Explosive in Rinse Solution**

A. Volume rinse solution: 250 mL
B. Extraction concentration factor: —
C. Analytical results: <0.5 ug/mL
D. Mass in rinse solution (AxBxCx10^{-3}): <0.125 mg

III. **Calculation of Explosive in Atmosphere in Chamber**

A. Impinger volume (total of four): 250 mL
B. Analysis results: 13.70 ug/mL
C. Mass in impingers (AxBx10^{-3}): 3.43 mg
D. Trap volume: 7.0 mL
E. Extraction concentration factor: —
F. Analysis result: 1.3 ug/mL
G. Mass in trap (FxDxEx10^{-3}): 0.43 mg
H. Total in atmosphere (C+G): 3.86 mg

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Run 3

Date 1/25/84

To H Benecke ▓▓▓▓▓▓▓▓

From AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5

- **EXPERIMENTAL CONDITIONS**

  Date of trial 1/20/84
  Explosive: (X)2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX
  Decon Technique: (X)Hot Gases   ( )Chemical 1
                    ( )Freon/RADKLEEN   ( )Chemical 2
                                        ( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: p 57

- **SUMMARY OF RESULTS**

  1. Residual explosive per coupon (mg)(average of _3_ coupons): _< 0.05_
  2. Explosive in rinse solution (mg): _0.23_
  3. Explosive in atmosphere in chamber (mg): _5.46_ × _.64 = 9/_

  I. Calculation of Residual Explosive on Coupons

  A. Surface area per coupon: 81 cm^2 (8.1 × 10^{-3} m^2)
  B. Contamination density: _4.94_ mg/cm^2
  C. Extraction volume per coupon: _100_ mL
  D. Recovery efficiency: _100%_
  E. Extraction concentration factor: _—_
  F. Analysis results for extracts:

     1. Coupon 1 _< 0.5_ ug/mL
     2. Coupon 2 _< 0.5_ ug/mL
     3. Coupon 3 _< 0.5_ ug/mL

  G. Residual agent per coupon [(F×E×C×10^{-3})/D]:

     1. Coupon 1 _< 0.05_ mg
     2. Coupon 2 _< 0.05_ mg
     3. Coupon 3 _< 0.05_ mg

  II. Calculation of Explosive in Rinse Solution

  A. Volume rinse solution: _250_ mL
  B. Extraction concentration factor: _—_
  C. Analytical results: _0.93_ ug/mL
  D. Mass in rinse solution (A×B×C×10^{-3}): _0.23_ mg

  III. Calculation of Explosive in Atmosphere in Chamber

  A. Impinger volume (total of four): _250_ mL
  B. Analysis results: _21.92_ ug/mL
  C. Mass in impingers (A×B×10^{-3}): _5.46_ mg
  D. Trap volume: _250_ mL
  E. Extraction concentration factor: _—_
  F. Analysis result: _< 0.5_ ug/mL
  G. Mass in trap (F×D×E×10^{-3}): _< 0.125_ mg
  H. Total in atmosphere (C+G): _5.46_ mg

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Subject Analytical Results of Subtask 5

Run # 4

● **EXPERIMENTAL CONDITIONS**

Date of trial 1/26/84

Explosive: (X)2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX

Decon Technique: (X)Hot Gases ( )Chemical 1
( )Freon/RADKLEEN ( )Chemical 2
( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: 5F-9

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of 3 coupons): < 0.010
2. Explosive in rinse solution (mg): 0.24
3. Explosive in atmosphere in chamber (mg): 14 × 16.54 = 232 mg

I. Calculation of Residual Explosive on Coupons

A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
B. Contamination density: <1.2×10^{-4} mg/cm^2
C. Extraction volume per coupon: 100 mL
D. Recovery efficiency: 100%
E. Extraction concentration factor: —
F. Analysis results for extracts:

   1. Coupon 1 < 0.10 ug/mL
   2. Coupon 2 < 0.10 ug/mL
   3. Coupon 3 < 0.10 ug/mL

G. Residual agent per coupon [(FxExCx10^{-3})/D]:

   1. Coupon 1 < 0.010 mg
   2. Coupon 2 < 0.010 mg
   3. Coupon 3 < 0.010 mg

II. Calculation of Explosive in Rinse Solution

A. Volume rinse solution: 250 mL
B. Extraction concentration factor: —
C. Analytical results: 0.95 ug/mL
D. Mass in rinse solution (AxBxCx10^{-3}): 0.24 mg

III. Calculation of Explosive in Atmosphere in Chamber

A. Impinger volume (total of four): 250 mL
B. Analysis results: 55 ug/mL
C. Mass in impingers (AxBx10^{-3}): 14 mg
D. Trap volume: 250 mL
E. Extraction concentration factor: —
F. Analysis result: < 0.10 ug/mL
G. Mass in trap (FxDxEx10^{-3}): < 0.025 mg
H. Total in atmosphere (C+G): 14 mg

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From AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5   Run 5

● **EXPERIMENTAL CONDITIONS**

Date of trial _1/27/84_

Explosive: (X)2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX

Decon Technique: (X)Hot Gases   ( )Chemical 1
                   ( )Freon/RADKLEEN   ( )Chemical 2
                                       ( )Chemical 3

LABORATORY RECORD BOOK 32959
NUMBER/PAGES: ▮▮ 5▮▮ - 9

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _3_ coupons): _< 0.010_
2. Explosive in rinse solution (mg): _0.030_
3. Explosive in atmosphere in chamber (mg): _3.5 × ▮▮ = 10▮,_

I. Calculation of Residual Explosive on Coupons

A. Surface area per coupon: 81 cm^2 ($8.1 \times 10^{-3} \text{ m}^2$)
B. Contamination density: _$< 1.2 \times 10^{-4}$_ mg/cm^2
C. Extraction volume per coupon: _100_ mL
D. Recovery efficiency: _100%_
E. Extraction concentration factor: _—_
F. Analysis results for extracts:

   1. Coupon 1 _< 0.10_ ug/mL
   2. Coupon 2 _< 0.10_ ug/mL
   3. Coupon 3 _< 0.10_ ug/mL

G. Residual agent per coupon [$(F \times E \times C \times 10^{-3})/D$]:

   1. Coupon 1 _< 0.010_ mg
   2. Coupon 2 _< 0.010_ mg
   3. Coupon 3 _< 0.010_ mg

II. Calculation of Explosive in Rinse Solution

A. Volume rinse solution: _250_ mL
B. Extraction concentration factor: _—_
C. Analytical results: _0.12_ ug/mL
D. Mass in rinse solution ($A \times B \times C \times 10^{-3}$): _0.05_ mg

III. Calculation of Explosive in Atmosphere in Chamber

A. Impinger volume (total of four): _250_ mL
B. Analysis results: _14_ ug/mL
C. Mass in impingers ($A \times B \times 10^{-3}$): _3.5_ mg
D. Trap volume: _2▮▮_ mL
E. Extraction concentration factor: _—_
F. Analysis result: _< 0.10_ ug/mL
G. Mass in trap ($F \times D \times E \times 10^{-3}$): _< 0.025_ mg
H. Total in atmosphere (C+G): _3.5_ mg

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Date 2/9/84

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From AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5

*Run 6 LRK 39324
PB 18;*

- **EXPERIMENTAL CONDITIONS**

  Date of trial 2/4/84

  Explosive: (x)2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX

  Decon Technique: (x)Hot Gases   ( )Chemical 1
                    ( )Freon/RADKLEEN   ( )Chemical 2
                                            ( )Chemical 3

  LABORATORY RECORD BOOK 38959
  NUMBER/PAGES: 64

- **SUMMARY OF RESULTS**

  1. Residual explosive per coupon (mg)(average of _3_ coupons): < 0.010
  2. Explosive in rinse solution (mg): < 0.025
  3. Explosive in atmosphere in chamber (mg): 9.6

  I. Calculation of Residual Explosive on Coupons

  A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
  B. Contamination density: _around_ 5 mg/cm^2
  C. Extraction volume per coupon: 100 mL
  D. Recovery efficiency: 100%
  E. Extraction concentration factor: —
  F. Analysis results for extracts:

     1. Coupon 1   < 0.10   ug/mL
     2. Coupon 2   < 0.10   ug/mL
     3. Coupon 3   < 0.10   ug/mL

  G. Residual agent per coupon [(FxExCx10^{-3})/D]:

     1. Coupon 1   < 0.010 mg
     2. Coupon 2   < 0.010 mg
     3. Coupon 3   < 0.010 mg

  II. Calculation of Explosive in Rinse Solution

  A. Volume rinse solution: 250 mL
  B. Extraction concentration factor: —
  C. Analytical results: < 0.10 ug/mL
  D. Mass in rinse solution (AxBxCx10^{-3}): < 0.025 mg

  III. Calculation of Explosive in Atmosphere in Chamber

  A. Impinger volume (total of four): 250 mL
  B. Analysis results: 38 ug/mL
  C. Mass in impingers (AxBx10^{-3}): 9.5 mg
  D. Trap volume: 250 mL
  E. Extraction concentration factor: —
  F. Analysis result: 0.28 ug/mL
  G. Mass in trap (FxDxEx10^{-3}): 0.095 mg
  H. Total in atmosphere (C+G): 9.6 mg

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Project Number G7920-0430   memo.

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Run 7

Date 3/1/84

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Subject Analytical Results of Subtask 5

● **EXPERIMENTAL CONDITIONS**

Date of trial 2/7/84

Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX (X)HMX

Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                      ( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: 66, 67

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _3_ coupons) ___168___
2. Explosive in rinse solution (mg): ___5.73___
3. Explosive in atmosphere in chamber (mg): ___0.323___

I. Calculation of Residual Explosive on Coupons

   A. Surface area per coupon: 81 cm^2 ($8.1 \times 10^{-3} \text{ m}^2$)
   B. Contamination density: ___4.4___ mg/cm^2
   C. Extraction volume per coupon: ___2500___ mL
   D. Recovery efficiency: ___100%___
   E. Extraction concentration factor: ___—___
   F. Analysis results for extracts:

      1. Coupon 1 ___77.3___ µg/mL
      2. Coupon 2 ___40.0___ µg/mL
      3. Coupon 3 ___84.7___ µg/mL

   G. Residual agent per coupon $[(F \times E \times C \times 10^{-3})/D]$:

      1. Coupon 1 ___193___ mg
      2. Coupon 2 ___100___ mg
      3. Coupon 3 ___212___ mg

II. Calculation of Explosive in Rinse Solution

    A. Volume rinse solution: ___250___ mL
    B. Extraction concentration factor: ___—___
    C. Analytical results: ___22.9___ µg/mL
    D. Mass in rinse solution $(A \times B \times C \times 10^{-3})$: ___5.73___ mg

III. Calculation of Explosive in Atmosphere in Chamber

    A. Impinger volume (total of four): ___250___ mL
    B. Analysis results: ___1.29___ µg/mL
    C. Mass in impingers $(A \times B \times 10^{-3})$: ___0.323___ mg
    D. Trap volume: ___250___ mL
    E. Extraction concentration factor: ___—___
    F. Analysis result: ___<0.100___ µg/mL
    G. Mass in trap $(F \times D \times E \times 10^{-3})$: ___<0.0250___ mg
    H. Total in atmosphere (C+G): ___0.323___ mg

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Subject Analytical Results of Subtask 5

Run 8

● **EXPERIMENTAL CONDITIONS**

Date of trial 2/15/84

Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX (x)HMX

Decon Technique: (x)Hot Gases   ( )Chemical 1
                 ( )Freon/RADKLEEN   ( )Chemical 2
                                     ( )Chemical 3

LABORATORY RECORD BOOK NUMBER/PAGES: 23-25

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of 3 coupons): <0.0100
2. Explosive in rinse solution (mg): 3.30
3. Explosive in atmosphere in chamber (mg): <0.0500

I. Calculation of Residual Explosive on Coupons

A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
B. Contamination density: 4.94 mg/cm^2
C. Extraction volume per coupon: 100 mL
D. Recovery efficiency: 100%
E. Extraction concentration factor: —
F. Analysis results for extracts:

   1. Coupon 1 left   <0.100 ug/mL
   2. Coupon 2 middle <0.100 ug/mL
   3. Coupon 3 right <0.100 ug/mL

G. Residual agent per coupon [(FxExCx10^{-3})/D]:

   1. Coupon 1 left   <0.0100 mg
   2. Coupon 2 middle <0.0100 mg
   3. Coupon 3 right <0.0100 mg

II. Calculation of Explosive in Rinse Solution

A. Volume rinse solution: 250 mL
B. Extraction concentration factor: —
C. Analytical results: 13.2 µg/mL
D. Mass in rinse solution (AxBxCx10^{-3}): 3.30 mg

III. Calculation of Explosive in Atmosphere in Chamber

A. Impinger volume (total of four): 250 mL
B. Analysis results: <0.100 µg/mL
C. Mass in impingers (AxBx10^{-3}): <0.0250 mg
D. Trap volume: 250 mL
E. Extraction concentration factor: —
F. Analysis result: <0.100 µg/mL
G. Mass in trap (FxDxEx10^{-3}): <0.0250 mg
H. Total in atmosphere (C+G): <0.0500 mg

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Run #9

Date   3/1/84

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Subject   Analytical Results of Subtask 5

● **EXPERIMENTAL CONDITIONS**

Date of trial 2/20/84

Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX (X)HMX
Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                      ( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: 71

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _3_ coupons): _59.9_
2. Explosive in rinse solution (mg): _90.5_
3. Explosive in atmosphere in chamber (mg): _0.745_

I. Calculation of Residual Explosive on Coupons

A. Surface area per coupon: 81 cm^2 ($8.1 \times 10^{-3} \text{ m}^2$)
B. Contamination density: _4.94_ mg/cm^2
C. Extraction volume per coupon: _2500_ mL
D. Recovery efficiency: _100%_
E. Extraction concentration factor: _—_
F. Analysis results for extracts:

   1. Coupon 1 left _19.1_ µg/mL
   2. Coupon 2 middle _43.9_ µg/mL
   3. Coupon 3 right _8.75_ µg/mL

G. Residual agent per coupon [$(F \times E \times C \times 10^{-3})/D$]:

   1. Coupon 1 left _47.8_ mg
   2. Coupon 2 middle _110_ mg
   3. Coupon 3 right _21.9_ mg

II. Calculation of Explosive in Rinse Solution

A. Volume rinse solution: _250_ mL
B. Extraction concentration factor: _—_
C. Analytical results: _36.2_ µg/mL
D. Mass in rinse solution ($A \times B \times C \times 10^{-3}$): _90.5_ mg

III. Calculation of Explosive in Atmosphere in Chamber

A. Impinger volume (total of four): _250_ mL
B. Analysis results: _0.298_ µg/mL
C. Mass in impingers ($A \times B \times 10^{-3}$): _0.745_ mg
D. Trap volume: _250_ mL
E. Extraction concentration factor: _—_
F. Analysis result: _<0.100_ µg/mL
G. Mass in trap ($F \times D \times E \times 10^{-3}$): _<0.0250_ mg
H. Total in atmosphere (C+G): _0.745_ mg

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Date 3/1/84

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From AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5

- **EXPERIMENTAL CONDITIONS**

  Date of trial 2/22/84

  Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX (x)HMX

  Decon Technique: (x)Hot Gases ( )Chemical 1
  ( )Freon/RADKLEEN ( )Chemical 2
  ( )Chemical 3

  **LABORATORY RECORD BOOK 38959
  NUMBER/PAGES: 72**

- **SUMMARY OF RESULTS**

  1. Residual explosive per coupon (mg)(average of 3 coupons): 71.4
  2. Explosive in rinse solution (mg): 159
  3. Explosive in atmosphere in chamber (mg): 1.42

  I. Calculation of Residual Explosive on Coupons

  A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
  B. Contamination density: 4.94 mg/cm^2
  C. Extraction volume per coupon: 5000 mL
  D. Recovery efficiency: 100%
  E. Extraction concentration factor: —
  F. Analysis results for extracts:

     1. Coupon 1 left 22.3 µg/mL
     2. Coupon 2 middle 14.0 µg/mL
     3. Coupon 3 right 6.41 µg/mL

  G. Residual agent per coupon [(FxExCx10^{-3})/D]:

     1. Coupon 1 left 112 mg
     2. Coupon 2 middle 70 mg
     3. Coupon 3 right 32.1 mg

  II. Calculation of Explosive in Rinse Solution

  A. Volume rinse solution: 25000 mL
  B. Extraction concentration factor: —
  C. Analytical results: 6.34 µg/mL
  D. Mass in rinse solution (AxBxCx10^{-3}): 159 mg

  III. Calculation of Explosive in Atmosphere in Chamber

  A. Impinger volume (total of four): 250 mL
  B. Analysis results: 0.892 µg/mL
  C. Mass in impingers (AxBx10^{-3}): 0.223 mg
  D. Trap volume: 250 mL
  E. Ext action concentration factor: —
  F. Analysis result: 5.01 µg/mL
  G. Mass in trap (FxDxEx10^{-3}): 1.25 mg
  H. Total in atmosphere (C+G): 1.42 mg

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Date 3/7/84

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from AJ Killmeyer/BC Garrett

(Run 11)

Subject Analytical Results of Subtask 5

● EXPERIMENTAL CONDITIONS

Date of trial 2/27/84

Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX (X)HMX

Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                        ( )Chemical 3

LABORATORY RECORD BOOK 38959
NUMBER/PAGES: 75

● SUMMARY OF RESULTS

1. Residual explosive per coupon (mg)(average of _3_ coupons): _21.9_
2. Explosive in rinse solution (mg): _93.5_
3. Explosive in atmosphere in chamber (mg): _0.319_

I. Calculation of Residual Explosive on Coupons

A. Surface area per coupon: 81 cm^2 $(8.1 \times 10^{-3} \text{ m}^2)$
B. Contamination density: _4.94_ mg/cm^2
C. Extraction volume per coupon: _100_ mL
D. Recovery efficiency: _100%_
E. Extraction concentration factor: _—_
F. Analysis results for extracts:
   1. left Coupon 1 _32.7_ ug/mL
   2. Coupon 2 _19.5_ ug/mL
   3. Coupon 3 _167_ ug/mL
G. Residual agent per coupon [$(F \times E \times C \times 10^{-3})/D$]:
   1. left Coupon 1 _3.27_ mg
   2. Coupon 2 _1.95_ mg
   3. Coupon 3 _16.7_ mg

II. Calculation of Explosive in Rinse Solution

A. Volume rinse solution: _500_ mL
B. Extraction concentration factor: _—_
C. Analytical results: _187_ ug/mL
D. Mass in rinse solution ($A \times B \times C \times 10^{-3}$): _93.5_ mg

III. Calculation of Explosive in Atmosphere in Chamber

A. Impinger+trap volume (total of four): _750_ mL
B. Analysis results: _0.425_ ug/mL
C. Mass in impingers ($A \times B \times 10^{-3}$): _0.319_ mg
D. Trap volume: _—_ mL
E. Extraction concentration factor: _—_
F. Analysis result: _—_ ug/mL
G. Mass in trap ($F \times D \times E \times 10^{-3}$): _—_ mg
H. Total in atmosphere (C+G): _0.319_ mg

Battelle
Columbus Laboratories

Project Number __G7920-0430__

Internal Distribution

AJ Killmeyer
BC Garrett
G7920 Files

Date __3/9/84__

To   H Benecke [redacted]

From AJ Killmeyer/BC Garrett

R_{un} 12

Subject Analytical Results of Subtask 5

● **EXPERIMENTAL CONDITIONS**   **LABORATORY RECORD BOOK** 38959
                                                NUMBER/PAGES: __77__

Date of trial __3/5/84__
Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl ( )RDX (X)HMX
Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                      ( )Chemical 3

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg){average of __3__ coupons): __0.044__
2. Explosive in rinse solution (mg): __2.54__
3. Explosive in atmosphere in chamber (mg): __2.19__

I. Calculation of Residual Explosive on Coupons

A. Surface area per coupon: 81 cm^2 (8.1×10^{-3} m^2)
B. Contamination density: __5.21__ mg/cm^2
C. Extraction volume per coupon: __100__ mL
D. Recovery efficiency: __100%__
E. Extraction concentration factor: __—__
F. Analysis results for extracts:

left→ 1. Coupon 1 __0.716__ µg/mL
mid→ 2. Coupon 2 __0.202__ µg/mL
r→ 3. Coupon 3 __0.408__ µg/mL

G. Residual agent per coupon [(FxExCx10^{-3})/D]:

left→ 1. Coupon 1 __0.0716__ mg
2. Coupon 2 __0.0202__ mg
right→ 3. Coupon 3 __0.0408__ mg

II. Calculation of Explosive in Rinse Solution

A. Volume rinse solution: __500__ mL
B. Extraction concentration factor: __—__
C. Analytical results: __5.07__ µg/mL
D. Mass in rinse solution (AxBxCx10^{-3}): __2.54__ mg

III. Calculation of Explosive in Atmosphere in Chamber

A. Impinger volume (total of four): __250__ mL
B. Analysis results: __2.57__ µg/mL
C. Mass in impingers (AxBx10^{-3}): __0.643__ mg
D. Trap volume: __250__ mL
E. Extraction concentration factor: __—__
F. Analysis result: __6.19__ µg/mL
G. Mass in trap (FxDxEx10^{-3}): __1.55__ mg
H. Total in atmosphere (C+G): __2.19__ mg

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Project Number G7920-0430

Battelle
Columbus Laboratories

R— A

Date **March 27, 1984**

To H Benecke/D Hopper

From AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5

- **EXPERIMENTAL CONDITIONS** 19?

  Date of trial 3/18/84

  Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT (X)Tetryl ( )RDX ( )HMX

  Decon Technique: (X)Hot Gases   ( )Chemical 1
                    ( )Freon/RADKLEEN   ( )Chemical 2
                                           ( )Chemical 3

  LABORATORY RECORD BOOK 3F9J-9
  NUMBER/PAGES: 82

- **SUMMARY OF RESULTS**

  1. Residual explosive per coupon (mg)(average of _3_ coupons): _0.0753_
  2. Explosive in rinse solution (mg): _<0.0250_
  3. Explosive in atmosphere in chamber (mg): _0.743_

  I. Calculation of Residual Explosive on Coupons

  A. Surface area per coupon: 81 cm^2 (8.1 x 10^{-3} m^2)
  B. Contamination density: _5_ mg/cm^2
  C. Extraction volume per coupon: _100_ mL
  D. Recovery efficiency: _100 %_
  E. Extraction concentration factor: _—_
  F. Analysis results for extracts:

  39324-48-2 1. Coupon 1 _0.834_ µg/mL
  39324-48-4 2. Coupon 2 _1.01_ µg/mL
  39324-48-6 3. Coupon 3 _0.414_ µg/mL

  G. Residual agent per coupon [(FxExCx10^{-3})/D]:

  1. Coupon 1 _0.0834_ mg
  2. Coupon 2 _0.101_ mg
  3. Coupon 3 _0.0414_ mg

  II. Calculation of Explosive in Rinse Solution

  A. Volume rinse solution: _250_ mL
  B. Extraction concentration factor: _—_
  C. Analytical results: _<0.100_ µg/mL
  D. Mass in rinse solution (AxBxCx10^{-3}): _<0.0250_ mg

  III. Calculation of Explosive in Atmosphere in Chamber

  A. Impinger volume (total of four): _250_ mL
  B. Analysis results: _2.97_ µg/mL
  C. Mass in impingers (AxBx10^{-3}): _0.743_ mg
  D. Trap volume: _250_ mL
  E. Extraction concentration factor: _____
  F. Analysis result: _<0.100_ µg/mL
  G. Mass in trap (FxDxEx10^{-3}): _<0.0250_ mg
  H. Total in atmosphere (C+G): _0.743_ mg

I-296 (to be filled in for each memo)

Battelle
Columbus Laboratories

Date March 27, 1984

To   H Benecke/D Hopper

From   AJ Killmeyer/BC Garrett

Subject   Analytical Results of Subtask 5

● **EXPERIMENTAL CONDITIONS**

Date of trial _3/19/84_

Explosive: ( )2,4 DNT ( )2,6 DNT (X)TNT ( )Tetryl ( )RDX ( )HMX
Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN ( )Chemical 2
                                     ( )Chemical 3

LABORATORY RECORD BOOK 3F959
NUMBER/PAGES: _84_

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _3_ coupons): _<0.00500_
2. Explosive in rinse solution (mg): _<0.0125_
3. Explosive in atmosphere in chamber (mg): _5.94_

I. Calculation of Residual Explosive on Coupons

   A. Surface area per coupon: 81 cm^2 ($8.1 \times 10^{-3} \text{ m}^2$)
   B. Contamination density: _4.32_ mg/cm^2
   C. Extraction volume per coupon: _100_ mL
   D. Recovery efficiency: _100%_
   E. Extraction concentration factor: _—_
   F. Analysis results for extracts:

   324-50-3 1. Coupon 1   _<0.0500_ µg/mL
   324-50-4 2. Coupon 2   _<0.0500_ µg/mL
   324-50-5 3. Coupon 3   _<0.0500_ µg/mL

   G. Residual agent per coupon [$(F \times E \times C \times 10^{-3})/D$]:

      1. Coupon 1   _<0.00500_ mg
      2. Coupon 2   _<0.00500_ mg
      3. Coupon 3   _<0.00500_ mg

II. Calculation of Explosive in Rinse Solution

   A. Volume rinse solution: _250_ mL
   B. Extraction concentration factor: _—_
   C. Analytical results: _<0.0500_ µg/mL
   D. Mass in rinse solution ($A \times B \times C \times 10^{-3}$): _<0.0125_ mg

III. Calculation of Explosive in Atmosphere in Chamber

   A. Impinger volume (total of four): _250_ mL
   B. Analysis results: _23.2_ µg/mL
   C. Mass in impingers ($A \times B \times 10^{-3}$): _5.50_ mg
   D. Trap volume: _250_ mL
   E. Extraction concentration factor: _—_
   F. Analysis result: _0.547_ µg/mL
   G. Mass in trap ($F \times D \times E \times 10^{-3}$): _0.137_ mg
   H. Total in atmosphere (C+G): _5.94_ mg

Battelle
Columbus Laboratories

Internal Distribution

Date _March 28, 1984_

To   H Benecke/D Hopper

From   AJ Killmeyer/BC Garrett

Subject   Analytical Results of Subtask 5

(Run 17)

AJ Killmeyer
BC Garrett
G7920 Files

Mezley

- **EXPERIMENTAL CONDITIONS**

  Date of trial _3/20/84_
  Explosive: ( )2,4 DNT (X)2,6 DNT ( )TNT ( )Tetryl ( )RDX ( )HMX
  Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                        ( )Chemical 3

  LABORATORY RECORD BOOK 38959
  NUMBER/PAGES: _p. 55_

- **SUMMARY OF RESULTS**

  1. Residual explosive per coupon (mg)(average of _3_ coupons): _0.0123_
  2. Explosive in rinse solution (mg): _0.0628_
  3. Explosive in atmosphere in chamber (mg): _33.0_

  I. Calculation of Residual Explosive on Coupons

       A. Surface area per coupon: 81 cm^2 ($8.1 \times 10^{-3} \text{ m}^2$)
       B. Contamination density: _____ mg/cm^2
       C. Extraction volume per coupon: _100_ mL
       D. Recovery efficiency: _100%_
       E. Extraction concentration factor: _—_
       F. Analysis results for extracts:
           1. Coupon 1 _0.121_ µg/mL
           2. Coupon 2 _0.127_ µg/mL
           3. Coupon 3 _0.120_ µg/mL
       G. Residual agent per coupon [$(F \times E \times C \times 10^{-3})/D$]:
           1. Coupon 1 _0.0121_ mg
           2. Coupon 2 _0.0127_ mg
           3. Coupon 3 _0.0120_ mg

  II. Calculation of Explosive in Rinse Solution

       A. Volume rinse solution: _250_ mL
       B. Extraction concentration factor: _—_
       C. Analytical results: _0.251_ µg/mL
       D. Mass in rinse solution ($A \times B \times C \times 10^{-3}$): _0.0628_ mg

  III. Calculation of Explosive in Atmosphere in Chamber

       A. Impinger volume (total of four): _500_ mL
       B. Analysis results: _65.8_ µg/mL
       C. Mass in impingers ($A \times B \times 10^{-3}$): _32.9_ mg
       D. Trap volume: _250_ mL
       E. Extraction concentration factor: _—_
       F. Analysis result: _0.275_ µg/mL
       G. Mass in trap ($F \times D \times E \times 10^{-3}$): _0.0688_ mg
       H. Total in atmosphere (C+G): _33.0_ mg $\times 9.1 = 20$,

Run 1

Project Number G7920-0430

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Columbus Laboratories

Internal Distribution

AJ Killmeyer
BC Garrett
G7920 Files

Date March 28, 1984

To H Benecke/D Hopper

From AJ Killmeyer/BC Garrett

Subject Analytical Results of Subtask 5

● **EXPERIMENTAL CONDITIONS**

LABORATORY RECORD BOOK 3895-9
NUMBER/PAGES: p. 89

Date of trial 3/21/84
Explosive: ( )2,4 DNT ( )2,6 DNT ( )TNT ( )Tetryl (X)RDX ( )HMX
Decon Technique: (X)Hot Gases   ( )Chemical 1
                  ( )Freon/RADKLEEN   ( )Chemical 2
                                          ( )Chemical 3

● **SUMMARY OF RESULTS**

1. Residual explosive per coupon (mg)(average of _3_ coupons): _<0.0100_
2. Explosive in rinse solution (mg): _0.173_
3. Explosive in atmosphere in chamber (mg): _0.147_

I. Calculation of Residual Explosive on Coupons

A. Surface area per coupon: $81 \ cm^2 \ (8.1 \times 10^{-3} \ m^2)$
B. Contamination density: _5_ mg/cm^2
C. Extraction volume per coupon: _100_ mL
D. Recovery efficiency: _100_
E. Extraction concentration factor: _—_
F. Analysis results for extracts:

324- 55- 41. Coupon 1   _<0.100_ ug/mL
   -15 2. Coupon 2   _<0.100_ ug/mL
   -16 3. Coupon 3   _<0.100_ ug/mL

G. Residual agent per coupon [(FxExCx10^{-3})/D]:

1. Coupon 1   _<0.0100_ mg
2. Coupon 2   _<0.0100_ mg
3. Coupon 3   _<0.0100_ mg

II. Calculation of Explosive in Rinse Solution

A. Volume rinse solution: _250_ mL
B. Extraction concentration factor: _—_
C. Analytical results: _0.690_ ug/mL
D. Mass in rinse solution (AxBxCx10^{-3}): _0.173_ mg

III. Calculation of Explosive in Atmosphere in Chamber

A. Impinger volume (total of four): _300_ mL
B. Analysis results: _0.490_ ug/mL
C. Mass in impingers (AxBx10^{-3}): _0.147_ mg
D. Trap volume: _250_ mL
E. Extraction concentration factor: _—_
F. Analysis result: _<0.100_ ug/mL
G. Mass in trap (FxJxEx10^{-3}): _<0.0250_ mg
H. Total in atmosphere (C+G): _0.147_ mg

GC/MS DATA

The following table is a summary of the interpretation of the GC/MS analysis of three samples (coupon, trap, and impinger) from tests conducted with each explosive. The actual GC/MS data is presently stored in the BCL project file.

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GC/MS DATA SUMMARY

Run #	Explosive Type	Sample	Product Peak	Approximate Mass, mg
3	2,4 DNT	Impinger	3-Methylene-2-pentanone	Trace
			Unidentified	Trace
			2,4-Dimethylheptane	Trace
			2-Methyl Octane	Trace
			Acetic Acid	Trace
			Unidentified	Trace
		Trap	2-Ethenyl-2-Butenal	2
			Unidentified	Trace
			1,1,2,3 Tetramethyl-cyclopropane	3
			Unidentified	Trace
8	HMX	Coupon Extract	Phthalates[a]	Trace
		Impinger	Unidentified	Trace
		Trap	Unidentified	Trace
14	Tetryl	Coupon Extract	Phthalates[a]	Trace
		Impinger	Unidentified	Trace
		Trap	No peaks above background	--
15	TNT	Coupon Extract	Phthalates[a]	Trace
		Impinger	Unidentified	Trace
		Trap	Phthalates[a]	Trace
17	2,6 DNT	Coupon Extract	2,4,6-TNT	0.5
			Unidentified	Trace
			4-(methylamino)-7-nitrobenzofuran	Trace
		Impinger	Unidentified	Trace
		Trap	1,1-Diphenylhydrazine	Trace
			Unidentified	Trace
			Phthalates[a]	Trace
18	RDX	Coupon Extract	Phthalates[a]	Trace
		Impinger	Unidentified	Trace
		Trap	Unidentified	Trace

(a) Plasticizer contaminant

APPENDIX III-4

SUBTASK 6 – EVALUATION OF CANDIDATE DECONTAMINATION
CONCEPTS ON BUILDING MATERIALS –
TEST REPORT

TEST REPORT

FOR

TASK 4 SUBTASK 6

EVALUATION OF
CANDIDATE DECONTAMINATION CONCEPTS ON
BUILDING MATERIALS

to

UNITED STATES ARMY
TOXIC AND HAZARDOUS MATERIALS AGENCY

October 20, 1984

by

David R. Hopper
Scott Lugibihl

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

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TEST REPORT

FOR

TASK 4 SUBTASK 6

EVALUATION OF
CANDIDATE DECONTAMINATION CONCEPTS ON
BUILDING MATERIALS

Contract No. DAAK11-81-C-0101

to

UNITED STATES ARMY

TOXIC AND HAZARDOUS MATERIALS AGENCY

October 20, 1984

1.0 INTRODUCTION

In order to restore for alternate use or safely excess facilities that have been previously used for the manufacture or loading of explosoves, these facilities must first be decontaminated or inerted. Studies to develop novel concepts to effect decontamination of such facilities are being conducted by Battelle Columbus Laboratories (BCL) for the United States Army Toxic and Hazardous Materials Agency (USATHAMA) under Contract No. DAAK11-81-C-0101. In Task 2 of this contract,[1] ideas were systematically developed into concepts that could be used for the decontamination of buildings. These concepts were also evaluated and ranked with respect to selected technical and economic factors. Five concepts were then selected for further evaluation and knowledge gaps pertaining to the implementation of these five concepts were identified. Task 4 was then established to permit laboratory resolution of these knowledge gaps.[2] Subtask 6 is the fourth of four experimental subtasks that were established in Task 4. The test plan for this subtask describing the experimental effort that was planned is included with this report as Appendix A.

2.0 PURPOSE AND OBJECTIVES

The purpose of this subtask was to address aspects of both surface and subsurface decontamination of concrete as well as the surface contamination of stainless and mild steels. The influence of paint on the decontamination process was also addressed on all three of these surfaces. Based on the previous experimental subtasks, the following concepts were selected for further evaluation in Subtask 6:

- Hot Gases
- Solvent Extraction
- Chemical Decontamination
- Combined Chemical/Thermal Decontamination

The objective of this task was to assess the viability of each concept, determine the adequacy of the selected operating parameters, and provide data required for the engineering and economic analysis that is scheduled in the subsequent Subtask 7. The objectives and procedures used to test each concept are discussed further in the experimental procedure section of this report.

3.0 FACILITIES, MATERIALS, AND EQUIPMENT

The procedures for handling the explosives and most of the test equipment that was designed and fabricated to provide test conditions closely resembling field conditions have been described in detail in previous Task 4 reports.[3,4] Only new hardware and modifications of existing hardware that are unique to this subtask are described in detail in this study.

3.1 Explosives

The same six conventional high explosives, 2,4 DNT, 2,6 DNT, TNT, Tetryl, RDX, and HMX described in the Subtask 5[4] report were used here.

3

3.2 Test Chamber

All decontamination tests were conducted in the test chamber described previously.[3,4] The hot gas configuration is described in the Subtask 5 Report.[4] The configuration used to evaluate solvent and reagent application is described below.

3.3 Spray Delivery System

Previous experimental studies [5] indicated that the most effective way to apply liquid solvents/reagents to concrete and achieve penetration is by means of a finely divided, evenly distributed spray applied for short durations in a periodic manner. To perform this function experimentally, the system shown schematically in Figure 1 was assembled and attached to the test chamber. As described in detail below, this system consisted of a constant volume pump and associated plumbing, three spray nozzles plumbed in parallel and mounted in the chamber, and a system to control the spraying intervals.

3.3.1 Pump and Plumbing

The purpose of this hardware was to withdraw solvent/reagent from a supply reservoir and deliver it to the spray nozzles at a constant and controlled pressure. To assure the integrity of the solutions, the entire system was constructed of brass, stainless steel, or Teflon®. The pump selected for this purpose was a Teel Model 1PM gear pump with carbon bearings. The graphite packing that this pump was initially equipped with was replaced with Gortex® rope. A small reservoir immediately downstream of the pump served as a surge volume to dampen pressure fluctuations. Since a constant volume pump was in use, the magnitude of the liquid pressure on the downstream side of the pump was controlled by adjusting the pump running speed. Precise control of this pressure was achieved by regulating the quantity of solution returned to the supply reservoir by the bypass line. This regulation was determined by the position of Needle Valve A

on the bypass line. A second line (return line) was also installed to return liquid to the supply reservoir when the pump was running and the spray lines were not activated. This function was controlled by the position of the normally open selenoid valve (Valve B) contained in the line. To assure that the backpressure produced by this line was identical to that produced by the spray lines, the return line terminated at the reservoir with a spray nozzle identical to those on the spray line.

The spray line connected the pump system to the nozzle system. A normally closed selenoid valve (Valve C) on this line controlled the spray application. Valve D was a manual shutoff valve.

3.3.2 Spray Nozzles and Manifold

As shown in Figure 1, a rigid stainless steel manifold was constructed that aligned with the three nozzle access ports on the front of the chamber. The three nozzles were attached to the end of 1/4" stainless steel tubing that penetrated the chamber via Swagelock® fittings attached to the entry ports. These fittings permitted easy and uniform adjustment of the distance each nozzle was held from the test coupons. During these tests, overspray was minimized by adjusting this distance so the spray pattern just covered the exposed portion of the test coupons.

The nozzles used were solid-cone type nozzles commonly used in fuel oil burners. This type nozzle is designed to produce a conical and uniform spray pattern at operating pressures between 30 and 100 psig. At these pressures each nozzle delivers between 30 and 50 ml/min of water. Due to viscosity and density differences in the aqueous solutions used in these tests, the amount of reagent delivered during tests would vary slightly from these rate. These differences were taken into consideration by calibrating the flow through the nozzles prior to the test and selecting the operating pressure that was so determined to deliver the desired rate of the reagent in use.
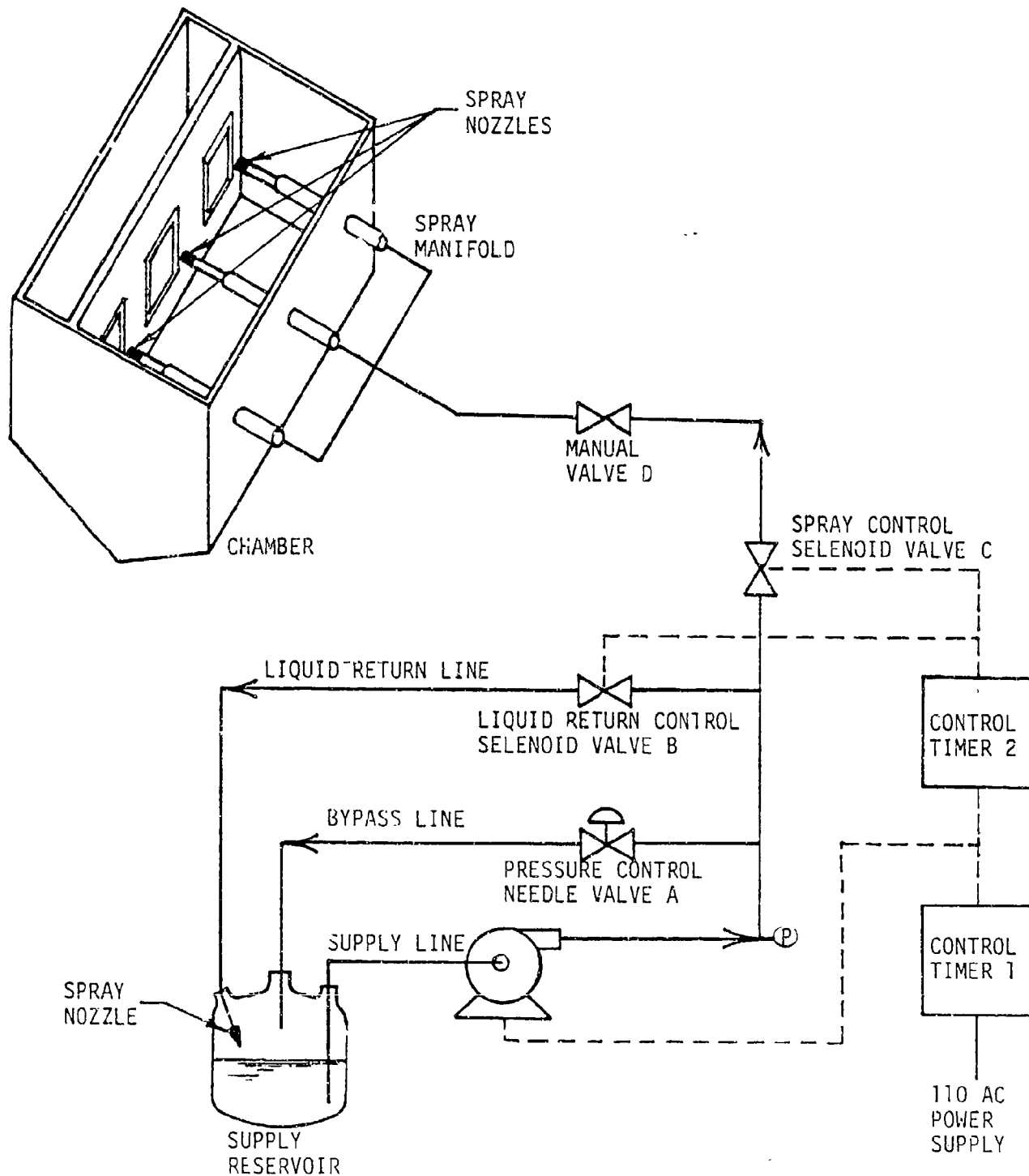
FIGURE 1. SPRAY DELIVERY SYSTEM

3.3.3 Control System

The spraying technique which was determined in previous studies to be the most effective,[5] required sequential application of short spray intervals over a long period of time. Consequently, while a test might last 8 hours, the actual total time during which spray would be applied was estimated to be only 8 minutes. To meet this criteria and minimize wear on the pump, the control system was designed to turn the pump on, allow the desired liquid pressure to be achieved, switch the valving so liquid went to the spray nozzles instead of the return line, and, after the appropriate spray interval was achieved, switch the valving back to the normal positions and turn off the pump. The control system was designed to reproduce this sequence on a periodic time schedule. This was achieved through the use of two timers. The first timer would supply power to both the pump and a second timer for a short duration time interval (30 seconds) on the desired periodic time schedule (once every 5 minutes). Thus started, the second double interval timer would first allow the pump to run with solution flowing through the return line until sufficient time elapsed for the liquid pressure produced by the pump to equilibrate (~ 20 seconds). This timer would then energize the two selenoids on the pumping system, thereby diverting the solution to the spray nozzles for the desired spray interval (5 seconds). After this spray interval elapsed, this timer would switch the valving back to the normal positions. Before this timer could initiate another spray interval (~ 20 seconds) the 30 second interval of the first timer would elapse and this first timer would turn off the power to the entire system. This system was thus capable of operating unattended, freeing the operator to make observations of the coupons.

3.4 Effluent Sampling System

During testing of both the Hot Gas and the combined chemical/Hot Gas concepts, the chamber effluent was sampled with the hardware and procedures previously used in subtask 5[4].

3.5 Reagents

As in Subtask 5, acetonitrile was used as the extraction solvent in the analysis of the coupons. During the analytical recovery studies, methanol was also used. The solvent extraction and chemical decontamination studies required the use of dimethyl sulfoxide (DMSO). In all of the above cases, UV grade distilled in glass solvents purchased from Burdick and Jackson Laboratories Incorporated were used. These solvents are commonly used in trace analysis by HPLC and gas chromatography.

In addition to the above solvents, low carbonate reagent grade sodium hydroxide purchased from J. F. Baker Chemical Company was used. Aqueous solutions of this reagent as well as the DMSO were made using dionized/distilled water purchased locally. The hydrochloric acid used was also reagent grade acid purchased from J. F. Baker Company.

3.6 Analytical Hardware

The same analytical hardware described in Subtask 5 was used in these studies.

3.7 Test Coupon

The 5 inch square coupons of 304 stainless steel as well as mild steel that were used in these tests were sheared from 18 gauge sheet stock. The 5-inch square, 1/4"-inch thick concrete coupons were the same as the "low porosity" coupons developed for the Task 3, Subtask 4 Diffusion Studies. These coupons are described in detail in that Subtask Report.[5]

Painted stainless steel coupons were prepared using the following procedure:

(1) Coupons were cleaned in hexane.

(2) Stainless steel coupons' surfaces were roughened with fine sandpaper in order to aid adherence of paint.

(3) One coat of Krylon green primer No. 1346 (conforms to MIL-P-8585) was applied and allowed to dry for one to three hours.

(4) Two coats of forest green alkyd paint (conforms to MIL-E-52798A NSN-8010-00-111-7937) were applied allowing each coat to dry for 24 hours.

(5) Painted coupons were allowed to cure under ambient conditions for at least two weeks prior to testing.

Painted concrete coupons were prepared by an identical procedure except that roughening with sandpaper was not required and that step was therefore omitted.

4.0 EXPERIMENTAL PROCEDURES

The concepts being tested and the rationale behind the test schedule are detailed in the Subtask 6 Test Plan (Appendix A). Many of the actual test procedures used are identical to those used in Subtask 5 and described in that report.[4] Consequently, in the discussion of procedures detailed below, whenever possible reference is made to these descriptions. Only procedures specific to Subtask 6 are described in detail.

4.1 Analytical Recoveries

In Task 2 of this contract[1] certification tests were conducted for the standard method that was specified for the analytical determination of the amount of explosive on a solid surface. The results of these tests, which are summarized in Table 1, indicate that, while reproducible, the recoveries from concrete are lower than desired. In addition, the spiking levels used during these tests were near the detection limit and were achieved through a precise but time consuming spiking procedure. Before begining decontamination tests with concrete, it was necessary to conduct method confirmation tests to determine if the spiking level, the distribution of the explosives on the coupon surface (spiking procedure), the use of more dense concerete, the presence of paint or heat treatment of the coupon would have a deleterious effect on the analytical recoveries.

TABLE 1. METHOD CERTIFICATION RESULTS

Detection Limit and Recovery from Coupon Types

Explosive	Unpainted Stainless Steel D.L.(a)	Unpainted Stainless Steel Recovery, percent	Painted Stainless Steel D.L.(a)	Painted Stainless Steel Recovery, percent	Unpainted Mild Steel D.L.(a)	Unpainted Mild Steel Recovery, percent	Painted Mild Steel D.L.(a)	Painted Mild Steel Recovery, percent	Unpainted Concrete D.L.(a)	Unpainted Concrete Recovery, percent	Painted Concrete D.L.(a)	Painted Concrete Recovery, percent
2.4 DNT	1.5	80	1.1	99	1.1	75	0.88	98	2.7	36	2.8	75
2,6 DNT	1.4	76	0.94	99	1.4	74	1.0	100	2.5	40	2.8	74
TNT	0.81	91	1.1	101	1.1	79	1.4	98	3.4	29	3.3	67
Tetryl	1.9	90	2.1	94	1.5	75	1.5	97	2.6	37	4.0	60
RDX	0.54	99	(b)	(b)	0.41	99	(b)	(b)	1.6	29	(b)	(b)
HMX	1.2	106	3.0	106	1.1	108	7.0	97	3.7	30	5.9	74

(a) Detection limit in milligrams/square meter of coupon surface.

(b) Not determined due to coelution with paint component.

For these confirmation tests, coupons were spiked with a quantity of a solution containing one or all of the six explosives. After permitting the solvent to evaporate, these spiked coupons were analyzed by either the standard or a slightly modified method. To determine the effect of the concrete density, two different concretes were used; the concrete used in the certification tests[1] and that used in the diffusion studies[5] and planned for use in this subtask.

The effect of concentration was evaluated by spiking coupons at several concentrations between the detection limit ($88 \ \mu g/m^2$) and the decontamination test spiking level ($50 \ g/m^2$). In addition the explosive was spiked on the coupon in both a well distributed manner and in a single spot.

Modifications of the standard method which appeared to hold promise of improving the recovery of explosives from concrete were also tested. Three different extractive solvents (acetonitrile, acetone, and methanol) were tested. In addition, the concrete coupons were broken up prior to extraction. They were typically broken into pieces smaller than 1/2 inch before extraction. In order to expose more surface area, coupons were also hand pulverized to a sand-like consistency in a mortar and pestal and then subjected to the extraction procedure.

4.2 Coupon Spiking

The coupon spiking procedures used to place the explosives on the coupons for the decontamination tests conducted in this task were identical to those used previously in the Subtask 5 Stainless Steel Surface Decontamination Studies.

4.3 Hot Gas Experiments

The results of the hot gas experiments in Subtask 5 indicated that all six explosives could be successfully removed from stainless steel by heating the coupon to 500 F and maintaining this temperature for one

hour. The purpose of these tests was to determine whether such a treatment will successfully remove the explosives from the other building material matrices. The first series of tests were conducted on mild steel using the procedures described in detail in the Subtask 5 test report. The second series of tests were conducted on painted stainless steel, painted concrete, and unpainted concrete using the modified procedure described below.

To minimize the number of tests necessary to produce duplicate results of each combination of the six explosives and three building material matrices, one coupon of each matrix was spiked with the explosive being tested and loaded in the test chamber as follows: the unpainted concrete nearest the gas inlet, the painted concrete in the center, and the painted stainless steel nearest the gas exit. In this way the interactions of emissions from the paint with the unpainted concrete as well as the contact of volatilized explosives with the pourous matrices were minimized. To evaluate the migration of the explosives through the concrete during processing, the rear chamber was purged with 10 scfh of heated nitrogen. The explosives in this stream were collected by passing the entire rear chamber effluent through an impinger trap system similar to that used to sample the front chamber effluent. In addition, after the test the rear chamber was washed down with acetonitrile and the wash solution collected. This solution along with the front chamber wash down, both impinger trains, and all three coupons were then analyzed for explosives as individual samples.

4.4 Solvent Extraction

The results of the Subtask 3 Enhanced Aqueous Solubilization Studies[6] indicated that a 60/40 mixture of DMSO in water is an adequate solvent for all six explosives. Further, the available literature data for two of the explosives (RDX and TNT) indicated that their solubility is markedly higher in neat DMSO. This series of tests was designed to determine not only the viability of the solvent extraction concept but also the quantity of solvent that must be applied, the required contact

time and the practical range of DMS concentrations. As described in the test plan (Appendix A) this was done by first conducting a series of screening test on stainless steel to determine conditions that produced sufficient removal, and then conducting a series of duplicate tests to confirm that similar results could be achieved on concrete and painted surfaces.

This testing was done using the test chamber and spray delivery system described previously. The spiked coupons were mounted in the chamber with the compartment dividers in place. The reagent selected for evaluation was placed in the receiver and the spray delivery system turned on and adjusted to apply a spray of 50 ml/min for 5 seconds on 5-minute intervals. These conditions were selected from the Task 3, Subtask 4 Diffusion Studies[5] as sufficient to produce rapid solution penetration of the concrete. The process control variable (contact time) then became the total time (hours) for which this treatment was applied.

At the end of the desired contact time, the treated coupons were removed from the chamber and submitted for analysis of explosive by the certified method. The solution in each of the three sumps was collected, its volume recorded, and an aliquot submitted for analysis. Each of the three sumps were then washed with measured volumes (~ 500 ml) of acetonitrile to remove any residual explosives. These wash solutions were collected and a sample of each was sent for analysis. The chamber was then dried and prepared for the next test.

4.5 Chemical Decontamination

It was previously demonstrated in Subtask 4[7] that sodium hydroxide will destroy these explosives in an aqueous DMSO solution. The objective of this test series was to determine if such destruction could also be accomplished on a surface or, more importantly, within a pourous matrix such as concrete.

The procedures used in these tests were essentially the same as the solvent extraction with the following exception. In all tests, the base concentration of DMSO in the aqueous solution was 30 percent.

Enough sodium hydroxide was then dissolved in this solution to produce either 0.1 or 0.01 normality.

The other procedural difference was the treatment of the solution collected in the sump. At the end of the test, enough concentrated HCl was added to the collected solution to lower the pH as indicated by pH paper to between 4 and 6. This pH change effectively quenched the reaction of sodium hydroxide with the explosives and thereby eliminated the time elapsed between the test and the performance of the chemical analysis as an experimental variable of importance.

4.6 Combined Chemical/Thermal Decontamination

With both the chemical and hot gas concepts, it was observed that some of the explosives could be destroyed at relatively mild conditions. The operating conditions selected for these concepts were usually determined by the one or two explosives that proved to be the most difficult to remove. However, the particular explosives that determined these more severe conditions differed between the two concepts, leading to speculation that a combination of the two concepts might effectively destroy all six c the explosives at more minimal conditions.

To evaluate this potential, one painted and two unpainted concrete coupons were spiked with explosives and loaded in the chamber. These coupons were first sprayed with 0.1 N sodium hydroxide/DMSO/water reagent for a 1-hour sequence and then immediately subjected to a hot gas treatment consisting of 1 hour at 300 F. The chemical treatment procedure was the same as discussed in the previous section except that the sump dividers were removed and the reagent was left in the sump. Besides the reduction in temperature and time of exposure, the hot gas treatment was further modified by substituting heated air for the simulated flue gas.

At the end of this combined treatment, the three coupons were removed from the chamber and submitted for analysis. The entire chamber interior was then washed, first with a measured amount of acetonitrile

and then with a volume of water. The water rinse was necessary to dissolve and remove a sodium hydroxide film from the chamber surfaces. Samples of both wash solutions were submitted for analysis.

4.7 Calculations

The calculations performed were similar to those used in Subtask 5 and consisted mainly of multiplying the concentration of the explosives in the liquid samples by the volume of the solution sampled to obtain the mass of explosives in the total solution.

5.0 DISCUSSION OF RESULTS

As stated previously, the major objective of this subtask was to assess the viability of the various concepts and demonstrate the adequacy of the operating conditions selected during previous subtasks. Due to scheduling conflicts, the four concepts evaluated in this task were not all in the same stage of development. Consequently the degree of testing varied from simple confirmation of stainless steel results on concrete (hot gas) to an almost complete parametric screening sequence (chemical decontamination). The results of these tests are discussed in detail below. Sample data can be found in Appendix C. The actual data can be found in BCL Record Books Number 39344 and 39748.

5.1 Analytical Recoveries

In the first tests of this series the effects of the spiking technique, the extraction solvent, and the physical state of the concrete coupons during extraction were examined. These tests were conducted on the same type of concrete coupons that were used in the method certification tests.[1] The spiking level was 50 g/m^2. The results of these tests indicated that the effects of the spiking technique and extraction solvents on recoveries was negligible. As can be seen in Table 2, both concentrated and distributed spikes of explosive produced the same recoveries. Table 3 shows that even though the solubility of explosives was greater in acetone

TABLE 2.   SENSITIVITY ANALYSIS RESULTS FOR COUPON SPIKING TECHNIQUES

Explosive	Dosing Techniques	Acetonitrile(b) I	B	Acetone(b) I	B	Comments
2,4-DNT	Single Spot	54.5	83.7	43.7	75.6	
	Multiple Spots	47.8	93.9	47.8	78.2	No demonstrated effect
	Variation(c)	+6.7	-0.2	-4.1	-2.6	
Tetryl	Single Spot	46.5	98.4	63.9	90.0	No demonstrated effect,
	Multiple Spots	48.7	97.8	44.9	92.2	assuming variation of
	Variation(c)	-2.2	+0.6	+19.0	-2.2	+19.0 to be an outlier

% Recovery for Parameters as Specified(a)

(a) I = Intact concrete

   B = Broken concrete.

(b) Extraction solvent used.

(c) Obtained by subtracting "multiple spots" value from "single spot" value.

than acetonitrile, acetonitrile produced a marginally higher recovery. As a result of these tests, it was determined that use of a time consuming spiking procedure designed to assure uniform distribution was not necessary. Continued use of acetonitrile, the preferred solvent from the standpoint of interferences with the analysis, was also recommended.

During these tests, the physical state of the concrete during the extraction was shown to be an important parameter. As shown in Table 4, breaking the coupon into pieces smaller than 1/2 inch prior to the extraction process improved the recovery at these high spiking levels was markedly improved. It was therefore decided to incorporate the step of breaking the concrete coupons into small pieces prior to the extraction process into the standard method.

When conducting an additional series of confirmation tests it was observed that the recoveries from concrete coupons, both painted and unpainted were erratic. Consequently this test series was expanded with greater attention being applied to details. As shown in Table 5, the recovery obtained from the painted stainless steel matrices both before and after heat treatment are comparable to those obtained in the past series. However, low recoveries were obtained from the concrete matrices, particularly with tetryl which yielded recoveries as low as 4 percent. Consideration was therefore given to what mechanisms could be the cause of these low recoveries and what modification of the analytical procedures might improve the recoveries. If the recovery is limited by mass transfer of the solvent within the concrete, then the percent recovery obtained should be independent of the spiking level and could be improved by increasing the surface area exposed to the solvent. On the other hand, if the recovery is limited by physical bonding to or by chemical reaction with the concrete, then the mass of concrete would be expected to determine the quantity of explosives held, not the quantity spiked. To explore these mechanisms further, additional extraction tests were conducted. First, all six explosives were spiked on a concrete coupon that was then broken into pieces smaller then 1/8 inch and extracted. This experiment was conducted in duplicate and compared with results obtained from a similarly spiked coupon that was similarly broken and then carefully crushed in a mortar and pestal to the consistency of coarse sand. These tests, shown

TABLE 3. SENSITIVITY ANALYSIS RESULTS FOR EXTRACTION SOLVENT

| Explosive | Extraction Solvent | % Recovery for Parameters as Specified[a] | | | | Comments |
| | | Intact | | Broken[b] | | |
		S	M	S	M	
2,4-DNT	Acetonitrile	54.5	47.8	83.7	83.9	Solvent has an effect.
	Acetone	43.7	47.8	75.6	78.2	Acetonitrile appears more effective than acetone at
	Variation[c]	+10.8	0	+8.1	+5.7	recovering explosive.
Tetryl	Acetonitrile	46.5	48.7	98.4	97.8	Same situation as for
	Acetone	63.9	44.9	90.0	92.2	2,4-DNT, assuming variation of -17.4 to be an
	Variation[c]	-17.4	+3.9	+8.4	+5.6	outlier

(a) S = "Single spot" or concentrated dosing technique

  M = "Multiple spots" or distributed dosing technique

(b) Pieces smaller then 1/2 inch

(c) Obtained by subtracting "Acetone" value from "Acetonitrile" value.

TABLE 4. SENSITIVITY ANALYSIS RESULTS FOR PHYSICAL STATE OF CONCRETE

Explosive	Physical State of Concrete	% Recovery for Parameters as Specified[a]				Comments
		Acetonitrile		Acetone		
		S	M	S	M	
2,4-DNT	Intact	54.5	47.8	43.7	47.8	Significant effect has been demonstrated. Crushed concrete consistently yields 50 to 75% greater recovery than does intact concrete
	Broken[b]	83.7	83.9	75.6	78.2	
	Variation[c]	-29.2	-36.1	-31.9	-30.4	
Tetryl	Intact [b]	46.5	48.7	63.9	44.9	Same situation was observed as that for 2,4-DNT
	Broken	98.4	97.8	90.0	92.2	
	Variation[c]	-51.9	-49.1	-26.1	-47.3	

(a) S = "Single spot" or concentration dosing technique
    M = "Multiple spots" or distributed dosing technique.

(b) Pieces smaller than 1/2 inch.

(c) Obtained by subtracting "crushed" value from intact value.

TABLE 5. EXPLOSIVES RECOVERY CONFIRMATION TESTS
FROM FIVE SUBSTRATES

Explosive	Spiking Level mg	Painted Stainless Steel	Thermally Processed [a][b] Painted Stainless Steel	Concrete	Painted Concrete	Thermally Processed [a][b] Painted Concrete
			Recoveries, Percent			
RDX	0.430	83	89	36	44	40
Tetryl	0.351	119	105	18	22	4
TNT	0.450	100	93	23	45	21
2,6 DNT	0.450	90	79	28	52	63
2,4 DNT	0.450	94	88	45	73	65

(a) 500 F for 1 hour
(b) Alkyd forrest green

TABLE 6. RECOVERIES OF SIX EXPLOSIVES SPIKED
ON CONCRETE COUPONS

Spiking Level (mg)	0.500	0.500	0.500
Pre extraction Treatment	Broken to < 1/8" pieces	Broken to < 1/8" pieces	Crushed to Coarse Sand
Explosive Recovery (%)			
HMX	47	41	64
RDX	38	33	53
Tetryl	< 5 (a)	< 5 (a)	4
TNT	18	15	27
2,6 DNT	47	38	63
2,4 DNT	42	36	60

(a) No peak visible.

in Table 6, indicate that the recoveries so obtained are low but reproducibile. The results also indicate that the time-consuming (>1 hour) grinding operation only marginally improves the recovery. This was further demonstrated by spiking another coupon with 0.356 mg of tetryl and analyzing the 1/8 inch and smaller pieces. These pieces were then crushed in a mortar and pestal and reanalyzed. While the first analysis only yielded 13 percent recovery, the second analysis performed with a larger surface area exposed yielded no additional recovery.

The results of the final series of recovery tests are shown in Table 7. In these duplicate tests, recoveries of the two explosives tetryl and TNT, previously shown to be the most difficult to recover, as well as 2,4 DNT, the most easily recovered explosive, were determined and compared at three different spiking levels. These analyses, which were performed after breaking the coupons to <1/8-inch pieces, indicate that the percent recovery, and hence the amount remaining in the concrete is dependent upon the spiking level.

In an attempt to further resolve the analyatical questions concerning the recovery of explosives from concrete, two coupons spiked with 4 and 40 mg of tetryl respectively were submitted for analysis by the standard method. However, methanol was used as the solvent in the extraction process instead of acetonitrile. It was anticipated that if physical bonding was limiting the recovery, the more polar methanol solvent would improve tne recovery. However, no significant improvement in recovery was observed.

The results from the analytical recovery tests taken as a whole, are confusing. It is apparent that the mechanisms that interfere with the analytical recovery of explosives from concrete are complex and probably involve a combination of mass transfer, physical bonding, and chemical reactions. The ramifications of each mechanism on the interpretation of the analytical results from decontamination tests are markedly different. This precludes correcting the analysis for recovery efficiency based on the present data base. However, a more detailed study of the fate of explosives in concrete necessary to permit such correction was beyond the scope of the program. Consequently the following procedures were selected for use in these tests. When concrete coupons were used in a decontamination

TABLE 7. RECOVERY OF EXPLOSIVES FROM CONCRETE COUPONS
VERSUS EXPLOSIVE SPIKING LEVEL

Spiking Level (mg)	Recoveries, Percent		
	Tetryl	TNT	2,4 DNT
0.4	10	32	34
	25	36	38
4.0	51	17	40
	48	37	46
400.0	73	63	63
	79	67	67

test, they were broken into smaller than 1/2-inch pieces prior to extraction with acetonitrile. The remainder of all analyses were performed with the certified method. The analytical result obtained by this procedure was reported as if 100 percent recovery of the explosive on the coupon was obtained.

5.2 Hot Gas Experiments

These tests, conducted to confirm that the conditions that effected satisfactory removal from stainless steel would also suffice for the other building material matrices, were divided into two parts. The interpretation of these test results, within the analytical limitations discussed previously, is presented below. The sample data, including time versus temperature profiles can be found in Appendix B.

5.2.1 Mild Steel

Since volatilization appeared to be an important mechanism in the removal of explosives from stainless steel and since both stainless and mild steel are relatively impervious to penetration by liquids and vapors, it logically follows that both surfaces would be adequately decontaminated by identical thermal conditions. To support this interpretation two tests were conducted on the baseline explosives (2,4-DNT and HMX). The results of these tests are presented in Table 8. As expected, no significant differences were observed between stainless steel and mild steel. based on this observation and the opinion that a like similarity would be observed between painted stainless steel and painted mild steel, no additional testing was conducted with mild steel coupons.

5.2.2 Concrete, Painted Concrete, and
Painted Stainless Steel

A total of twelve confirmation tests were conducted on these matrices. In eleven of these tests, the conditions selected in Subtask

TABLE 8. COMPARISON OF MILD AND STAINLESS STEELS

Test No.	Coupon Material	Explosive	% Recoveries				
			Coupon Position in Chamber			Chamber Wash Down %	Flue Gas %
			Exit %	Middle %	Entrance %		
4	Stainless Steel	2,4-DNT	<0.003	<0.003	<0.003	0.24	19.3
16	Mild Steel	2,N-DNT	0.014	0.014	0.014	<0.0013	17.6
8	Stainless Steel	HMX	<0.003	<0.003	<0.003	0.3	<0.06
19	Mild Steel	HMX	<0.0012	<0.0012	<0.0012	0.33	0.019

5 (500 F for 1 hour) were utilized. Due to a failure in the gas heating system, the twelfth test was conducted at 400 F. These tests are summarized in Table 9. It appears that the hot gas concept developed for stainless steel (500 F for 1 hour) will sufficiently decontaminate all six explosives from these building materials as well. However, the rear chamber analysis, along with the observation of crystals of explosives on the rear of the coupon of the treated coupons, indicated that migration of the explosives through the concrete occurs even with the less volatile explosives. This fact must be taken into consideration when designing decontamination systems for concrete walls as thick as one foot. A second observation made during analysis of the coupon that is of importance to the use of this concept was the brittleness of heat-treated coupons. After being subjected to the hot gas treatment, the coupon was much easier to break into pieces for extraction. Heat treatment also resulted in darkening and occasional blistering of the paint.

5.3 Solvent Extraction

Fourteen tests were conducted to evaluate the concept of solvent extraction of explosives from building materials. As can be seen in Table 10, the results obtained from stainless steel were inconsistant. For example:

- The residual explosive found on the three coupons frequently varied by an order of magnitude
- Two tests for the removal of 2,4-DNT at identical conditions yielded markedly differing results (98.2 and >99.99 percent removed).

However it was shown that spray applications of solvent is capable of removing a high percentage of explosive contamination. Further, it was shown that the removal of explosives by this process is a strong function of the DMSO concentration. This is more clearly demonstrated when the results of runs 1-4 are directly compared as in Table 11. In these runs, the quantity of solvent applied to each coupon was approximately double that required to solubilize the quantity of explosive spiked on the coupon.

TABLE 9. HOT GAS DECONTAMINATION TEST RESULTS USING SIX EXPLOSIVES

Run No.	Explosive testing	Spiked level mg/coupon	Coupons				Recovery		Chamber[a]			
			Painted Stainless		Painted Concrete		Concrete		Front		Rear	
			mg	percent	mg	percent	mg	percent	mg	percent	mg	percent
20	HMX	421	0.029	0.007	<0.013[b]	<0.003	<0.013[b]	<0.003	0.598	0.047	0.072	0.006
21	2,4 DNT	304	0.015	0.005	0.041	0.013	0.063	0.021	153	16.8	0.10	0.01
22	Tetryl	416	<0.01[b]	<0.002	<0.025[b]	<0.006	<0.025[b]	<0.006	0.613	0.049	<0.025[b]	<0.002
23	RDX	388	<0.01[b]	<0.003	<0.025[b]	<0.006	<0.025[b]	<0.006	6.13	0.53	<0.025[b]	<0.002
24	2,6 DNT	404	0.041	0.010	<0.025[b]	<0.006	0.015	0.004	155	12.8	0.122	0.010
25	TNT	352	<0.005[b]	<0.001	<0.005[b]	<0.001	<0.005[b]	<0.001	57.9	5.49	0.037	0.004
26	HMX	421	<0.005[b]	<0.001	0.022	0.005	<0.013[b]	<0.003	0.279	0.022	<0.025	<0.002
27	2,4 DNT	304	<0.013[b]	<0.004	<0.013[b]	<0.004	0.041	0.013	146.88	16.1	4.43	0.48
28	2,6 DNT	416	<0.01[b]	<0.002	<0.025[b]	<0.006	<0.025[b]	<0.006	182	15.0	1.62	0.13
29	RDX	388	0.028	0.007	<0.025[b]	<0.006	<0.025[b]	<0.006	2.37	0.20	<0.05[b]	<0.004
30	Tetryl	(~400)	0.13	0.033	<0.025[b]	<0.006	0.051	0.013	0.65	0.054	<0.05[b]	<0.004
31[c]	TNT	220	23	10.5	0.423	0.19	0.90	0.41	--	--	--	--

(a) Effluent and wash down
(b) Below detectable limit
(c) Coupons only achieved 400 F

TABLE 10. SOLVENT EXTRACTION PRELIMINARY STAINLESS STEEL SCREENING RESULTS

Run No.	Explosive	DMSO Application			Residual Explosive				Average Removal, percent
		DMSO, percent	Application period, hours	ml/coupon(a)	Coupon, percent	Sump, percent	Chamber Wash, percent	Mass Balance, percent	
1	HMX	60	8	377	12-46	20-24		66	72.0
2	HMX	100	1	105	0.03-0.3	62-77	2.6	72	99.9
3	2,4-DNT	100	1	70	1.1-5.7	58-72	6.3	74	97.0
4	2,4-DNT	60	8	551	14-17	64-78	1.7	91	81.1
5	2,4-DNT	75	8	563	0.9-2.8	88-127	-	>110	98.2
6	RDX	60	8	563	7.8-36.9	70-92	4.8	110	75.9
7	RDX	75	8	597	0.002-0.03	90-110	1.0	103	99.97
8	RDX	75	6	448	0.01-0.02	85-116	6.6	104	99.96
16	2,4-DNT	75	4	335	5.4-6.8	95-117	3.9	114	93.97
18	HMX	75	4	322	0.17-5.9	81-96	11.9	94	97.31
19	Tetryl	75	6	412	<0.0036	112-127	9.7	125	>99.997
20	TNT	75	6	482	0.7-2.6	57-95	2.4	85	98.54
26	TNT	75(b)	8	588	0.007-0.014	12.6-24.3	0.15	17.5	99.998
27	2,4-DNT	75	8	568	<0.002-0.011	95-112	1.6	106	>99.993

(a) Volume recovered in sump.
(b) Possibly contaminated with NaOH.

TABLE 11. THE EFFECT OF DMSO CONCENTRATION ON REMOVAL OF EXPLOSIVES

Run No.	Explosive	DMSO Concentration	Application Period, hours	Volume ml	Percent removal
1	HMX	60	8	377	72
2	HMX	100	1	105	99.9
3	2,4 DNT	100	1	70	97.0
4	2,4 DNT	60	8	551	81.1

Neat DMSO removed a markedly higher fraction of the explosive than did an aqueous DMSO solution.

Due to the limit of sodium hydroxide solubility in DMSO/water, a 75 percent DMSO solution is considered the upper limit for the chemical concept. To provide a direct comparison and guidance for testing the chemical concept, several solvent extraction tests were conducted applying this concentration of DMSO for different time periods. These results are summarized in Table 12. Of interest is the observation that increased application time produced no significant increase in the removal of RDX. However it appears that a 6-hour application is sufficient in all cases.

After the above observations were made, the two tests summarized in Table 13 were conducted to confirm similar extractive removal of explosives from the more pourous matrices. In the first test (Run 17), coupons of stainless steel, painted concrete, and unpainted concrete were spiked with RDX and subjected to 4 hours of spray application. Much higher removal of explosives was achieved on stainless steel (99.998) than on concrete either painted (98.9) or unpainted (99.16). In the second test (Run 21), TNT was spiked on three separate concrete coupons. In an attempt to evaluate the ability of this concept to achieve decontamination in depth, a "sandwich" coupon was mounted in the center section of the test chamber. This sandwich was made by spiking the 400 mg of TNT on a single 14-inch thick coupon and then placing identical but unspiked concrete coupons both in front and in back of it as shown in Figure 2. In order to give direct comparison to the stainless coupon results, a 6-hour application period was used. It is interesting to note that this comparison indicates that the removal from a single concrete coupon was higher than that achieved from stainless steel. This observation more likely reflects the lack of quantitative recovery of explosives from concrete discussed previously. The removal observed from the "sandwich" coupon has other significance. During the test it was observed that a significant quantity of solvent was dripping into the sump from the bottom edge of the sandwich coupon.

TABLE 12. SOLVENT EXTRACTION[a] FROM
STAINLESS STEEL VERSUS
APPLICATION TIME

Explosive	Application Time		
	8 hours	6 hours	4 hours
2,4-DNT	98.2->99.99	--	93.97
RDX	99.99	99.96	99.988
TNT	99.998	98.54	--
HMX	--	--	97.31
Tetryl	--	>99.997	--

(a) 75 percent DMSO/water solvent - 5 second spray
     for 5 minutes.

TABLE 13. SOLVENT[a] EXTRACTION OF EXPLOSIVES FROM BUILDING MATERIALS

Explosive	Solvent[b] Application Period, hours	% Removal			
		Stainless Steel	Painted Concrete	Unpainted Concrete	Concrete Sandwich
RDX	4	99.968	98.90	99.16	--
TNT	6	98.54	--	-99.73->99.999	87.0

(a) 75 percent aqueous solution of DMSO.

(b) Period spray applied for 5 second intervals every 5 minutes.

(c) Previous test result.
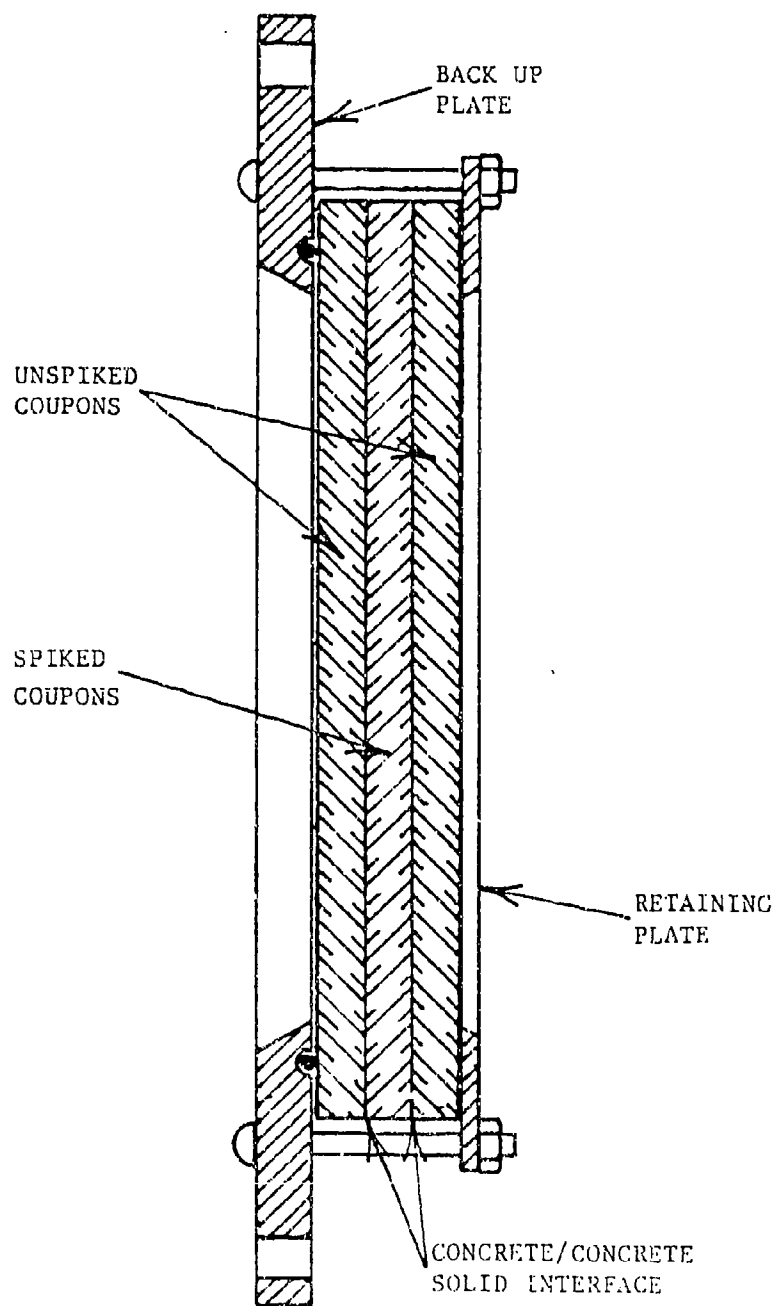
FIGURE 2. SANDWICH COUPON

This was probably due to solvent flowing down the interface between the front and middle coupons of the sandwich and therefore not penetrating the spiked middle coupon. The 87 percent removal that was observed was probably removed from the front surface of the spiked coupon. While this is relatively low when compared to the >99 percent removal obtained from a single coupon, it does indicate that a significant penetration and solubilization process was occuring at least to the depth of this interface.

To further evaluate the depth to which this solvent will penetrate concrete, and consequently the ability of solvent application to extract explosives, a blank test was conducted. In this blank test, an unspiked sandwich coupon, similar to that described in the previous paragraph was placed in one chamber section and a 2-inch thick single coupon was placed in another. Both were subjected to the spray application process. The coupons were periodically removed from the chamber and weighed to determine the amount of solvent contained in each coupon. As can be seen in Figure 3, both the front coupon of the sandwich as well as the 2-inch thick coupon gained weight rapidly and were still gaining weight after over 6 hours of spraying. However, the middle and back coupons of the sandwich showed only a small, immediate gain in weight (1.5 grams total), but after 8 hours had not gained further. This small and identical weight gain might be due to overspray penetrating the edge of the sandwich. Why identical results were obtained is not completely understood. The ramnification of this test result is that this type of sandwich coupon is not adequate to evaluate migration of solvent and/or explosives into and through the concrete. Since the time and financial resources available to this task were not adequate to effect design and construction of an alternative coupon, this investigation of possible explosive-solvent migration was discontinued.

5.4 Chemical Decontamination

The ten tests shown in Table 14 were conducted as the stainless steel prescreening of this concept originally scheduled in Subtask 5. In Subtask 3 the use of a NaOH/DMSO/water system to both extract and destroy
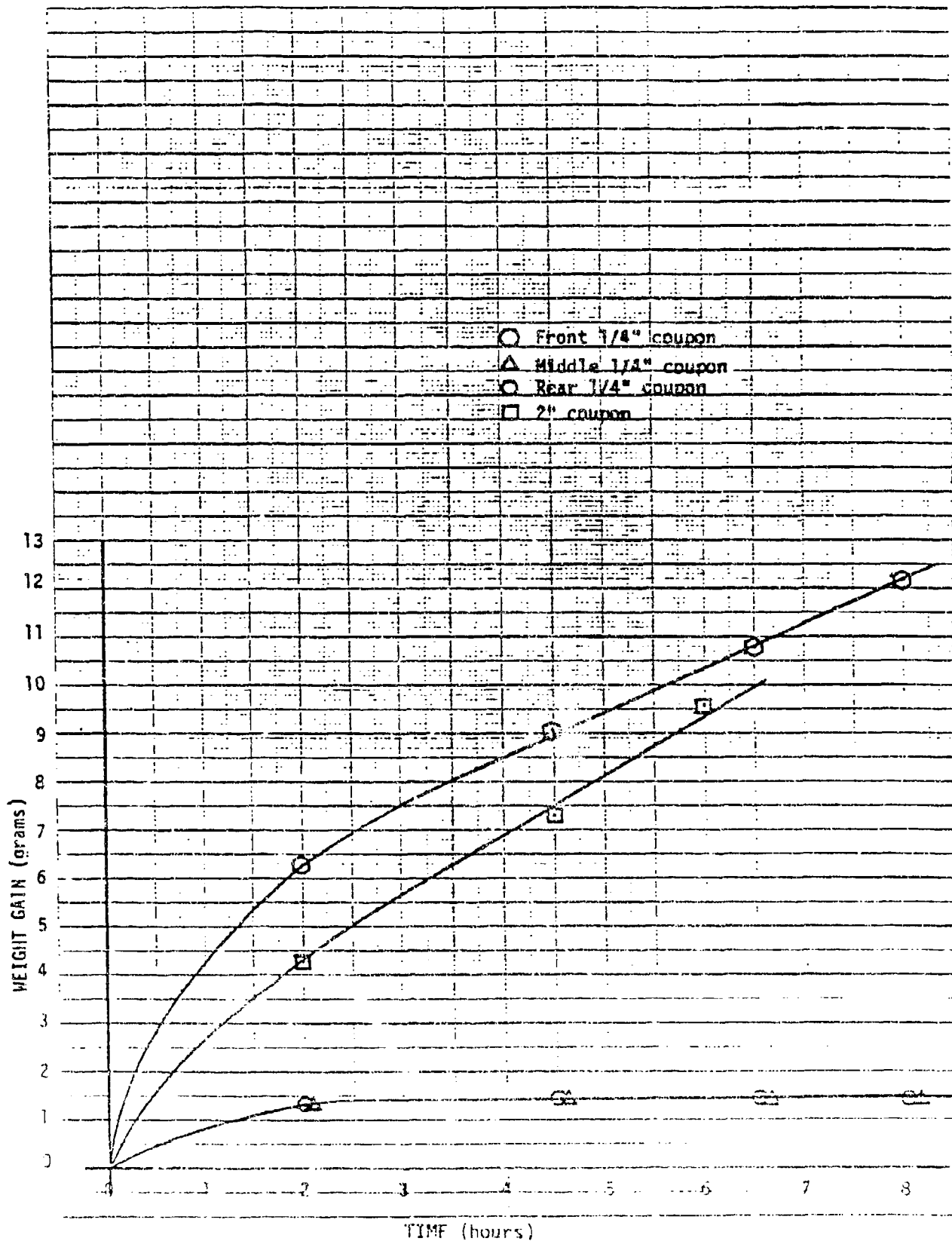
FIGURE 3. WEIGHT GAIN BY CONCRETE DURING SPRAYING

TABLE 14. CHEMICAL DECONTAMINATION PRELIMINARY STAINLESS STEEL SCREENING RESULTS

(seventy-five percent DMSO aqueous system)

Run No.	Explosive	Normality NaOH	Applied NaOH(a) Solution, ml/coupon	Residual Explosive			Mass Balance, percent	Average Removal, percent
				Coupon, percent	Sump, percent	Chamber Wash, percent		
9	RDX	0.1	275	0.1-1.1	0.04-3.9	0.15	1.8	99.44
10	2,4-DNT	0.1	123	0.6-1.8	0.05-0.08	0.08	1.5	98.62
11	2,4-DNT	0.1	283	0.007-3.6	0.04-0.07	0.004	1.4	98.7
12	RDX	0.1	151	0.004-6.3	0.004-0.28	0.06	3.6	96.6
14	2,4-DNT	0.01	158	0.06-5.5	5.4	0.28	3.2	97.3
15	RDX	0.01	288	<0.003-0.005	6.3-26.1	0.5	14.6	99.997
22	TNT	0.1	306	0.03-0.27	0.68-0.75	0.012	0.8	99.88
23	Tetryl	0.1	256	<0.003	<0.01-0.17	0.008	0.06	>99.997
24	2,6-DNT	0.1	298	0.01-0.35	30.5-40.1	0.04	25.3	99.88
25	HMX	0.1	232	<0.003	0.008-8.7	<0.014	0.003	>99.997

(a) Volume recovered in sump.

the explosives was identified. These stainless steel test results indicate that the inclusion of NaOH in the reagent appears to enhance the removal of explosives from stainless steel. Using an application period (4 hours) of half that used for DMSO/water alone (8 hours), >99.4 percent removal was achieved for five of the six explosives. The relatively low removal of 2,4-DNT that was achieved (98.73 percent) is unexplained. It is also not clear why appreciable quantities of 2,6-DNT, RDX, and 2,4-DNT were found in the undestroyed sumps.

Encouraged by the above results, additional tests were conducted using concrete coupons. In each of these tests, two unpainted concrete coupons and one painted concrete coupon were utilized. The results of these tests are summarized and compared with the stainless steel test results in Table 15. In all cases, the removal from concrete was not as high as that achieved from stainless steel. Particularly poor removal of 2,6-DNT and HMX were produced. This disappointing result is probably due to lack of penetration of the reagent into the concrete coupon as discussed in the previous section. This hypothesis is further supported by the higher removal achieved from painted concrete. During coupon spiking it was observed that the paint inhibits the penetration of the explosive into the concrete. This results in a surface deposit which would be more easily removed.

5.5 Combined Chemical/Thermal Decontamination

Four tests, which were conducted to evaluate this concept in duplicate with both RDX and 2,6-DNT. The results, summarized in Table 16, are encouraging. The benefits of this concept are even better demonstrated in Table 16. The removal of these two explosives was accomplished at relatively mild conditions and was significantly greater than that produced by either concept alone. While a residual sulphur-like odor as well as a sodium hydroxide film were left on the concrete coupon after treatment, a water rinse appeared to easily remove the film and

TABLE 15. SUMMARY OF CHEMICAL DECONTAMINATION(a)
RESULTS

	Average Removal, Percent		
Explosive	Stainless Steel	Concrete	Painted Concrete
2,4-DNT	98.7	99.47	99.911
2,6-DNT	99.88	51.05	95.41
TNT	99.88	99.988	99.992
Tetryl	>99.997	93.52	99.78
RDX	99.44	95.15	99.22
HMX	>99.997	73.05	98.62

(a) 0.1 N NaOH in 75 percent aqueous DMSO solution.

TABLE 16.   COMPARISON OF CHEMICAL/HOT GAS CONCEPT WITH
HOT GAS AND CHEMICAL DECONTAMINANTS

	Percent Removal		
Explosive[a]	Chemical/Hot Gas (4 hr spray/1 hr at 300 F) percent	Chemical (4 hr spray) percent	Hot Gas (1 hour at 300 F) percent
RDX	99.98	95.15	91.87
2,6 DNT	99.64	51.05	<50.0[b]

(a)   Concrete coupons spiked with -300 mg of explosive.
(b)   Anticipated result based on literature as well as hot gas results
with HMX during Subtask 5.

decrease the odor. Resources did not permit a more detailed study of this most promising concept.

6.0 CONCLUSIONS AND RECOMMENDATIONS

The results of the tests conducted in this subtask indicate that at least two concepts, Hot Gas and Chemical Decontamination, have applicability to the restoration of facilities previously used for the manufacture or loading of explosives. The third concept, solvent extraction, appears to have limited applicability due to the lack of penetration of concrete by the solvent. The most promising concept however, appears to be the combination of the chemical decontamination and hot gas concepts. Based on the minimal number of tests conducted, this combination not only produces effective decontamination at milder operating conditions, but also appears to provide solutions to other shortcomings of the two individual concepts. For example, the Hot Gas Concept by itself has the worrysome result of promoting migration of some explosives into and through concrete. In the combination, the sodium hydroxide tends to destroy these explosives on contact minimizing the concern for migration. Similarly the Chemical Decontamination Concept is limited by penetration of the concrete by the reagent and the difficulty in removing the DMSO from the concrete after the treatment. In the combined process, the heating cycle first promotes further penetration of concrete by the reagent, then increases both the solubility of the explosive in the reagent as well as the rate of reaction of the explosive with the sodium hydroxide, and finally at higher temperatures volatilizes the DMSO and many of the products produced by degradation of the DMSO and the explosives thereby driving them out of the concrete.

Due to the low and variable recoveries of explosives by the standard analytical method, certification of decontamination effectiveness remains a more difficult problem. While the assumption of 100 percent recovery discussed previously permitted satisfactory interpretation of

the experimental results, it would most likely not be an acceptable assumption when certifying 5X decontamination of a building. Such a certification would in all likelihood require additional studies to resolve or determine the cause of the inability to satisfactorily recover explosives.

Based on the above conclusions it is recommended that the engineering and economic analysis scheduled for the final subtask (Subtask 7) of this task be performed on the Hot Gas Concept, the Chemical Decontamination Concept, and the Combined Chemical/Hot Gas Concept. It is further recommended that consideration be given to additional studies aimed at resolving the fate of the explosives in a concrete matrix.

REFERENCES

(1) "Development of Novel Decontaminating and Inerting Techniques for Explosives Contaminated Facilities, Phase I Identification and Evaluation of Concepts", Contract DAAK-11-81-C-0101 to USATHAMA, July 1983, by H. P. Benecke, et al.

(2) "Design Plan for Task 4 - Development of Novel Decontaminating and Inerting Techniques for Explosives Contaminated Facilities", Contract DAAK-11-81-C-0101 to USATHAMA, August 29, 1983, by H. P. Benecke and D. R. Hopper.

(3) "Test Plan for Design and Construction of Agent/Explosives Dedcontamination Concepts Test Chamber", Contract DAAK-11-81-C-0101 to USATHAMA, September 12, 1983, by D. R. Hopper et al.

(3) "Test Plan for Design and Construction of Agent/Explosives Decontamination Concepts Test Chamber", Contract DAAK-11-81-C-0101 to USATHAMA, September 12, 1983, by D. R. Hopper et al.

(4) "Test Report for Task 4, Subtask 5 - Stainless Steel Surface Decontamination by Hot Gases", Contract DAAK11-81-C-0101, to USATHAMA, August 3, 1984, by D. R. Hopper et al.

(5) "Test Report for Task 3, Subtask 4 - Diffusion Studies", Contract DAAK11-81-C-0101 to USATHAMA, February 10, 1984, by E. R. Zamejc et al.

(6) "Task 4, Subtask 3 - Enhanced Aqueous Solubilization of Explosives", Copntract DAAK11-81-C-0101 to USATHAMA May 17, 1984, by H. P. Benecke et al.

(7) "Test Report for Task 4, Subtask 4 - Prescreening of Chemical decontamination Concepts", Contract DAAK11-81-C-0101 to USATHAMA, July 6, 1984, by H. P. Benecke et al.

APPENDIX A

TEST PLAN TASK 4 SUBTASK 6

TASK 4, SUBTASK 6
TEST PLAN
FOR
EVALUATION OF CANDIDATE DECONTAMINATION
CONCEPTS ON BUILDING MATERIALS
to
UNITED STATES ARMY
TOXIC AND HAZARDOUS MATERIALS AGENCY
May 8, 1984

1.0 INTRODUCTION

The development of novel concepts for the decontamination of
explosives contaminated buildings is being carried out by Battelle
Columbus Laboratories (BCL) for the United States Army Toxic and
Hazardous Materials Agency (USATHAMA) under Contract No. DAAK11-81-C-0101.
In a previous phase of this program, decontamination concepts were
systematically developed and five were recommended for further study.[1]
Previous experimental subtasks in the present phase (Task 4) [2] have
resolved some of the knowledge gaps concerning these concepts and
developed operating parameters for the most promising.[3,4,5] In this
subtask, the final experimental subtask in Task 4, selected decontamina-
tion processes will be applied to contaminated coupons made of the
common building materials (stainless steel, mild steel, and concrete)
in both painted and unpainted configurations.

2.0 PURPOSE AND OBJECTIVES

The purpose of this subtask is to address aspects of both
surface and subsurface decontamination of concrete as well as the
surface contamination of stainless and mild steels. The influence of
paint on the decontamination process will also be addressed for all

three surfaces. The concepts to be evaluated in this subtask include:

- Hot Gases
- Solvent Extraction
- Chemical Decontamination
- Combined Chemical/Thermal Decontamination

The objective of this task is to determine the viability of each concept and provide data for the engineering and economic analysis that is scheduled in the subsequent Subtask 7.

3.0 FACILITIES

The evaluations in this subtask will be carried out in the test chamber and laboratory facilities used previously in Subtask 5. [5]

4.0 EXPERIMENTAL PROCEDURES

The general testing procedures to be used are the same as those used previously in Subtask 5 with the following modification. Since porous coupons (concrete) will be used in some of the tests, effluents from the rear chamber will be collected and analyzed to determine if migration of the explosives through the coupons occurs. The rear chamber will also be washed down with acetonitrile after each test and the washdown solution similarly analyzed.

4.1 Analytical Recovery of Explosives

The Certification Studies on the analytical method that were conducted in the initial phase of this program[1] indicated that between 29 and 40% of the explosive spiked on 25 cm^2 coupons made of Sakrete$^®$ could be recovered. Additional tests conducted as part of the present task [6] have indicated that breaking the coupons into

pieces smaller than 1/8 inch would increase the recoveries to between
94 and 98 percent. However, the concrete used in these certification
studies was of different porosity and composition than that to be
used in this subtask. In addition, none of the coupons, painted or
unpainted, had been subjected to a thermal or chemical process that
might form new substances. The tests described below are designed
to determine whether similar recovery of explosives can be achieved
from these new matrices.

4.1.1 Test Description

To investigate the possibility of the formation of substances
that might interfere with the certified analytical method in use,
painted coupons of stainless steel and concrete will be subjected to
either the hot gas process (500 F for 1 hour) or one of the chemical
processes and then submitted as analytical blanks. In addition,
coupons of concrete, painted concrete, painted stainless steel,
thermally treated painted stainless steel, thermally treated painted
concrete, chemically treated painted concrete, and chemically treated
painted stainless steel will be spiked to levels approximately 15
times greater than the detection limit with a solution containing all
six explosives and then submitted for analysis. The purpose of these
tests is to verify that satisfactory recoveries can be achieved and
not to establish the analytical recoveries for data processing purposes.

4.1.2 Contingency Plans

As stated previously, such tests have the objective of
simply confirming that the recoveries previously reported can be
achieved with these test matrices. If insufficient or erratic
recoveries are obtained, consideration will be given to expanding

these tests to explain such behavior. However, time and resources do not permit conducting a matrix of tests to determine the fate of explosives in these materials.

4.2 Hot Gases

4.2.1 Test Description

The results of the hot gas experiments in Subtask 5 have indicated that all six explosives can be successfully removed from stainless steel by heating the coupon to 500 F and maintaining this temperature for one hour. To determine whether such a treatment will successfully remove the explosives from the other building material matrices, coupons of these matrices will be subjected to a similar test procedure. As in Subtask 5, decontamination effectiveness will be determined from the chemical analysis of the treated coupons as follows:

$$\text{Percent Decontamination Effectiveness} = \left(1 - \frac{\text{residual concentration}}{\text{spiked concentration}}\right) \times 100$$

4.2.1.1 Mild Steel. Since volatilization appears to be important in the removal of explosives from stainless steel and since both stainless steel and mild steel are relatively impervious to penetration by vapors, it logically follows that both surfaces would be adequately decontaminated by identical thermal conditions. To test this premise two tests will be conducted with one of the baseline explosives (2, 4 DNT and HMX) spiked on mild steel coupons. If no significant differences are observed between these tests and the Subtask 5 tests with stainless steel, no additional hot gas tests will be conducted on mild steel coupons.

4.2.1.2 Concrete, Painted Concrete, and Painted Stainless

Steel. The ability to remove each of the six explosives from these three matrices by the hot gas concept will be determined in duplicate as follows. One coupon of each matrix will be spiked with 400 mg of the explosive to be tested. The chamber will then be loaded as follows; the concrete coupon nearest the gas inlet, the painted concrete coupon in the middle, and the painted stainless steel coupon nearest the gas exit. This configuration should minimize both the interaction of the emissions from paint with the unpainted concrete and the contact of vaporized explosives with porous matrices. Simultaneous testing of all three matrices in this manner will permit completion of these tests in duplicate with a minimum number of runs (12).

The test procedure to be followed for each run is identical to that used in Subtask 5 with the following additions. To evaluate the migration of the explosives through the concrete during processing, the rear chamber will be purged with 10 scfh of heated nitrogen. The explosives in this stream will be collected by passing the entire rear chamber effluent through an impinger trap system similar to that used to sample the front chamber effluent. In addition, after the test the rear chamber will be washed down with acetonitrile and the wash solution collected. This solution along with the front chamber wash down, both impinger trains, and all three coupons will be analyzed as individual samples.

4.2.2 Performance Tests

While the chamber and its operating procedure are essentially unchanged from Subtask 5, coupons of concrete or coupons that have been painted have not been previously tested. To determine whether the use of such coupons interfere with the test procedures or the analytical methods, the chamber will be loaded with unspiked

coupon of either concrete, painted concrete, or painted stainless steel and thermally cycled to 500 F for 1 hour in combustion product gases. The coupons from these runs will be spiked with explosives and used for the analytical recovery tests discussed in a preceding section.

4.2.3 Contingency Plans

If any combination of explosive and building material is found to resist adequate decontamination by hot gases at 500 F for 1 hour, more severe conditions will be tested until satisfactory decontamination is achieved. Considerations of economics and integrity of the building indicates that exposure for longer times would be given preference over exposure to higher temperatures.

4.3 Solvent Extraction

The results of the Subtask 3 Enhanced Aqueous Solubilization Studies[3] have indicated that a 60/40 mixture of DMSO in water is an adequate solvent for all six explosives. Further, literature data for two of the explosives (RDX and TNT) indicates that their solubility in neat DMSO is markedly higher than in a 60/40 mixture with water. In addition, the relatively high flash point of DMSO makes it a very safe solvent for use in the extractive removal of explosives. For these reasons a DMSO/water solvent has been chosen for both the solvent for extractive removal of explosives from surfaces as well as the base solvent for chemical decontamination concepts.

4.3.1 Test Description

The process variables to be investigated in the screening tests for extractive removal are the quantity of solvent applied, the

total contact time, and the DMSO concentration in water. The
practical lower limit on the DMSO concentration appears to be 60%.
Therefore, solutions containing 60, 75, and 90% DMSO will be tested.
The quantity of solvent applied will be controlled by varying the
length and number of spray applications. The contact time will be
controlled by varying the intervals between sprayings.

The solvent extraction test will consist of two series.
The first series will be the stainless steel prescreening studies
that were deferred from Subtask 5. Following the rationale used in
Subtask 5, a matrix of process conditions will be tested with coupons
spiked with HMX or 2,4 DNT. The results of the Task 3 Subtask 4
Diffusion Studies [7] and the Task 4 Subtask 3 Enhanced Aqueous
Solubilities Studies [3] will be used to select the conditions to be
tested. Once conditions are found that adequately remove both of
these explosives from stainless steel, the ability of these conditions
to adequately remove the other four explosives from stainless steel
will also be determined in duplicate.

A second series of tests will also be conducted in duplicate.
These tests will evaluate the ability of the DMSO solvent system to
remove explosives from concrete and painted surfaces. Following the
logic developed for the selection of the procedure used in the hot
gas experiments, the chamber will be loaded with one concrete, one
painted concrete, and one painted stainless steel coupon spiked with
400 mg of explosive. The decontamination conditions developed in
the screening studies will then be applied to the coupon. As in
previous studies, evaluation for the effectiveness of the removal
of explosive will be made by submitting both the coupon and the
solvent for analysis. During this series of tests particular atten-
tion will be given to the possible migration of solvent and/or explo-
sives into and through the concrete coupons.

4.3.2 Performance Tests

Prior to conducting tests with explosives, performance tests will be made with unspiked coupons of stainless steel and concrete, both painted and unpainted. These tests will not only assure that the solvent delivery system, the only hardware not previously tested, is working properly, but will also produce sample blanks for an analytical baseline determination. Particular care will be given to observing possible phase separations in the chamber wash down solutions due to the presence of DMSO/water.

4.3.3 Contingency Plans

As in the previous tests, if a combination of explosive and and building material is found not to be adequately decontaminated by the conditions selected in the prescreening, additional tests will be conducted on that combination using more severe decontamination procedures (e.g. longer contact time). It will be assumed that the combinations of explosives and building materials that were already successfully decontaminated during the prescreening conditions would be adequately removed by these more severe conditions as well.

4.4 Chemical Decontamination

Successful destruction of the explosives by sodium hydroxide in aqueous DMSO was demonstrated in Subtask 4 [4]. If extractive removal of the explosives can be effected with a DMSO/water solution, then the Subtask 4 results have direct application for destruction of the solubilized explosives. If extractive removal from a porous medium like concrete cannot be accomplished, the sodium hydroxide must then

destroy the explosives within the concrete matrix. Investigation
of this aspect will be the major thrust of these experiments.

4.4.1 Test Description

Both painted and unpainted concrete coupons will be spiked
with explosive and then treated by spraying them with 0.1 N sodium
hydroxide in a 70/30 percent DMSO/water solution. The process
variables will be the quantity of solution applied and the contact
time. The total quantity of solution will be controlled by the length
and number of spray applications. The contact time will be controlled
by the time interval between spray applications.

A matrix of six screening tests to select sufficient opera-
ting conditions will be conducted with HMX and 2, 4 DNT. Again, the
results of of previous studies will be used to select the operating
conditions to be evaluated in this matrix of tests. The remaining
four explosives will be tested at the conditions selected during these
screening tests.

4.4.2 Contingency Plans

As was the case in other test series, if inadequate destruc-
tion of any single explosive is obtained using the processing conditions
selected in the screening studies, that explosive will be further tested
either at higher sodium hydroxide concentrations or higher DMSO content.
It will be assumed that these new conditions will also suffice for the
explosives that were adequately decontaminated at the milder conditions.

4.5 Combined Chemical/Thermal Decontamination

In Subtask 5 [5] it was observed th   the volatile explo-
sives such as 2, 4 DNT could be removed by relatively mild tempera-
tures (<400 F) but a higher temperature treatment (500 F for 1 hour)
was required to destroy the HMX in place.

In Subtask 4 [4] it was demonstrated that RDX and probably
HMX could be effectively destroyed at ambient temperatures by a
Sodium Hydroxide/DMSO/water system. While such a system was found to
be less effective with TNT and 2, 4 DNT, it was shown that higher
temperatures (65 C) would increase the decontamination effectiveness.

The above facts imply that a combined process of sodium
hydroxide application followed by a relatively low temperature hot
gas treatment might effectively combine the best aspects of both
concepts and permit processing all six explosives at conditions
markedly milder than those required if each concept was applied singly.

4.5.1 Test Description

The building material medium determined in the previous
tests to be the most difficult to decontaminate, will be used for
this test series. This medium appears most likely to be painted or
unpainted concrete. Similar to the other test series, screening
test will first be conducted on coupons individually spiked with
HMX, 2, 4 DNT or TNT. Destruction of the other three explosives will
then be demonstrated at the best conditions selected from these
screening studies. The test conditions to be evaluated will be
selected from the results of previous test series on the individual
concepts. For example, it is anticipated that the hot gas treatment
could be performed at conditions as low as 300 F.

4.5.2 Contingency Plans

The purpose of these tests is to determine if synergistic effects produced by combining the two processes will permit operating at significantly milder conditions. As such the emphasis will be in investigating the operating limits rather than, as in the previous tests, determining some conditions under which the process will operate successfully.

5.0 MEASUREMENTS

Since determination of decontamination effectiveness is the major objective of these tests, the critical measurements will again be the analysis of low concentrations of explosives on the test coupons. This determination will be made by the standard High Performance Liquid Chromatography (HPLC) used previously. [3,4,5] To assure that this critical measurement can be performed satisfactorily, verification tests (Section 4.1) have been scheduled.

6.0 COUPON SPIKING

Procedures for spiking the coupons were developed in Subtask 5 [5] and shown to be effective. The same procedures and spiking levels will be used for these tests.

7.0 SAFETY

To minimize the possibilities of an accident, all handling of explosives will be performed by experienced personnel using the procedures previously reviewed by the Army [8]. Handling of solvents

and sodium hydroxide will also be performed by experienced staff wearing the proper protective clothing (face shield, rubber gloves, and aprons). All other general test procedures are identical to those used safely in previous subtasks.

8.0   SCHEDULE

To supply an economy of efforts, the hot gas experiments have been continued as a logical extension of Subtask 5. In addition, the similarity of Tasks 3 and 4 has permitted solvent and chemical application hardware to be ordered in advance. Consequently, the tests outlined in this plan can be completed by early June, 1984.

REFERENCES

(1)   "Development of Novel Decontaminating and Inerting Techniques
      for Explosives Contaminated Facilities, Phase I Identification
      and Evaluation of Concepts", Contract DAAK11-81-C-0101 to
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(2)   "Design Plan for Task 4 - Development of Novel Decontaminating
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      H. P. Benecke and D. R. Hopper.

(3)   "Task 4, Subtask 3 - Test Plan for Enhanced Aqueous Solubiliza-
      tion", Contract DAAK11-81-C-0101 to USATHAMA, September 12, 1983,
      by H. P. Benecke. Test Report in preparation

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(6)   January "Monthly Cost and Performance Report - Novel Processing
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      1984.

(7)   "Test Report for Task 3, Subtask 4, Diffusion Studies", Contract
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      et al.

(8)   "Custodial Plan for Physical Security of Sensitive Explosives at
      Battelle Columbus Laboratories", Contract DAAK11-81-C-0101 to
      USATHAMA, September 22, 1983, by F. J. Mezey.

APPENDIX B

EXPERIMENTAL DATA

473

Run # 16 2,4 DNT Time—Temperature Profiles

MILD STEEL COUPON

Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmosphere
⊠ Entrance Coupon
⊠ Middle Coupon
✳ Exit Coupon
◈ Exit Gas
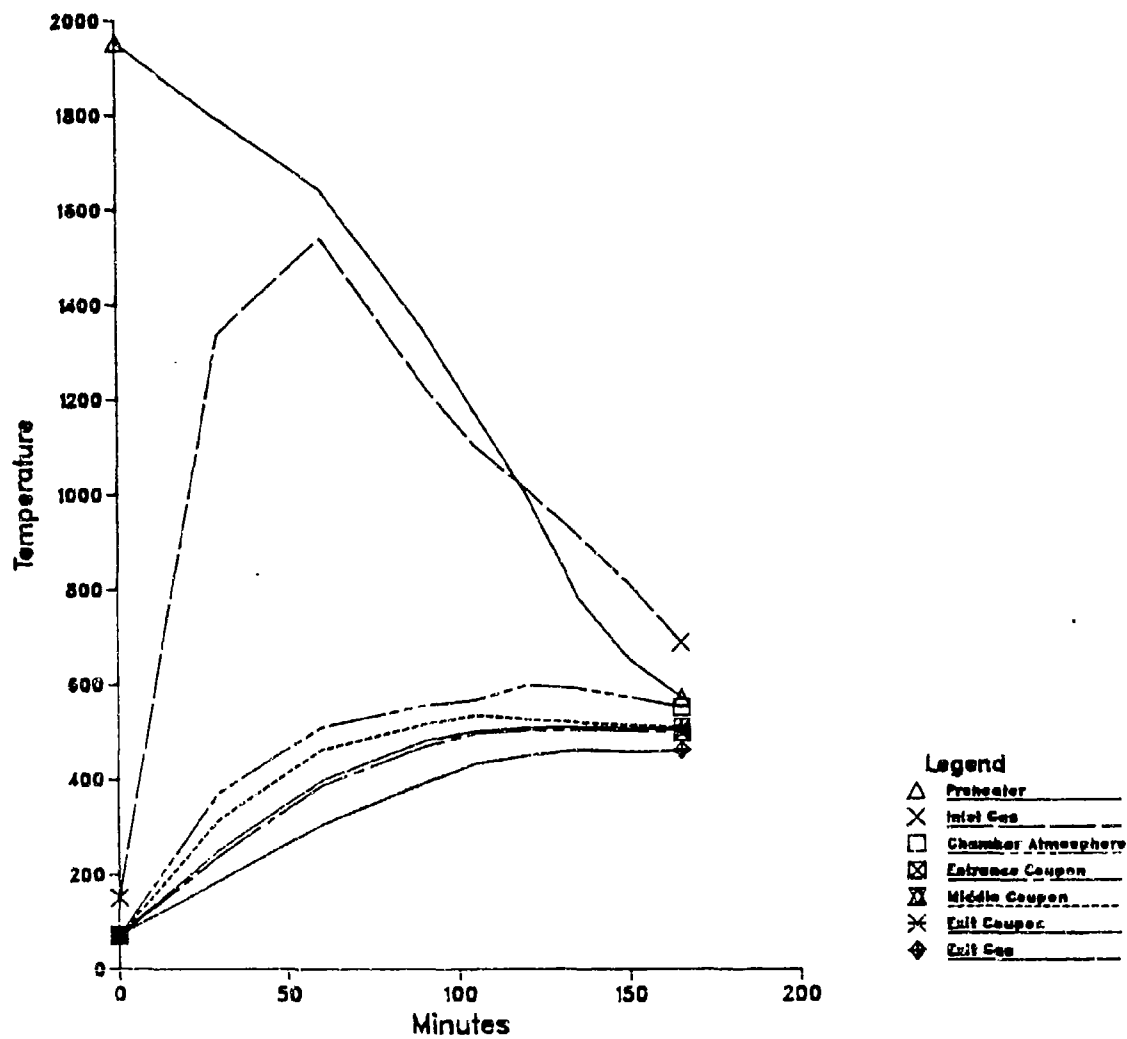
Run # 19 HMX Time-Temperature Profiles

MILD STEEL COUPONS
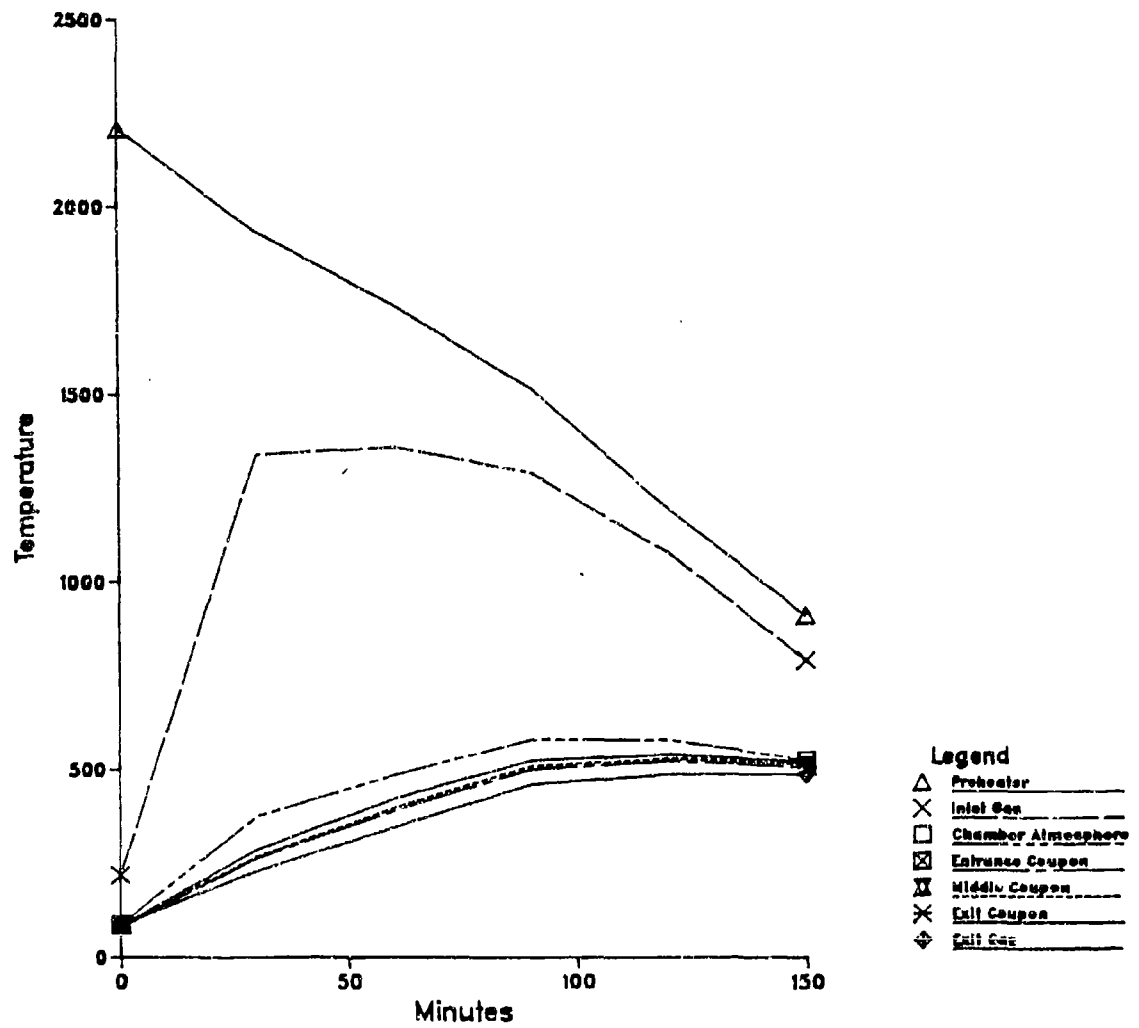
Run # 20 HMX Time−Temperature Profiles
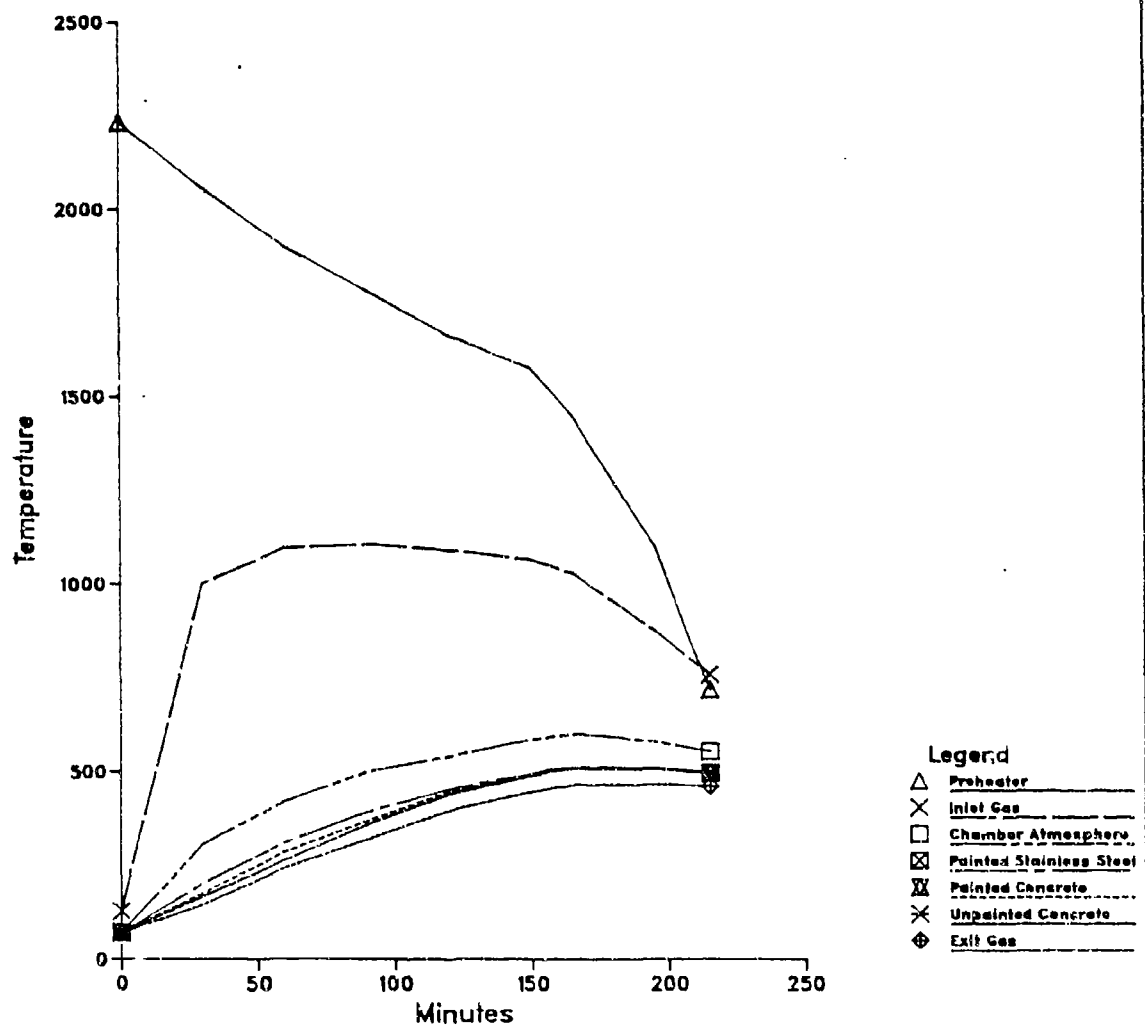
Run # 21 2,4 DNT Time–Temperature Profiles

Run # 22 Tetryl Time—Temperature Profiles
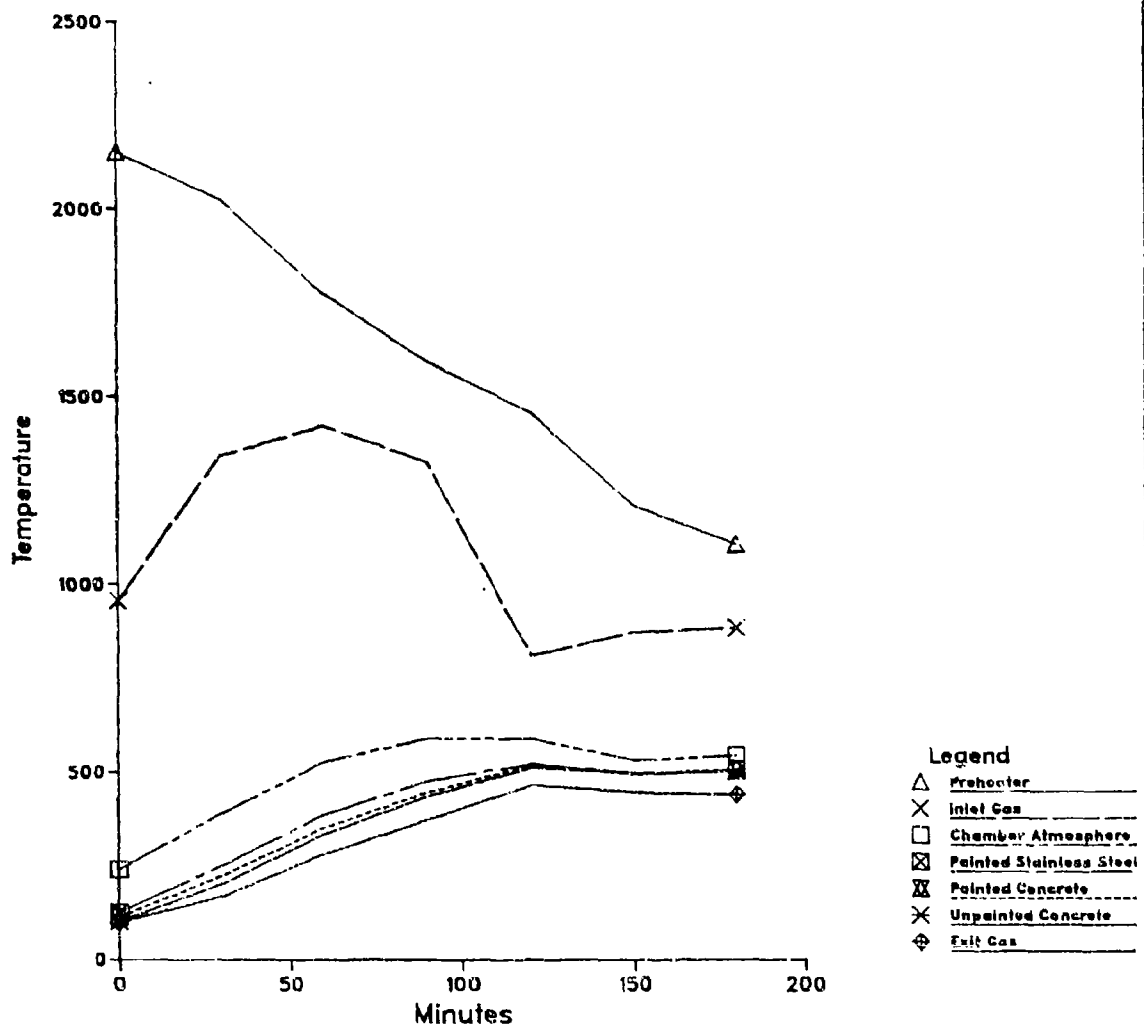
Legend
△ Preheater
✕ Inlet Gas
▢ Chamber Atmosphere
⊠ Painted Stainless Steel
⊠ Painted Concrete
✳ Unpainted Concrete
⊕ Exit Gas
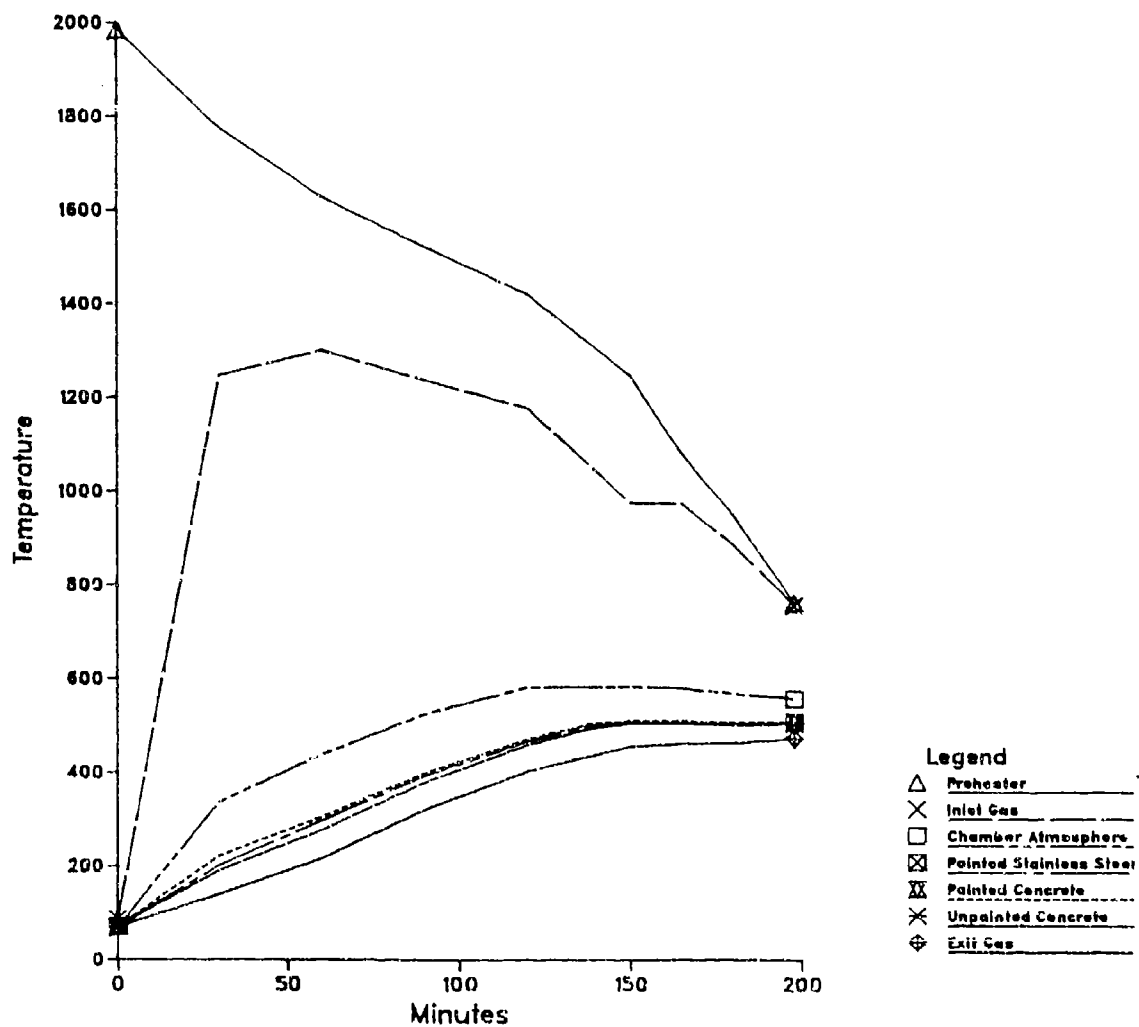
Run # 23 RDX Time-Temperature Profiles
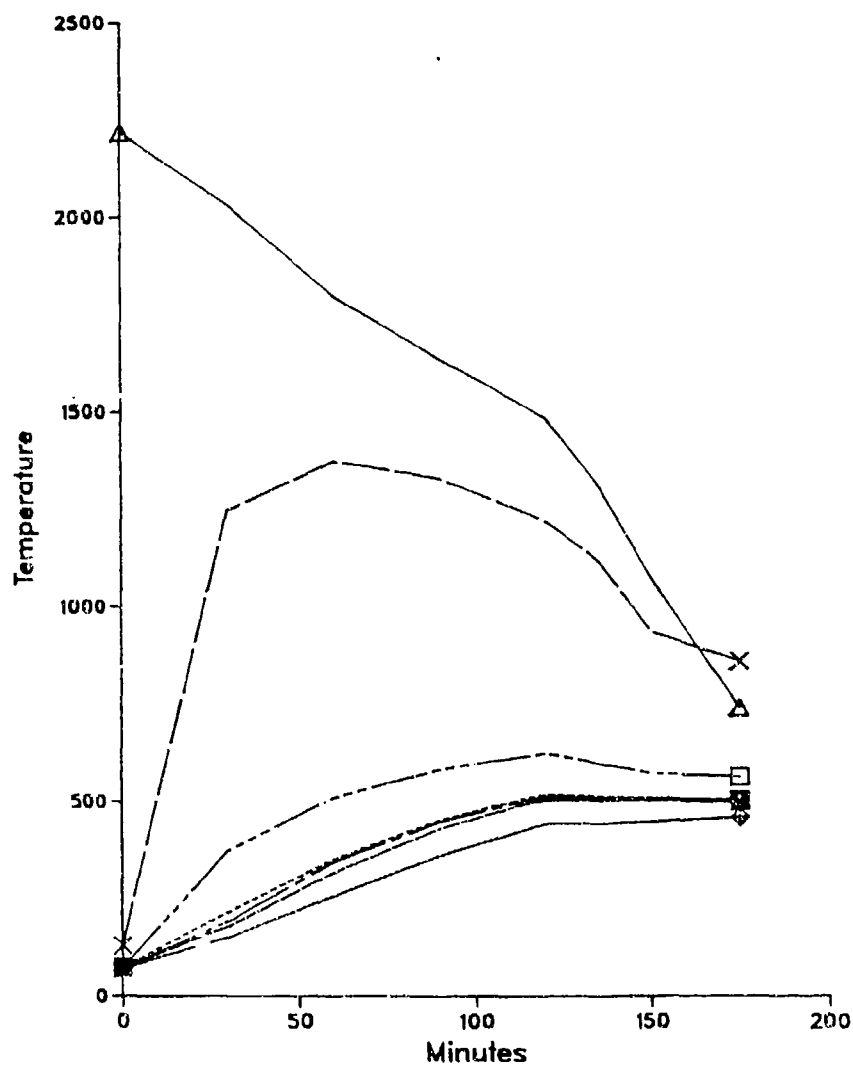
Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmosphere
⊠ Painted S
⊠ Painted Concrete
✕ Unpainted Concrete
⊕ Exit Gas

Run # 24 2,6 DNT Time—Temperature Profiles
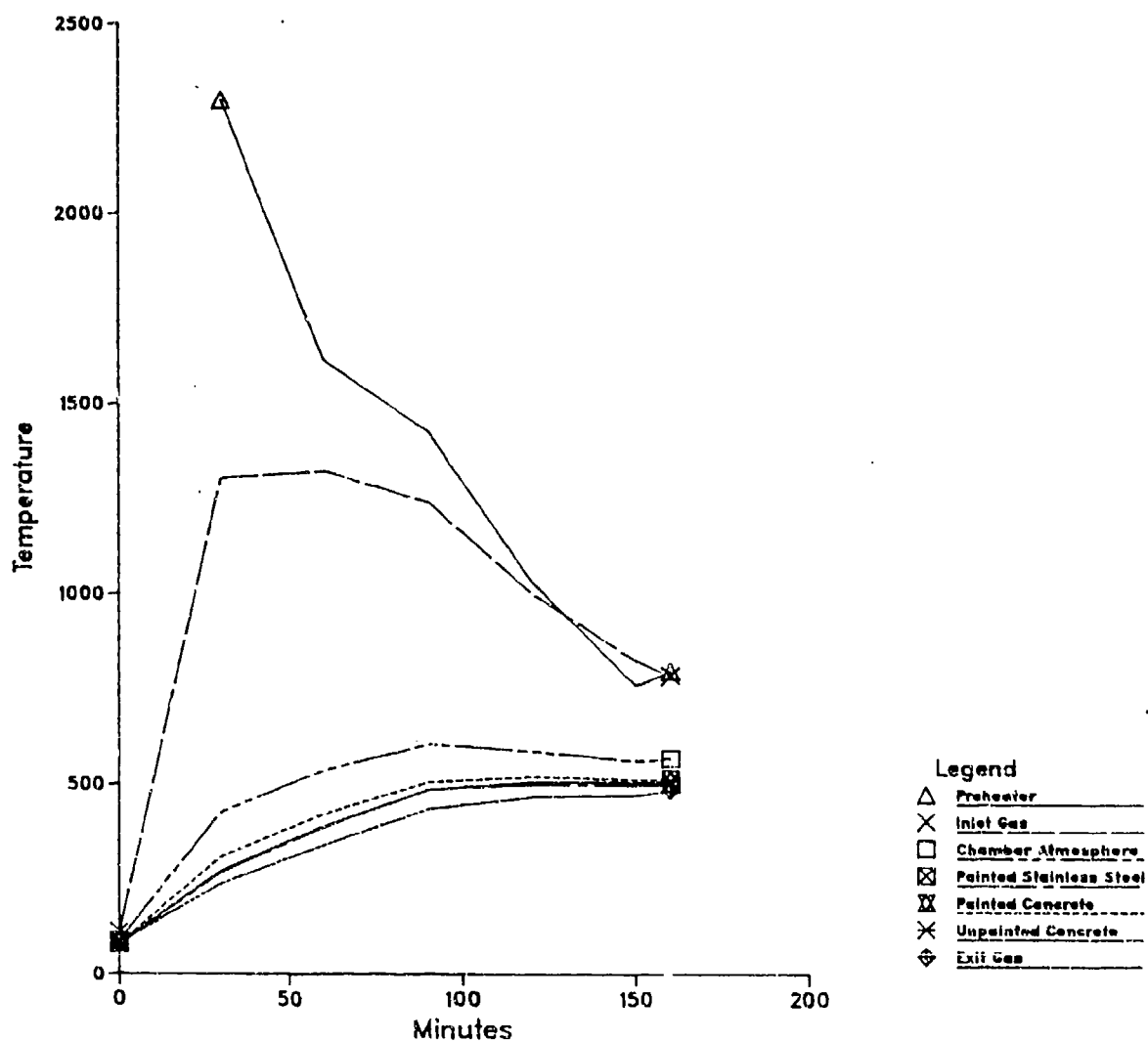
Run # 25 TNT Time–Temperature Profiles

Run # 26 HMX Time—Temperature Profiles
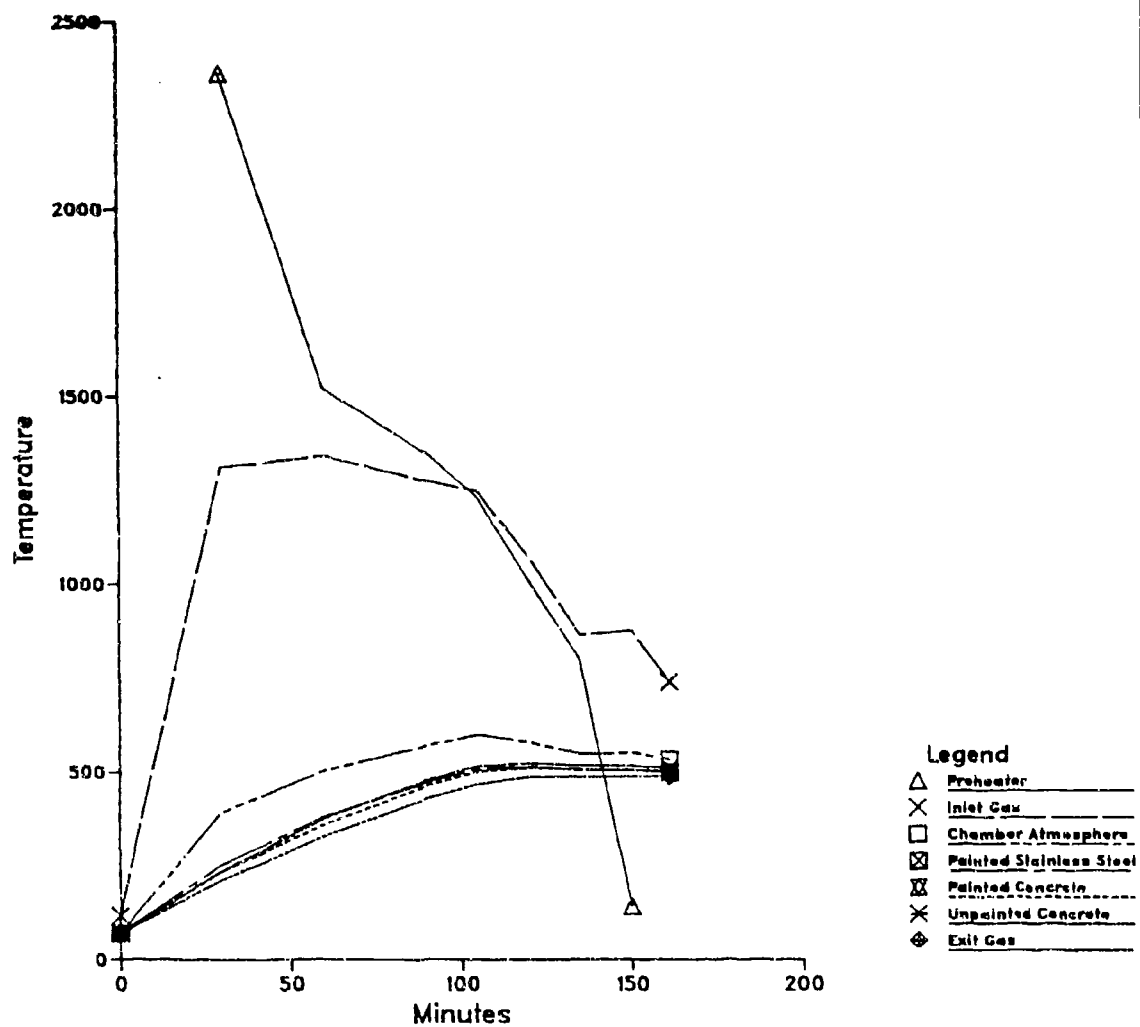
Legend
△ Preheater
✕ Inlet Gas
☐ Chamber Atmosphere
⊠ Painted Stainless Steel
⋈ Painted Concrete
✳ Unpainted Concrete
⊕ Exit Gas
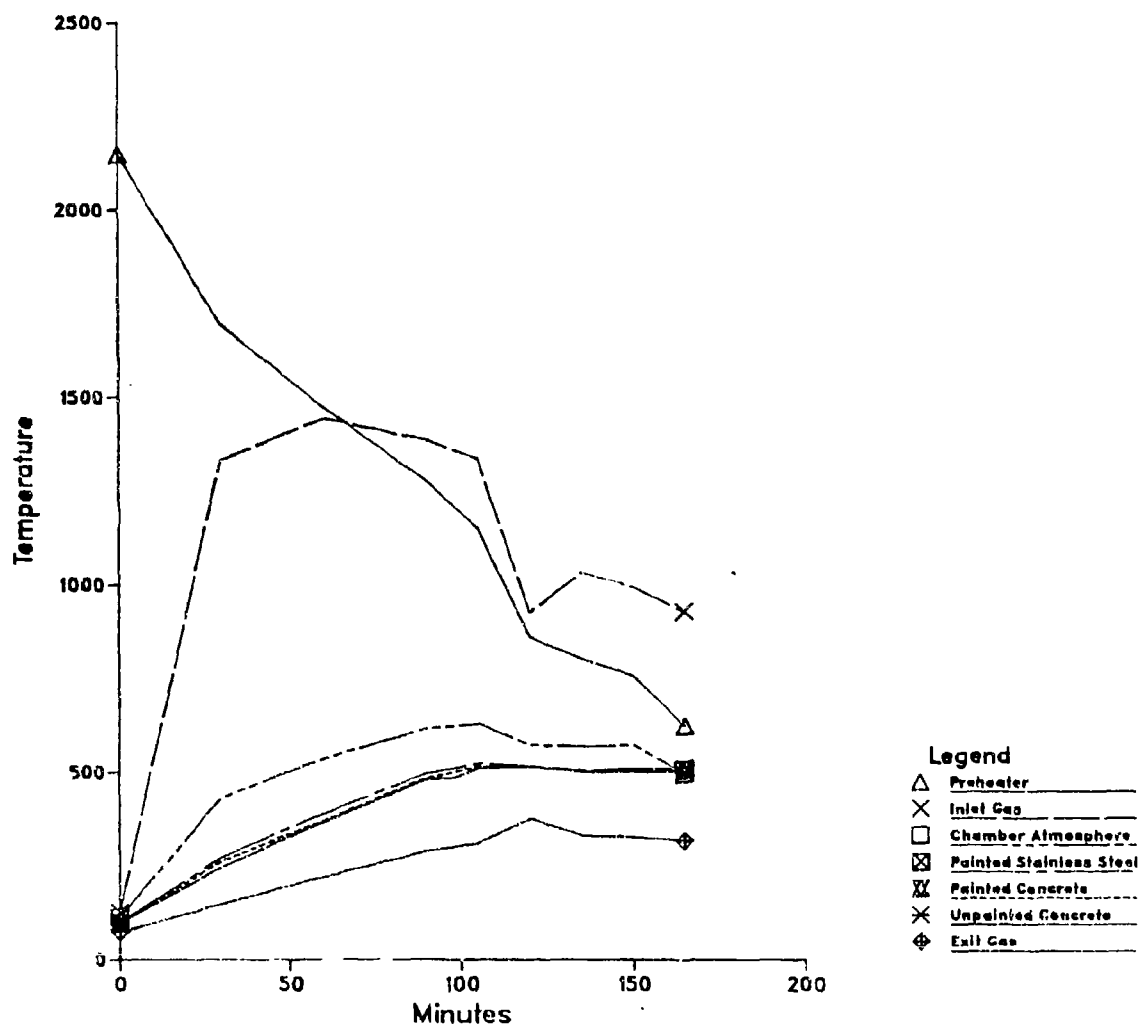
Run # 27 2,4 DNT Time–Temperature Profiles
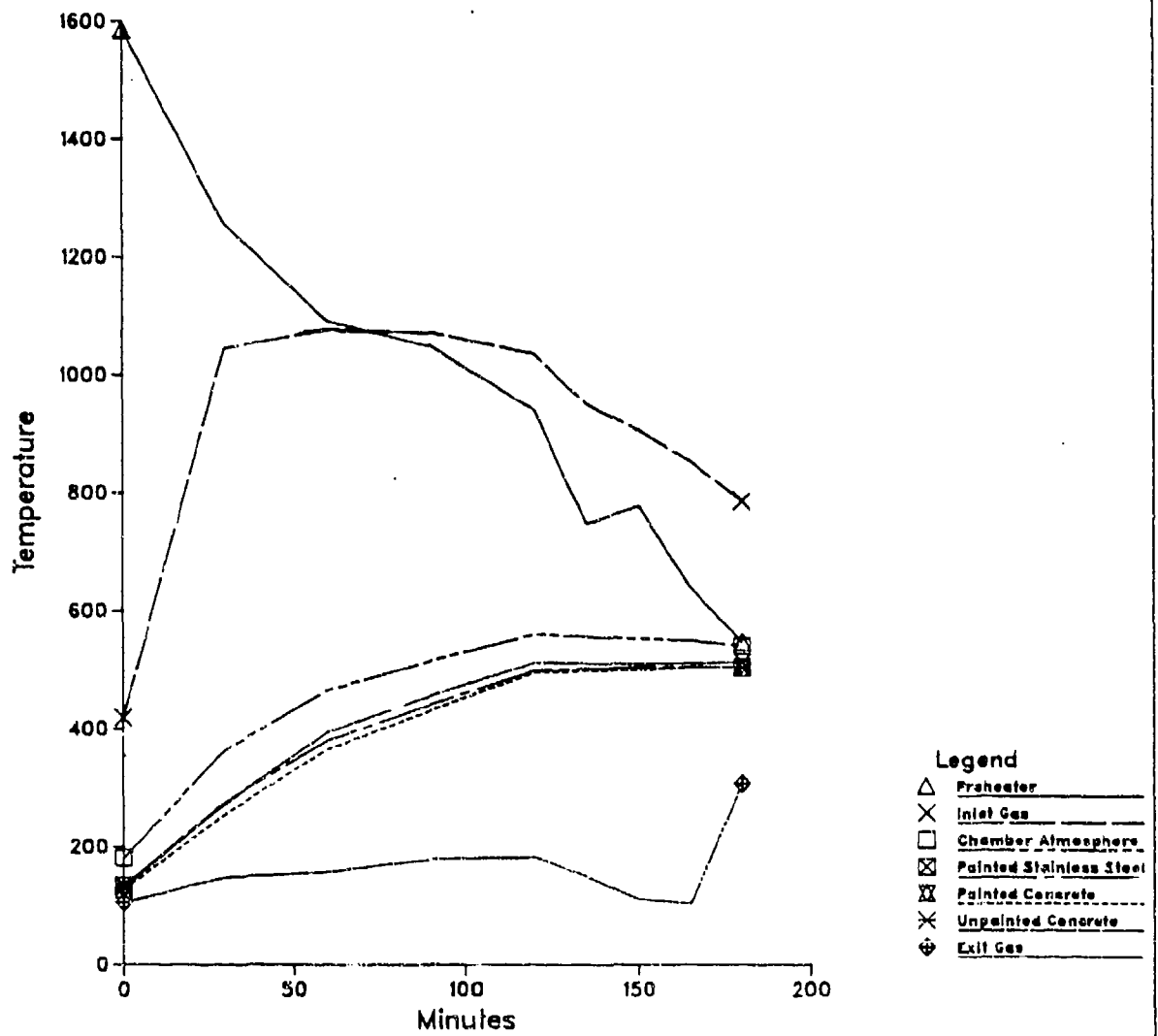
Run # 28   2,6 DNT   Time–Temperature Profiles

485

Run # 29 RDX Time—Temperature Profiles
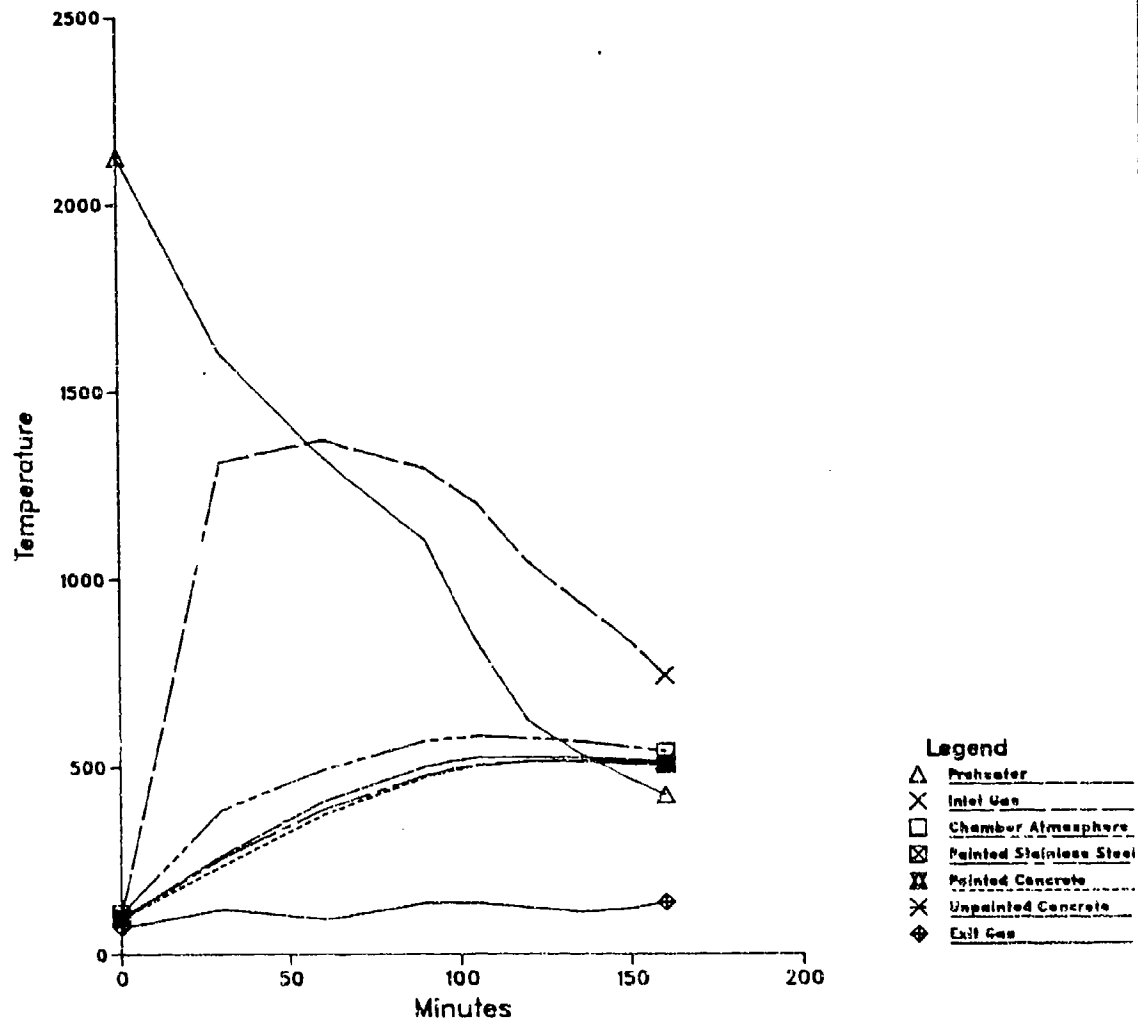
Legend
- X Inlet Gas
- □ Chamber Atmosphere
- ⊠ Painted Stainless Steel
- ⊠ Painted Concrete
- ✕ Unpainted Concrete
- ⊕ Exit Gas

Run # 30 Tetryl Time–Temperature Profiles
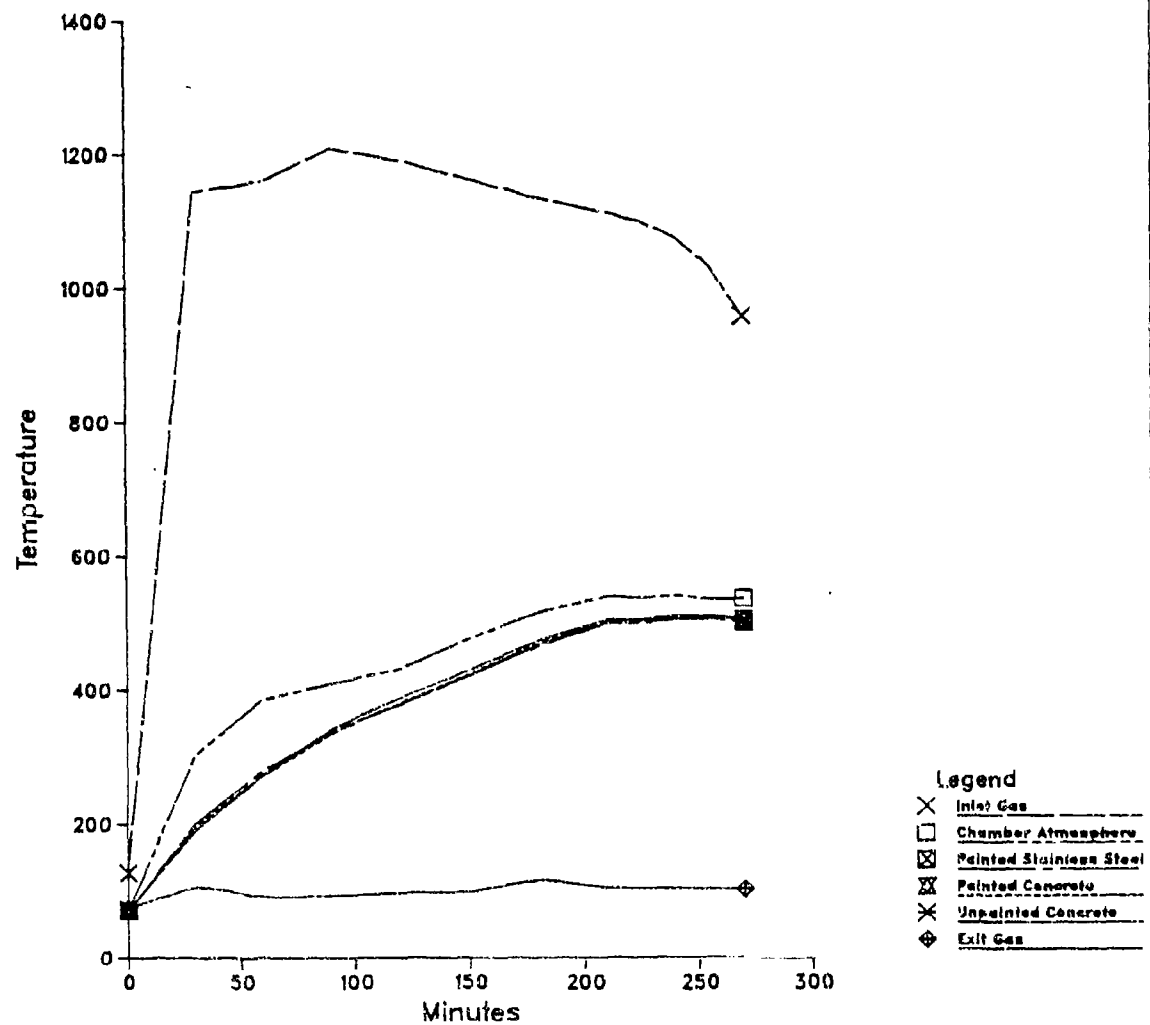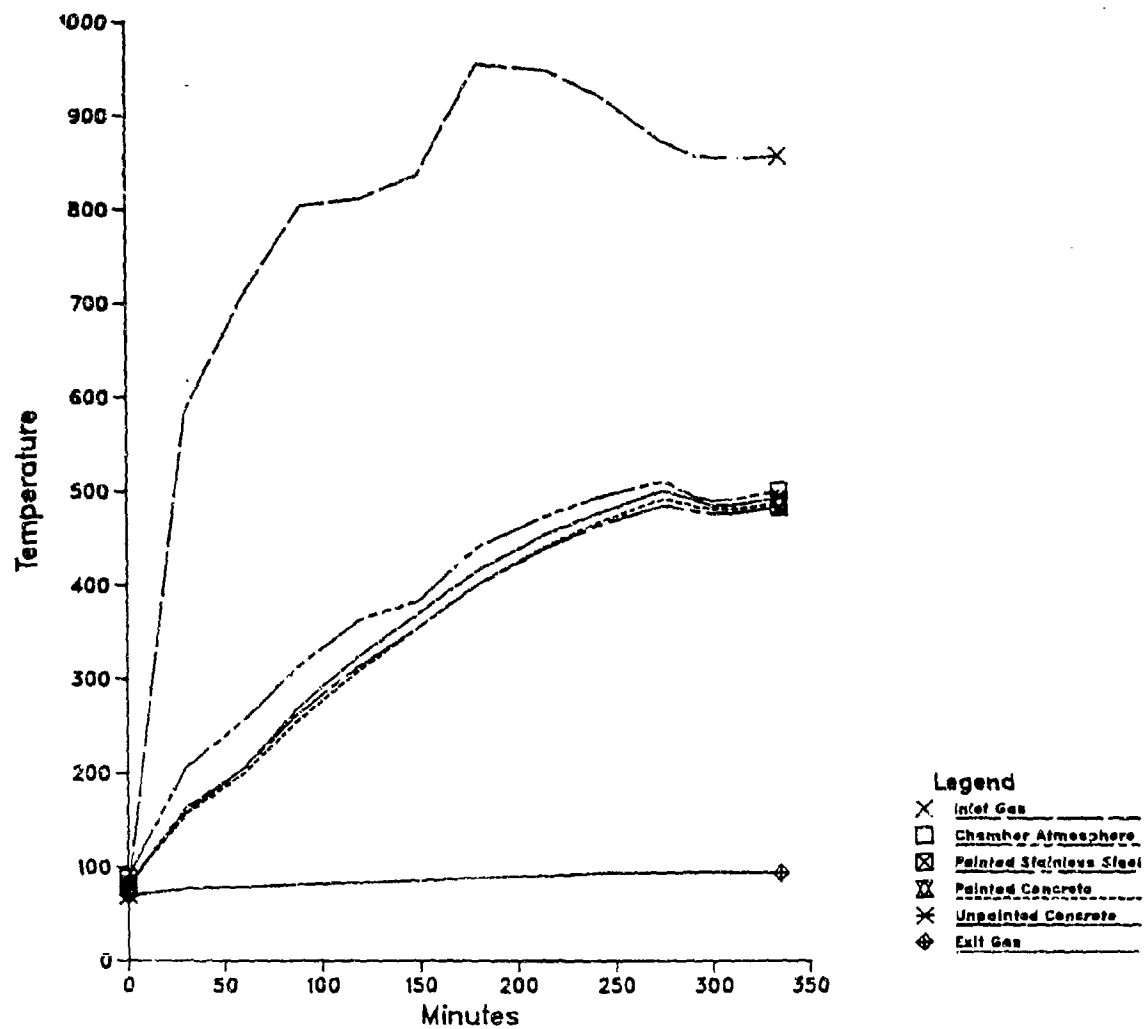
Legend
X Inlet Gas
□ Chamber Atmosphere
⊠ Painted Stainless Steel
▨ Painted Concrete
✳ Unpainted Concrete
◈ Exit Gas

Run # 31 TNT Time–Temperature Profiles

Legend
× Inlet
□ Chamber Atmosphere
⊠ Painted Stainless Steel
Ⅺ Painted Concrete
✳ Unpainted Concrete
✦ Exit Gas
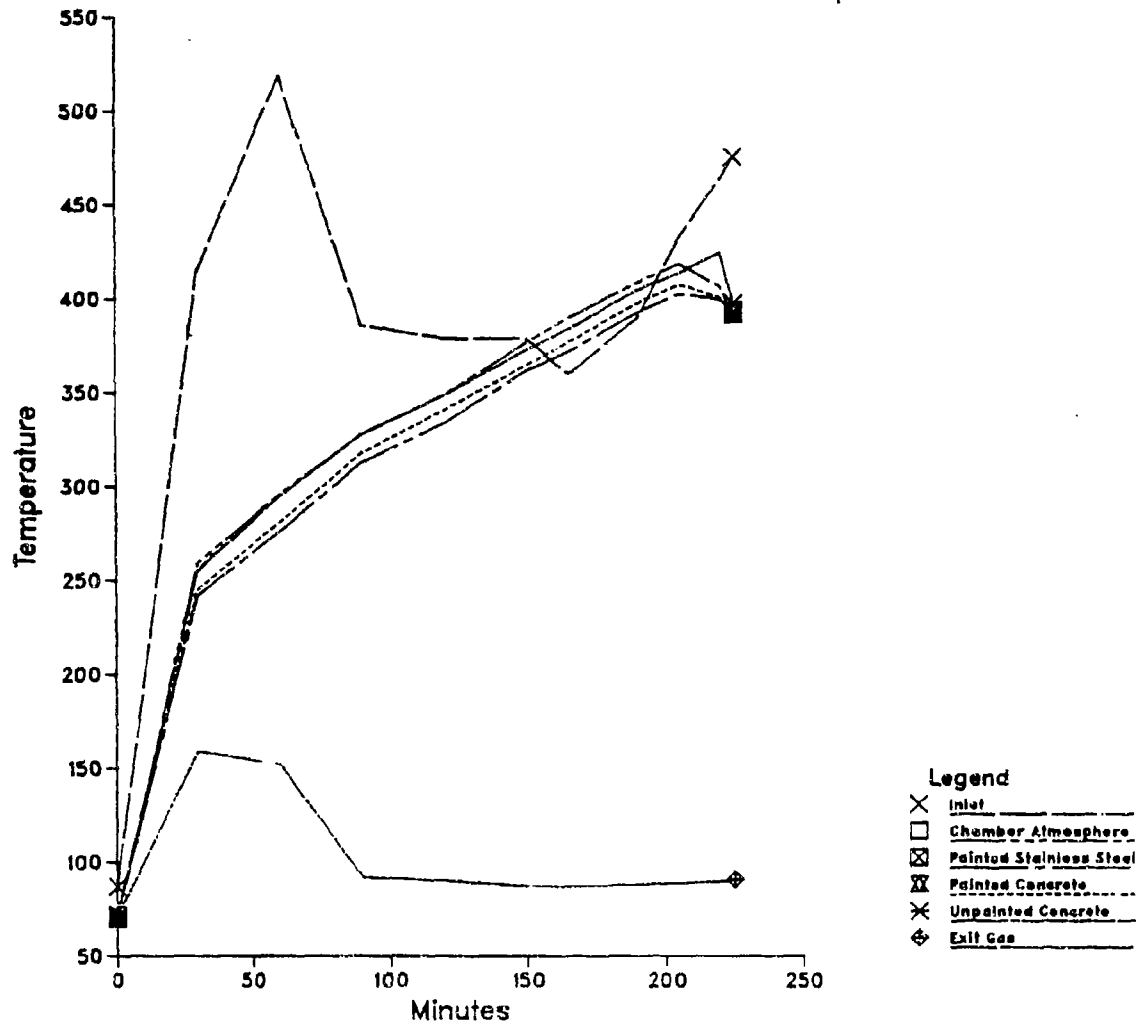
Run # 1   Date 6/10/74

Explosive __H M X; (39657-23-5)__   nominal spike of 400 mg/coupon
                                     actual spike of "_____"

Test Conditions 40% DMSO, 21 °
Spray interval - 5 min   nozzle type - ...
Spray time - 5 sec   80 psi
                                    . stainless steel

Sample

Description	Sample Volume	Concentration	Weight of Explosive	
Left Coupon	100 ml	1648 μg/ml	158.8 mg	4...
Middle Coupon	10 ...	40 μg	... mg	
Right Coupon	100.	1030 .	...	
Left Sump	377	210 " "	..	
Middle Sump		259 " "		...
Right Sump		237 " "	...	
Chamber Wash	500	387 " "	...	
			7 15...	

Remarks:

_____ for ____ore

~~IH~~
No chromatographic disturbance ...
only slight response to other compounds
was noted in the chromatograms.

Avr Removal 72.0

Run #2

Date 6/11/84

actual spike of
nominal spike of 400mg

Explosive HMX (39651-23-5)

Test Conditions 100% DMSO, 1hr
spray interval - 4min 55sec   nozzle type - 1.5
spray time - 5 sec.   60psi
                                stainless steel

Sample Description	Sample Volume	Concentration	Weight of Explosive
Left Coupon	100	13.2 %	1.35 mg
Middle Coupon	100	1.05 mg/l	0.15
Right Coupon	100	2.36 %mi	0.52
Left Sump	102	3552 mg	302 20
Middle Sump	106	2347 mg	
Right Sump	107	1517 mg	6.111
Chamber Wash	500	62.2	

Remarks: Sump samples were filtered before doing instru...
analysis
Sump samples were dark before filtering, but filtered was
clear and colorless

IA 408   2,6-DNT ; 2,4-DNT   small

409   2,6-DNT ; 2,4-DNT   small

410   2,6-DNT ; 2,4-DNT   small

404
407   No gas chromatograph...
442
411

Avg Removal 99.5

] Run __#3__   Date __/11/74__

Explosive __2,4 DNT (39652-21-11)__   actual spine of 398.4 mg/coupon

Test Conditions __100% DMSO, 1 hrs__
Spray interval - 4 min 55 sec   nozzle type ...
Spray time - 5 sec   80 psi
Sample   stainless steel

Description	Sample Volume	Concentration	Weight of Explosive
Left Coupon	100	3,44. $\frac{ug}{ml}$	4.41 mg
Middle Coupon	100	2.28 $\frac{ug}{ml}$	2... ..
Right Coupon	100	0.875 $\frac{ug}{ml}$	1... .
Left Sump	64	4333 $\frac{ug}{}$	75... .
Middle Sump	72.5	35.. $\frac{ug}{}$	...3..
Right Sump	73.5	3.12 $\frac{ug}{}$	2... .
Chamber Wash	434	... $\frac{ug}{}$	... ..

Remarks: Some of the left sump liquid was lost in transfer.
The sump solutions were amber in color.
Sump samples were titrated to ... analysis.

I4 45-7 negative disturbance ...... 5 min <
        5 min HMX small

    458   HMX small
    459   HMX small
    40.5   HMX small

    44.0
    45.1   No ... chromatographic ... disturbance
    46.2

Avg Removal $\frac{97.0}{85}$

Run # 4   Date 6/13/14

Explosive 2,4 DNT (#34457-25-11)   actual spike of 345.4 mg/coupon

Test Conditions 60% DMSO, 2 hrs
Spray interval - 5 min.   nozzle - 1.2V
Spray time - 5 sec.   54 psi
                           Stainless steel

Sample Description	Sample Volume	Concentration	Weight of Explosive
Left Coupon	100	993 $\frac{ug}{100}$	99.2 mg
Middle Coupon	100	270 $\frac{ug}{ml}$	67.5
Right Coupon	100	572 $\frac{ug}{ml}$	57.2
Left Sump	542	473 $\frac{ug}{ml}$	257.0
Middle Sump	554	417 $\frac{ug}{ml}$	
Right Sump	558	561 $\frac{ug}{ml}$	313
Chamber Wash	442	45.5 $\frac{ug}{ml}$	20.11 g
			1093.5

Remarks: No gross chromatographic disturbances
or contamination were found.

Avr Removal 81.1

492

Run # 5   Date 6/13/84

Explosive 2,4 DNT (#71451-23-11) actual spike of 5454.4 mg/coupon

Test Conditions 15% Dmso, 2 hrs
Spray interval - 5 min.   needle type - 10
Spray time - 5 sec   54 psi
                   stainless steel

Sample Description	Sample Volume	Concentration ($\mu g/ml$)	Weight of Explosive (mg)
Left Coupon	100	110	11.0
Middle Coupon	100	25.9	3.59
Right Coupon	~~100~~	—	—
Left Sump	230 / ~~542~~	664	352
Middle Sump	616	823	507
Right Sump	544	817	444
Chamber Wash	—		

Remarks: The chamber wash was lost before
samples were taken
The right coupon extract was also lost
There are no gross disturbances in
the chromatograms.

Avg Removal
98.2

G-7... ... 449

Run #1   Date 6/15/74

Explosive RDX ("A")   actual spike of 342 mg/coupon

Test Conditions 60% DMSO, 1 hrs
Spray interval - 5 min   nozzle type full
Spray time - 5 sec   50-60 psi
stainless steel

Sample Description	Sample Volume	Concentration	Weight of Explosive
Left Coupon	100	269 ug/ml	26.9
Middle Coupon	100	943 ug/ml	...
Right Coupon	100	1263 ug/ml	126.3
Left Sump	550	574 ug/ml	315.70
Middle Sump	626	440 ug/ml	275.44
Right Sump	582	414 ug/ml	240...
Chamber Wash	1354	6.2 ug/ml	...

Remarks: I.A 470
471
472 HMX small, 2,4-DNT small
473
474
2,75
476

Avg Removal
75.9

Run # 7   Date 6/13/14

Explosive RDX ("A")   actual spike of 342 mg/coupon

Test Conditions 75% Dinse, 8 hours
    spray interval - 5 min   nozzle type - 1.50
    spray time - 5 sec.   50-60 psi
Sample   stainless steel

Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)
Left Coupon	100	<0.100	<0.01
Middle Coupon	100	1.13	0.113
Right Coupon	100	1.05 (avg. of 2)	0.105
Left Sump	594	600	356
Middle Sump	596	522	311
Right Sump	602	623	375
Chamber Wash	510	19.9	10.1

Remarks: There were no gross chromatographic disturbances except for the negative and positive peaks before 4.4 min.

Avr Removal
99.97

Run # 8   Date 6/17/74

Explosive RDX ("4")   actual sp. heat 342 mg/spike

Test Conditions 75% DMSO, 25% H₂O, 6 hrs
spray interval - 5 min   needle type - 1...
spray time - 5 sec.   50-60 psi
                              stainless steel

Sample

Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)
Left Coupon	100	3.42	0.342
Middle Coupon	100	0.365	0.0365
Right Coupon	100	0.746	0.0746
Left Sump	430	824	354
Middle Sump	460	637	293
Right Sump	455	870	396
Chamber Wash	446	50.9	22.7

Remarks: There were no gross chromatographic disturbances except for the negative and positive peaks before 4.4 min.

Avg Removal
99.96

Run # 4

Date 6/4/84

Explosive _KDX ("4")_ _actual spike of 342 mg/spine_

Test Conditions 75% DMSC, 25% H₂O, C.1 N N CH, 4 ln
spray interval - 5 min.   nozzle type - 1c
spray time - 5 sec   50-60 psi
                            stainless steel

Sample

Description	Sample Volume	Concentration(μg/ml)	Weight of Explosive (mg)
Left Coupon	100	3.78	0.378
Middle Coupon	100	16.4	1.64
Right Coupon	100	37.6	3.76
Left Sump	268	0.842	0.226
Middle Sump	282	41.1	11.6
Right Sump	275	0.500	0.138
Chamber Wash	100 ?	3.08	—

Remarks: There were no gross chromatographic
disturbances except for the
negative and positive peaks befor
4.4 min.

Run # 10   Date 6/__/__

Explosive 2,4 DNT (39248-28-30) actual spine of 4.5 mg/coupon

Test Conditions 75% DMSO, 25% H2O, 0.1 N NaOH, 5 hr
spray interval - 5 min   nozzle type - 1.0
spray time - 5 sec.   50-60 psi
    stainless steel

Sample Description	Sample Volume	Concentration ($\mu g/ml$)	Weight of Explosive (mg)	
Left Coupon	100	28.5	2.85	
Middle Coupon	100	76.0	7.60	1.__
Right Coupon	100	76.8	7.68	
Left Sump	110	2.87	0.316	
Middle Sump	136	2.44	0.332	
Right Sump	(120)*	1.89	0.2__	
Chamber Wash	670	1.16	1.01	

Remarks: I 4 > 18   RDX small, a small peak ____
             RDX
       5 20   RDX small, a small peaks after
             RDX, a wide peak ____
       5 23   a series of peaks ____
       5 22   RDX small, a wide ____
       4 92   several wide ____ 2,4-DNT

       5 30   several wide peaks ____
             2,4 DNT
       5 21   several ____

* estimated volume

Run =F 11   Date 6/21, 79

Explosive 2,4 DNT (34748-55-56) actual spike of 437 mg/coupon

Test Conditions 75% DMSO, 25% H₂O, 1N NaOH, 4 hr
spray interval 5 min   nozzle type -1
Spray time - 5 sec   50-60 psi
                           stainless steel

Sample

Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)
Left Coupon	100	13.5	1.35
Middle Coupon	100	156	15.6
Right Coupon	100	0.313	0.0313
Left Sump	260	1.07	0.278
Middle Sump	260	1.13	0.294
Right Sump	330	0.595	0.196
Chamber Wash	760	0.0647	0.0492

1.30

Remarks: There is a shoulder on the ___ ___
of the 2,4-DNT peak in IA 543.
Extraneous wide peaks ___ ___
2,4-DNT in IA 543. In 545,
extraneous peaks appear > RBS _ 2,4-DNT
This sample is reshot in IA 551 and
the peaks are missing. The ___
peaks are apparently from IA 543 or
545

Run _#12_ Date _6/21_

Explosive _RDX (Sol 5)_ actual spray rate of 555 mg/coupon

Test Conditions _75% DMSO . 1N UrOA. 24HR_
Spray interval - 5 min nozzle type - 1 00
Spray time - 5 sec 50-60 psi
Stainless steel

Sample

Description	Sample Volume	Concentration (ug/ml)	Weight of Explosive (mg)	
Left Coupon	~~110~~ 100	225	22.5	6.
Middle Coupon	100	140	14.0	
Right Coupon	100	0.129	0.0129	
Left Sump	148	<0.100	<0.0148	
Middle Sump	164	6.00	0.984	
Right Sump	140	<0.100	<0.0140	
Chamber Wash	766	0.833	0.638	.05

Remarks: There are no gross chromatographic disturbances.

Run ___#14___   Date __6/22.__

Explosive ___7, 4 OWT (BWS-51-16) actual spiked at 437 mg/coupon___

Test Conditions .01 W NaOH 75% DMSO 4HR
Spray interval - 5 min   noeele type 1.00
Spray time - 5 sec   50-60 psi
                             stainless steel

Sample

Description	Sample Volume	Concentration (ug/ml)	Weight of Explosive (mg)
Left Coupon	100	111	11.1
Middle Coupon	100	238	23.8
Right Coupon	100	2.76	0.276
Left Sump	156	1.07	
Middle Sump	159	5.44	
Right Sump	157.5		
Chamber Wash	613	5.96	

Remarks: There are no gross chromatographic disturbances.

Run # 15   Date 1/26/84

Explosive RDX ("B")   actual spike at 350 mg/coupon

Test Conditions 75% DMSO, 0.01N NaOH, 4 hr
Spray interval - 5 min   Nozzle type - 100
Spray time - 5 sec   50 - 60 psi
Sample   stainless steel

Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)	
Left Coupon	100	0.167	0.0167	
Middle Coupon	100	<0.100	<0.0100	<0.003
Right Coupon	100	<0.100	<0.0100	<0.003
Left Sump	263	86.2	22.7	
Middle Sump	330	108	35.6	
Right Sump	270	346	93.4	
Chamber Wash	460	11.6	5.34	9.4

Remarks: IA 576   HMX moderate, sharp peak >
          < RDX

          579   same as 576 +

          643   HMX moderate, Tetryl   TNT
                  small, 2,4-DNT small

          644   same as 643

          645   TNT trace 2,6-DNT

          646   2,4-DNT small

          647   Peak at   DNT   not
                  2,6-DNT, 2,4-DNT

Run #16   Date 6/27/14

Explosive 2,4 DNT 34743-38-50 actual spiked 457 mg/coupon

Test Conditions

75% DMSO 4 Hrs,

Sample

Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)
Left Coupon	100	235	23.5
Middle Coupon	100	257	25.7
Right Coupon	100	298	29.8
Left Sump	315	1310	413
Middle Sump	360	1410	508
Right Sump	330	1330	439
Chamber Wash	590	86.5	51

Remarks: No gross disturbances or contaminants were found in the chromatograms.

Run _#17_   Date _1/27/74_

Explosive _RDX("B") 400 mg/coupon actual spike at 355 mg/coupon_

Test Conditions _75% DMSO - air_

Sample

Description	Sample Volume	Concentration(μg/ml)	Weight of Explosive(mg)	⊗
Left Coupon	100	35.4	3.54	1.96
Middle Coupon	100	29.9 (avg)	2.99	
Right Coupon	100	0.425 / 0.43	0.0425	
Left Sump	270	1225	331	
Middle Sump	325	970	315	
Right Sump	310	1190	369.1	
Chamber Wash	515	83.2	42.8	
			|4,13|

Remarks:

IAS 88   HMX small
          2,4-DNT small

      591   HMX small
            2,4-DNT small

      594   HMX small
            2,4-DNT small

      595   HMX small
            2,4-DN small

      596   2,4-DNT small

      613   HMX small

      623   HMX small
            2,4-DNT small

      633   HMX small   504

Run # 12   Date 6/29/84

Explosive HMX (14652-5-13) 400 m /var actual spiked + 370 7 mg/coupon

Test Conditions 75 % DMSO - 4L

Sample

Description	Sample Volume	Concentration (ug/ml)	Weight of Explosive (mg)	% rem
Left Coupon	100	6.26	0.626 .169	0.16
Middle Coupon	100	220	22.0 5.	5.5
Right Coupon	100	72.1	7.21 1.95	1.80
Left Sump	310	1020	316 85.24	79.0
Middle Sump	335	900	302	
Right Sump	320	1105	354 95.49	.5
Chamber Wash	420	105	44.1 11.93	.6
			1.06 94.55	

Remarks: No gross ✗ chromatographic disturbance
or contaminants were found in the
chromatograms.

Run #19   Date 7/3/84

Explosive Tetry (1979-53-2) actual spray rate of 279.2 mg/sample

Test Conditions 75% DMSO, 6 hours, 400 mg
Spray interval 5 min   nozzle type 1.50
Spray time 5 sec   ≤5 psi
                         stainless steel

Sample

Description	Sample Volume (ml)	Concentration (µg/ml)	Weight of Explosive (mg)	
Left Coupon	100	< 0.100	< 0.0100 <.0036 <100
Middle Coupon	100	< 0.100	< 0.0100 <.00
Right Coupon	100	< 0.100	< 0.0100 <.00
Left Sump	380	822	312	111.75
Middle Sump	449	779	350	
Right Sump	407	872	355	
Chamber Wash	420	63.4	27	

Remarks: The coupons were spray initially 3 times with almost no
space in between

IA 626   - # + Disturbance < 4.4 min
   627   - # +   "   < 4.4 "
   630   "   "   "   "
   631   "   "   < 4 min

          2,6-DNT small
          2,4-DNT small

   650   - # + Dist. < 4 min.
          TNT large
          2,4-DNT small

   651   - # + Dist. < 4 min.
          TNT large
          2,6-DNT small
          2,4-DNT small

   652   2,4-DNT small

I-505

Run # 20   Date 7/4/74

Explosive __TNT (#39748-53-6) aerosol need 366.4 mg___

Test Conditions 75% DMSO, 6 hrs 400mg
Spray interval 5 min   nozzle type 100
Spray time 5 sec   55 psi

Sample Description	Sample Volume	Concentration	Weight of Explosive
		Stainless steel	
Left Coupon	~~457~~ 100 ml	96.2 mg/l	9.52
Middle Coupon	~~530~~ 100		3.50
Right Coupon	~~465~~ 100		3.3
Left Sump	457	643	
Middle Sump	530		
Right Sump	460	748	
Chamber Wash	440		

Remarks: About 10 mg of explosive fell off right coupon into
right sump during installation
The first spray was about 20 psi

I4 640   2,4-DNT small
    653   Tetryl small
           2,6-DNT small
           2,4-DNT small

        4   ...l ...
           2,6 DNT small
           2,4 DNT small

        ...5   ... DNT ...
           ... DNT small

Run __21__   Date __1/4/95__

Explosive __TNT (29718-53-6)__   actual spike at 366.4 mg/coupon

Test Conditions _Lt and Rt. 1/4" concrete coupons spiked
with 8 ml TNT. Center coupon sandwiched between 2-1/4" coupon_
Spray time 5 sec interval 5 min total run time 6 ARS- Spray solvent (75%
0 MJD

Sample

Description	Sample Volume (ml)	Concentration µg/ml	Weight of Explosive (mg)	
Left Coupon	100	< 0.05	—	
Middle Coupon	100	476	47.6	12.94 HMX, 2.4 UNK PRE?
Right Coupon	100	9.9	.99	.??
Left Sump	480 ml	386	185.~~285~~	..7 TETRYL, 24 DNT
Middle Sump	460	0.3	0.14	? HMX PRESE
Right Sump	380	89.8	~~34,124~~ 34,2	TETRYL PRESE
Chamber Wash	580	5.2	3.01	
REAR CENTER EXTR.	?	< 0.05	—	
FRONT " "	?	< 0.05	—	

received with this batch.

Remarks:

* _IA 664 R21/CNTR SUMP/TNT has what may be an artifact eluting at 4.2 m_
(where HMX would elute). The chromatogram shows no other peaks of interes

[remaining handwritten lines illegible]

…CIAL PRC ECT OFFICE
… FILE CO Y 463

Run # 22 Date 7/6/84

Explosive TNT (39244-536) actual sprayed 366.4 mg/hour

Test Conditions 0.1 N NaOH 75% DMSO, 25% H_2O, 4 hours
spray time - 5 sec
spray interval - 5 min
pressure 50-60 psi
nozzle type - 1.00
stainless steel

Sample Description	Sample Volume ML	Concentration µg/ML	Weight of Explosive (mg)		
Left Coupon	100	1.0	0.1	.027	
Middle Coupon	100	2.2	0.22	.0260	
Right Coupon	100	1.2	0.12		
Left Sump	325	8.4	2.73	[illegible]	~~(2,6 + 2,4 DNT HMX, TETRYL PIC~~
Middle Sump	340	7.3	2.48		~~(RDX, TETRYL, 2,6 + 2,4 DNT PROB~~
Right Sump	260	10.1	2.63		~~(HMX, TETRYL, 2,6 + 2,4 DNT~~
Chamber Wash	640	0.2	0.13	.118	
				.765	

Remarks:

~~R22/ST EXTR/TNT double peak~~, {which is TNT}? large shoulder (IA669)

IA668 Peak at 4.71 is not HMX, Tetryl, 2,4-[illegible]
small peak at [illegible] min

669 blob in front of TNT, 2,4-DNT small,
wide peak at ~ 20 min

671 2,6 + 2,4-DNT small, peak at ~ [illegible] min

672 Wide peak at for TNT, Tetryl, ~~2,4~~ DNT
small, blip at 10.41 [illegible] DNT

673 Peak at 4.71 is not HMX, Tetryl, 2,4-DNT
moderate, Peak at [illegible] DNT

674 Peak at [illegible] is not HMX, Tetryl, [illegible]
DNT small

67[illegible] Tetryl, 2,4 DNT small, [illegible]

Run # 23   Date 7/9/84

Explosive _Tetry (Run-53-2) actual spills of 274.2 ms/spike_

Test Conditions 0.1 N NaOH 75% DMSO, 25% H₂O, 4 hours
spray time - 5 sec.   Pressure 55 psi
spray interval - 5 min.   nozzle type - 1.00
                              stainless steel

Sample

Description	Sample Volume ML	Concentration ug/ML	Weight of Explosive (mg)	
Left Coupon	100	< 0.1	—	TNT PRESENT
Middle Coupon	100	< 0.1	—	TNT, 2,4 DNT PRESENT
Right Coupon	100	< 0.1	—	2,4 DNT PRESENT
Left Sump	284	0.1	0.03 .0107	2,6 DNT PRESENT
Middle Sump	267	< 0.1	—	
Right Sump	216	2.2	0.475 .170	
Chamber Wash	630	< 0.1	—	2,4 DNT PRESENT

.060

Remarks:

* poorly shaped and integrated peak appears as HMX   R23/LT SUMP/TE
                                                          IA 679

IA 677   TNT, 2,4-DNT ......
  "   "S   RDX, TNT, 2,4-DNT small
    679   Peak at 14.4?, ... not 2,4 DNT
    "8"   Peak at 12.5? ... not ....
    ...   2,4-DNT small ....
    682   moderate peak at 8.05
    ...   ... Peak at 13.4? ... ...

Run # 24   Date 7/10/74

Explosive 2,6 DNT(IA74-53-4) actual spill of 261.6 mg/coupon

Test Conditions 0.1N NaOH, 75% DMSO, 25% H₂O 4 hours
Spray time 5 sec.   nozzle tip - 1.00
Spray interval 5 min   Pressure 55 psi
Sample   stainless steel

Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)	%
IA 699 Left Coupon	.100	0.289	0.0289	0.006
695 Middle Coupon	100	9.08	1.93	
695 Right Coupon	100	0.341	0.034	
750 Left Sump	320	249	79.7	19.93
Middle Sump	335	313	105	
Right Sump	240	382	91.7	22.9
694 Chamber Wash	470	64.3	30.2	2.52
Total				

Remarks:
IA 694   small humps between 5 min & 2,6-DNT
    695   wide hump at 9.45, rolling base line
          after 2,6-DNT
    698   wide peaks before 2,6-DNT seems
          to be from IA 697 (ctr. sump)
    699   TNT small, rolling baseline after
          2,6-DNT
    750   TNT small, a few small peaks
    751   Tetryl, TNT small, several small peaks
    752   TNT small, several small peaks

Run _J5_   Date _July 10_

Explosive _HMX (34748-53-12) actual spike of 175.56 ms/coupon_

Test Conditions _.1 N NaOH, 75% DMSO 25% H₂O 4Hrs_
Spray time 5 sec   nozzle type 1.00
Spray interval 5 min   Pressure 55 psi
                        Stainless steel

Sample

Description	Sample Volume	Concentration ($\mu g/ml$)	Weight of Explosive (mg)	
EA 701 Left Coupon	'')	< 0.05	< .05	
702 Middle Coupon	''))	< 0.05	< .55	
704 Right Coupon	'))	< 0.05		
706 Left Sump	280	< 0.05	< 0.0140	
707 Middle Sump	310	< 0.05	< 0.0155	
708 Right Sump	255	5.95	1.52	
708 Chamber Wash	480	< 0.05	< 0.024	.0018
				.003

Remarks:

EA 701   RDX, # 2,6-DNT, 2,4-DNT, TNT small

702   RDX, TNT small

702   RDX, tetryl, TNT, 2,6-DNT small

704   Tetryl, 2,4-DNT small
        TNT moderate

706   Tetryl, TNT small

707   RDX, tetryl, TNT, 2,4-DNT small

708   2,6-DNT, 2,4-DNT small

Run _26_   Date _7/4/71_

Explosive _TNT (39748-53-6)_ ...

Test Conditions _75% Dmso 25% H₂O_ _84°F³_ stainless steel
Spray time _5 sec_   nozzle type _1.0_
Spray interval _5 min_   pressure _55 ps._

Sample

Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)	
I4 719 Left Coupon		0.494		
710 Middle Coupon		0.258		
712 Right Coupon		0.520		
713 ~~726~~ Left Sump	545	~~10.3~~ ~~2.12~~	5.56 ~~1.16~~	
714 Middle Sump	680	67.7	46.0	
715 Right Sump	540	165	89.1	
718 Chamber Wash	460	3.58	1.65	.15

Remarks:

I4 719   Tetryl, 2,4-DNT small

710   2,4-DNT small

712   RDX, Tetryl, 2,6-DNT small

713   RDX moderate
      Tetryl, 2,6-DNT, 2,4-DNT small

714   Tetryl, 2,6-DNT, 2,4-DNT small

715   peak at 2.59 is not RDX,
      Tetryl, 2,6-DNT, 2,4-DNT small

718   peaks at 2.55, ... moderate
      2,4-DNT small

Run #27   Date 7/12/84

Explosive 2,4 DNT(μ74,-53-8) (actual spike of 34 μg (μg.coupon))

Test Conditions 75% DMSO, 8 hours
Spray time 5 sec   nozzle type 1.00
Spray interval-5 min   pressure
                          stainless steel

Sample

Description	Sample Volume	Concentration(μg/ml)	Weight of Explosive (mg)
Left Coupon	100	<0.05	<0.00500
Middle Coupon	100	0.386	0.0386
Right Coupon	100	0.332	0.0332
Left Sump	515	~~2.43~~ 633	326
Middle Sump	685	560	384
Right Sump	505	722	365
Chamber Wash	450	35.9	16.2
			30.77

Remarks:
£4 711   TNT small
   713   TNT small
   720   TNT, 2,6-DNT small
   72   2,6-DNT small
   735   2,6-DNT small
   754   2,6-DNT small
   723   RDX, tetryl, TNT, 2, DNT small

Run #28   Date July 17,

Explosive TNT ( )

Test Conditions

3 concrete

<u>Sample</u>

Description	Sample Volume	Concentration (ug/m)	Weight of Explosive (mg)
TA 740 Left Coupon		0,547	
739 Middle Coupon		0,325	
741 Right Coupon		0,299	
746 Left Sump	300	11,3	
745 Middle Sump	290	8,62	
744 Right Sump	260	13,1	
743 Chamber Wash	560	0,260	

Remarks:

Run #.30   Date 7/23/84

Explosive _Tetryl (39748-.53-2) actual(spin) of 279.2 mg/gram_

Test Conditions 75% DMSO, 25% H₂O, 0.1N NaOH, 4 hours
Spray time - 5 sec   Nozzle type 1.00
Spray interval - 5 min   pressure - 53 psi
                            2 concrete, 1 painted concrete

Sample

Description	Sample Volume (ml)	Concentration (µg/ml)	Weight of Explosive (mg)	
I4 756 Left Coupon	100	94.2	9.42	
771 Middle Coupon	100	26.7	26.7	
painted - 772 Right Coupon	100	6.02	0.602	
757 Left Sump	276	1.44	0.397	
773 Middle Sump	330	<0.100	<0.033	
painted - 774 Right Sump	330	2.90	0.957	
758 Chamber Wash	610	1.50	0.915	

Remarks:

I4 756   major peak at 13.54 min, 2,4-DNT sm.
          a few small peaks

758   peak at 13.53 ... twice the size of T

757   major peak at 13.58, a few small ten
          peaks

771   major peak at 13.48, 2,4-DNT small
          a few small peaks

772   2,4-DNT small, major peak at

773   13.60 major peak

774   13.61 large peak

Run #31   Date 7/24/34

Explosive 2,6 DNT (#39741-53-4) actual spread of 261.6mg/coupon

Test Conditions 75% DMSO 0.1N NaOH 4hrs
spray time 5 sec   nozzle type 1.00
spray interval 5 min.   pressure 53.5 psi
                              concrete & painted concrete

Sample

Description	Sample Volume (ml)	Concentration (ug/ml)	Weight of Explosive (mg)	
775 Left Coupon	150	1480	148	.30
776 Middle Coupon	100	1080	108	.5
778 painted — Right Coupon	100	120	12.0	_.0
779 Left Sump	268	146	39.1	
780 Middle Sump	356	24.3	8.65	.15
75-1 painted — Right Sump	200	430	86.0	
761 Chamber Wash	440	15.0	6.60	

Remarks:
F4 775   7,4-DNT small
     776   2,4-DNT small
     778   —
     779   TNT small
     780   TNT small
     781   TNT small, several small peaks
     761   RDX TNT small, several small peaks

Run #32   Date 7/25/84

Explosive RDX (29741-53-10) all or sp heat 2.8% as loaded

Test Conditions 75% DMSO 25% H₂O 0.1N NaOH 4 hrs
Spray time 5 sec   nozzle type 1.00
Spray interval 5 min   pressure 53 psi
                                     concrete and painted concrete

Sample Description	Sample Volume	Concentration (µg/ml)	Weight of Explosive (mg)
IA 75-3 Left Coupon	100	227	22.7
75-4 Middle Coupon	100	51.7	5.17
painted 75-5 Right Coupon	100	22.3	2.23
75-6 Left Sump	176	<0.100	<0.0176
75-7 Middle Sump	240	<0.100	<0.0240
painted 75-8 Right Sump	444	0.148	0.0657
75-9 Chamber Wash	430	0.390	0.165

Remarks: The painted concrete may have been contaminated
with some 2,6 DNT
IA 75-3   major peak at 4.74 (HMX?), 5,49 -RDX ?
   2,6 - DNT small
75-4   HMX large, 2,6-DNT, 2,4-DNT
75-5   HMX moderate, TNT 2,6-DNT, 2,4-DNT
       small
75-6   5,50 major peak, TNT small
75-7   5,49 major peak, TNT small
75-8   5,35 major peak
75-9   HMX small

Run # 33   Date 7/25/84

Explosive 2,4 DNT (99748-53-8) at 4 ppm at 203.6 mg/cm²

Test Conditions 75% DMSO 25% H₂0 0.1N NaOH 4 hrs.
  spray time 5 sec   nozzle type 1.00
  spray interval 5 min   pressure 53 psi
  Sample   concrete and painted concrete

Sample Description	Sample Volume	Concentration (μg/ml)	Weight of Explosive (mg)	
IA 793 Left Coupon	100	7.31	0.731	.356
794 Middle Coupon	100	14.3	1.43	
painted 795 Right Coupon	100	1.83	0.183	
796 Left Sump	560	<0.0500	<0.0185	
797 Middle Sump	358	<0.0500	<0.0179	
painted 798 Right Sump	370	<0.0500	<0.0185	
799 Chamber Wash	550	0.0942	0.0518	

Remarks:
  IA 793   9.02 major peak, 2,6-DNT small, RDX,
    & 794   TNT trace

  795 794   9.02 small, 2,6-DNT small, TNT trace

  796   9.15 moderate, tetryl small, RDX, TNT
                                                 trace

  797   RDX, tetryl small, TNT, 2,6-DNT trace,
               numerous small peaks

  798   TNT moderate, RDX small numerous
               small peaks

  799   TNT small

Run # 34   Date 7/26/84

Explosive _HMX (39741-53-12) actual ppi not 2.11.3 mg/ml_

Test Conditions 75% DMSO 0.1N NaOH 4 hours
spray time 5 sec   nozzle type 1.00
Spray interval 5 min   concrete & painted concrete
                                    pressure 50 psi

Sample

Description	Sample Volume (ml)	Concentration (µg/ml)	Weight of Explosive (mg)
I4 512 Left Coupon	100	651	65.1
513 Middle Coupon	100	487	48.7
804 Right Coupon	100	29.2	2.92
805 Left Sump	300	<0.05000	<0.0150
806 Middle Sump	304	<0.05000	<0.0152
807 Right Sump	382	<0.05000	<0.0191
808 Chamber Wash	350	<0.05000	<0.0175
			.79

Remarks:

I4 512   2,6-DNT trace

513   ——

804   Tetryl, 2,6-DNT, 2,4-DNT trace

805   Tetryl trace, 5.34 moderate

806   5.31 moderate, tetryl trace

807   5.40 small

808   ——

Run # 35   Date 5/13/64

Explosive 2,6 - DNT (stock solution #34741-53-4) atmospheric distillation oxygen

Test Conditions
Spray: 1 hours of a 75% DMSO, 25% H₂O, 0.1% ; Internal Strain Pressure 50psig Spray Time 5 sec
Heat: Heat to 250-300°F for 1 hour

Sample

Description	Sample Volume (ml)	Concentration (µg/ml)	Weight of Explosive (mg)	% Recovered
1 A S 17 Left Coupon	250	26.7	6.68	2.50
S-18 Middle Coupon	250	9.94	2.49	0.95
S-19 Right Coupon	250	15.5	3.88	1.48
Left Sump				
Middle Sump				
Right Sump				
S20 Chamber Wash CH₃CN	700	2.06	1.44	0.18
S21 Chamber Wash H₂O	500	0.403	0.202	0.026
			14.69	1.88

Remarks:

I A S-17;
S 18 } TNT large, RDX small, HMX, tetryl, numerous trace peaks
S 19 J

S 20 RDX, 2,4-DNT small, HMX, tetryl, TNT trace, numerous small peaks.

S 2.1 Tetryl, TNT, 2,4-DNT + trace

Run #36   Date 8/15/8_

Explosive R D X (stock solution # 8174-58-_) _____

Test Conditions

Spray: 75% DMSO, 25% H₂O, 0.1N for 1 Hr ; Interval: 5 min
                                                Pressure: 35 psig
                                                Spray time: 5 sec

Heat: 250-300°F for one Hr, min: 250°F
                            max: 330°F

Sample

Description	Sample Volume (ml)	Concentration (μg/ml)	Weight of Explosive (mg/ml)	% actual spike
IA 825 Left Coupon	250	0,132	0,0330	.0144
826 Middle Coupon	250	46,7	11,7	**5.09**
827 Right Coupon	250	0,147	0,0368	.0160
~~Left Sump~~				
~~Middle Sump~~				
~~Right Sump~~				
828 Chamber Wash H₂O	500	0,341	0,171	.0248
829 Chamber Wash CH₃CN	500	0,106	0,0530	.00734
830 ~~Remarks:~~ Imp. Trap	250	0,231	0,0575	.00839
			Total 12,0576	1.73

Remarks!

IA 825   HMX moderate, Tetryl, TNT, 2,6-DNT, 2,4-DNT
              trace

826 }   HMX small, Tetryl, TNT, 2,6-DNT, 2,4-DNT
827 }   trace

829   peak at 4.92 is not HMX, ...
              2,6-DNT, 2,4-DNT trace

Run ___#37___   Date ___5/16/84___

Explosive __RDX__ stock solution 5974r-5570, actual spillage of 227.6 mg/coupon

Test Conditions

Spray: 25% MnSO 25%H2O, 0.1N for 4 hours; Interval 5 min, Pressure 55 psig, Spray time 15 sec.

Heat: One hour range of 250-300°F   max Temp: 326
                                    min Temp: 242

Sample

Description	Sample Volume (ml)	Concentration (µg)	Weight of Explosive (mg)	
IA 834 Left Coupon	250	0.310	0.0775	
835 Middle Coupon	250	<0.100	<0.0250	
836 Right Coupon	250	<0.100	<0.0250	~0.?
542 {Left Sump, Middle Sump, Right Sump}	500	0.448	0.224	
839 Injection Chamber Wash	500	0.136	0.0680	0.??
837 C13CN	500	<0.100	<0.0500	<0.00
838 H2O	750	<0.100	<0.0750	<0.011
			0.5445 0.079	

Remarks:

IA 834 }
835 }   HMX small to large, tetryl, TNT, 2,6-DNT,
836 }   2,4-DNT trace

837   Tetryl, TNT, 2,6-DNT, 2,4-DNT trace

838   HMX trace

839   HMX, TNT, 2,6-DNT small, 2,4-DNT trace

842   Tetryl, TNT small

Run _#38_   Date _8/20/84_

Explosive _2,6 DNT Spectr solution #34745-53-4, octoclay, nut F 2,2 Bitri/trops_

Test Conditions

Spray: 4 Hours 75% DMSO 25% H₂O O₁N₂ Interval 5min, Spray time 5sec, Pressure 5.5psig,

Heat: for 1 Hour in 250-500°F   max Temp
                                min Temp

[note: 1 coupon (the middle) was omitted from the spray test + only submitted to the heat unit test]

Sample

Description	Sample Volume	Concentration(μg)	Weight of Explosive mg	
IA 843 Left Coupon	250	0.343	0.0858	
844 Middle Coupon	250	166	41.5	
845 Right Coupon	250	5.82	1.46	0.63?
848 { Left Sump / solvent pools / Middle Sump / Right Sump }	500	<0.100	0.0500	
849 impinger trap Chamber Wash	250	18.0	4.50	1.?
847 CH₃CN	500	0.304	0.152	0.?
846 H₂O	500	0.187	0.0935	?

Remarks:

+.541   6.97

IA 843   RDX, TNT small

844   RDX, 2,4-DNT small, TNT large

845   TNT large, RDX small

~~846~~

847   RDX small

848   RDX moderate, tetryl small, numero
       small peaks

849   peak at 10.54 is not TNT, 2,4-D
       trace

HOT GAS DECONTAMINATION TEST DATA

Chamber Residue Distribution

Run No.	Explosive	Front Chamber			Rear Chamber		
		Effluent, mg	Washdown, mg	Total, mg	Effluent, mg	Washdown, mg	Total, mg
20	HMX	0.293	0.305	0.598	(a)	0.072	0.072
21	2,4 DNT	152.32	0.0463	153.0	(a)	0.179	0.179
22	Tetryl	0.613	<0.025	6.13	(a)	<0.025	<0.025
23	RDX	6.13	<0.025	6.13	(a)	<0.025	<0.025
24	2,6 DNT	155.04	<0.025	155.0	(a)	0.122	0.122
25	TNT	57.9	<0.0125	57.7	(a)	0.0373	0.037
26	HMX	0.148	0.131	0.279	<0.0125	<0.0125	<0.025
27	2,4 DNT	146.88	<0.0125	146.88	<0.0125	4.43	4.43
28	2,6 DNT	182.09	<0.025	182.0	<0.025	1.62	1.62
29	RDX	2.37	<0.025	2.37	<0.025	<0.025	<0.05
30	Tetryl	0.65	<0.025	0.65	<0.025	<0.025	<0.0ᶠ
31	TNT	(b)	(b)	(b)	(b)	(b)	(b)

(a) No rear chamber purge flow in use.

(b) No data available.

APPENDIX IV

HOT GAS CONCEPT SAMPLE

HEAT TRANSFER CALCULATIONS

Hot Gas Concept Concept Sample Heat
Transfer Calucation

The sample calculations detailed on the following pages are intended as an example of the methodology used in performing heat balances on the model structures. The valves used in this example for building parameters are arbitrary and do not match either of the model structures used in this task. In order to duplicate the exact task calculations, these building specific parameters must be changed to match the building being analyzed.

BUILDING HEAT TRANSFER CALCULATIONS

Heat balances were performed on each of the representative explosive contaminated structures. In the heat balances the hot gas flow rate, inlet gas temperature and insulation thickness were varied to evaluate their effect on the feasibility and cost of the concept. The range of values used for the variables are as follows.

- o Hot gas flow rate-1000, to 20,000 scfm
- o Inlet gas temperature-1500 and 2000 F
- o Insulation thickness-2, 4 and 12 inches

A description of the calculation procedure, including a sample calculation, and the results of the calculation are given in the next sections.

Building Heat Transfer Calculation Procedure

The method used to calculate heat requirements and times for each of the buildings involved an iterative method. The following are the steps in the calculation procedure:

Step 1: The hot gas outlet temperature (TOUT) is estimated.

Step 2: The inside heat transfer coefficient (HI) is calculated.

Step 3: The inside wall temperature (TW1) is calculated.

Step 4: The temperature profiles in the concrete walls and/or floor are calculated.

Step 5: A heat balance is performed where the heat released by the gas is compared with the heat absorbed by the concrete and the heat losses. Steps 1 through 4 are repeated until a heat balance is achieved.

Step 6: Steps 1 through 5 are repeated for the next time increment.

An example of the calculations using the Building parameters specific to Task 3 of H=15 ft, L=150 ft, W=70 ft and a 2000 scfm hot gas flow at a 2000 F inlet gas temperature is given as follows. These building parameters must be changed to match the specific structure being modled. Definitions of the variables used in the calculations are given in Table 1.

Preliminary Calculations

$$AREA1 = (150 \times 70) + (150 \times 15) \times 2 + (70 \times 15) \times 2 = 17100 \text{ ft}^2$$
$$AREA2 = 150 \times 70 = 10500 \text{ ft}^2$$
$$CSAREA = 70 \times 15 = 1050 \text{ ft}^2$$
$$HYDD = 4 \times CSAREA/PERIMETER = 4 \times 1050/(140 + 30) = 24.7 \text{ ft}$$

Step 1

In the first approximation TEMP1 = 70 F. Assume TOUT increases by 50 F in the first time interval of one hour.
$$TOUT = TEMP1 + 50 = 70 + 50 = 120 \text{ F}$$

Step 2

The specific heat and average temperature of the hot gas is then calculated as follows:
$$TAVG = 460 + (TOUT + TIN)/2 = 1520 \text{ R}$$
$$CP^* = .2232 + 2.69 \times 10^{-5} \times TAVG = .2642 \text{ BTU/lb/F}$$
The heat released by the gas is calculated as follows:

---

* Derived from physical properties of air at 80 to 2060 F.

TABLE 1. DEFINITIONS OF VARIABLES USED IN THE
BUILDING HEAT TRANSFER CALCULATIONS

FLOW = Hot Gas flow rate = 1000, 2000, 3000, or 4000 SCFM
TIN = Inlet gas temperature = 1500 or 2000 F
TO = Initial building temperature = 70 F
TEMP1 = Current inside wall temperature (F)
FINF = Infiltration air flowrate = 100 SCFM
H = Height of building (ft)
L = Length of building (ft)
W = Width of building (ft)
AREA1 = Building surface area exposed to soil (ft^2)
AREA2 = Building surface area exposed to air (ft^2)
CSAREA = Cross Sectional area of hot gas flow (ft^2)
HYDD = Hydraulic diameter of building in direction of the hot gas flow (ft)
TIME = Calculation time increment = 1 hr
DEPTH = Concrete thickness (ft)
CCON = Thermal conductivity of concrete = .7 BTU/ft/hr/F
CPCON = Specific heat of concrete = .21 BTU/lb/F
DENSC = Density of concrete = 144 lb/ft^3
ALPHA = Thermal diffusivity of concrete = CCON/(CPCON X DENSC)
CINS = Thermal conductivity of insulation = .05 BTU/ft/hr/F
DINS = Insulation thickness = 0.167, 0.333 or 1.000 ft
CSOIL = Thermal conductivity of soil = .3 BTU/ft/hr/F
CPSOIL = Specific Heat of soil = .44 BTU/lb/F
DENSSL = Density of soil = 128 lb/ft^3
TOUT = Hot gas outlet temperature (F)
HI = Inside heat transfer coefficient (BTU/ft^2/hr/F)
TAVG = Average Temperature of the hot gas (R)
CP = Specific heat of the hot gas (BTU/lb/F)
QGAS = Heat released by the gas (BTU)
CONG = Thermal conductivity of the hot gas (BTU/ft/hr/F)
VIS = Viscosity of the hot gas (lb/ft/sec)
DENS = Density of the hot gas (lb/ft/sec)
VEL = Velocity of the hot gas (ft/sec)
HRAD = Radiation heat transfer from the hot gas (BTU/ft^2/hr/F)
RE = Reynolds number
PR = Prandtl number
HFCON = Forced convection heat transfer from the hot gas (BTU/ft^2/hr/F)
GR = Grashof number
HNCON = Natural convection heat transfer from the hot gas (BTU/ft^2/hr/F)
HCOND = Conduction heat transfer from the hot gas (BTU/ft/hr/F)
TW1= Inside wall temperature (F)
RATIO = Dimensionless spacing interval
CMASS = Mass of concrete in the building (lb)
QABS = Heat absorbed by the concrete (BTU)
QHL1 = Heat loss to the soil (BTU)

TABLE 1. (Continued)

QINF = Heat loss due to air infiltration (BTU)
QHL2 = Heat loss to air and heat loss to air infiltration (BTU)
QTOTAL = Total heat loss (BTU)
CPINS = Specific heat of insulation = 0.2 BTU/lb/F
DINS = Thickness of insulation (ft)
WINS = Weight of insulation = DINS X AREA2 X 10 lb/ft^3
QINS = Heat absorbed by the insulation (BTU)
CPMET = Specific heat of the metal = 0.11 BTU/lb/F
WMET = Weight of metal = 10 ton X 2000 + other metal (lbs)
QMET = Heat absorbed by the metal (BTU)

$$QGAS = q_{gas} = TIME = mCp\Delta T$$

where m = FLOW(SCFM)x60(min./hr)x29(1b/1b-mole)/359(SCF/1b-mole)

           TIME = 1 hr increment

           CP = .2642 (BTU/1b/F)

           ΔT = (TIN - TOUT)(F)

           QGAS = 2000x60x29x1/359x.2642x(2000 - 70) = 4.9428×10^6 BTU

The effects of heat transfer by radiation, forced convection, natural convection, and conduction are considered in calculating the inside heat transfer coefficient as follows:

Radiation:

$$HRAD = \sigma \varepsilon_{EFF}(TAVG^4 \ T2^4)/(TAVG - T2)$$

where σ = 1.714×10^{-9} BTU/hr/ft^2/R^4

           ε_{EFF} = Effective emissivity

           T2   = TEMP1 + 460 = 70 + 460 = 530 R

           TAVG = 1520 R

For combustion of methane in 100 percent excess air, the resulting flue gas contains 10 molar percent H_2O and 5 molar percent CO_2.

$$\text{Let } L = 3.4 \times \frac{volume}{surface\ area}$$

$$= 3.4 \ \frac{H \times L \times W}{AREA1 + AREA2}$$

$$= 3.4 \ \frac{15 \times 150 \times 70}{17100 + 10500} \ = 19.4$$

PwL = 0.1 x 19.4 = 1.9

At TAVG = 1520 R, ε_W = 0.33[a]

PcL = 0.05 x 19.4 = 1.0

At TAVG = 1520 R, ε_c = 0.15[b]

The emissivity correction factor due to spectral overlap of water vapor and carbon dioxide is neglected. The gas emissivity is then,

$$\varepsilon_{GAS} = 0.33 + 0.15 = 0.48$$

$$\varepsilon_{CONCRETE}(unpainted) = 0.63$$

$$\varepsilon_{EFF} = \frac{1}{\frac{1}{0.48} + \frac{1}{0.63} - 1} = 0.37$$

$HRAD = 6.342 \times 10^{-10} \times (TAVG^4 \ T2^4)/(TAVG - T2)$

$HRAD = 6.342 \times 10^{-10} \times (1520^4 - 530^4)/(1520 - 530) = 3.3690 \ BTU/ft^2/hr/F$

Forced Convection:

$CONG(c) = 4.077 \times 10^{-4} + 3.071 \times 10^{-5} \times TAVG - 6.557 \times 10^{-9} \times TAVG^2$
$+ 7.890 \times 10^{-13} \times TAVG^3 = .0347 \ BTU/ft/hr/F$

$VIS(c) = (.2067 + 2.216 \times 10^{-3} \times TAVG - 5.779 \times 10^{-7} \times TAVG^2 +$
$8.031 \times 10^{-11} \times TAVG^3) \times 10^{-5} = 2.52 \times 10^{-5} \ lb/ft/sec$

$DENS(c) = 39.733/TAVG = .0261 \ lb/ft^3$
$VEL = FLOW(SCFM) \times TAVG(F)/CSAREA(ft^2)/60(sec/min)/492(F)$
$VEL = 2000 \times 1520 \times /1050/60/492 = .098 \ ft/sec$

$RE = HYDD(ft) \times DENS(lb/ft^3) \times VEL(ft/sec)/VIS(lb/ft/sec)$
$RE = 24.7 \times .0261 \times .098/2.52 \times 10^{-5} = 2507$

$PR =$
$VIS(lb/ft/sec) \times CP(BTU/lb/F) \times 3600(sec/hr)/CONG(BTU/ft/hr/F)$
$PR = 2.52 \times 10^{-5} \times .2642 \times 3600/.0347 = .6907$

$HFCON = Nu \times CONG/HYDD$

where $\quad CONG = .0347 \ BTU/ft/hr/F$
$HYDD = 24.7 \ ft$
$Nu = .023 \ RE^{.8} PR^{.3}$
$RE = 2507$
$PR = .6907$

$HFCON = (CONG/HYDD) \times .023 \times RE^{.8} \times PR^{.3}$
$HFCON = .0347/24.7 \times .023 \times 2507^{.8} \times .6907^{.3} = .01515 \ BTU/ft^2/hr/F$

Natural Convection:

$BETA(c) = (4.17 - 6.088 \times 10^{-3} \times TAVG + 3.684 \times 10^{-6} \times TAVG^2 -$
$7.812 \times 10^{-10} \times TAVG^3) \times 10^{-3} = .0684 \times 10^{-3} \ F^{-1}$

(a) Table 4-15 from Heat Transmission by W. H. McAdams, pub. by McGraw-Hill, 1954.

(b) IBID, Table 4-13.

(c) Derived from physical properties of air at 80 to 2060 F.

$$GR = DENS^2(lb^2/ft^6) \times 32.174(ft/sec^2) \times BETA(F^{-1}) \times HYDD^3(ft^3) \times$$
$$(TAVG - T2)(F)/VIS^2(lb^2/ft^2/sec^2)$$
$$GR = (.0261)^2 \times 32.174 \times .000684 \times (24.7)^3 \times (1520 - 530)/(2.52 \times 10^{-5})^2$$
$$GR = 3.5218 \times 10^{11}$$

$$HNCON = Nu \times CONG/HYDD$$

where $CONG = .0347$ BTU/ft/hr/F
         $HYDD = 24.7$ ft
         $Nu = .548(GrPr)^{.25}$
         $Gr = GR = 3.5218 \times 10^{11}$
         $Pr = PR = .6907$

$$HNCON = (CONG/HYDD) \times .548 \times (GR \times PR)^{.25}$$
$$HNCON = .0347/24.7 \times .548 \times (3.5218 \times 10^{11} \times .6907)^{.25}$$
$$= .5407 \text{ BTU/ft}^2/hr/F$$

Conduction:

$$HCOND = CONG/\sqrt{(4 \alpha_{gas} \times TIME)}$$

where $CONG = .0347$ BTU/ft/hr/F
         $TIME = 1$ hr
         $\alpha_{gas} = CONG/(DENS \times CP)$
         $CP = .2642$ BTU/lb/F

$$HCOND = .0347/\sqrt{(4 \times 1 \times .0347/(.0261 \times .2642))} = .0077 \text{ BTU/ft}^2/hr/F$$

The total inside heat transfer coefficient is:

$$HI = HRAD + HFCON + HNCON + HCOND$$
$$HI = 3.3690 + .01515 + .5407 + .0077 = 3.9325$$
$$\text{BTU/ft}^2/hr/F$$

Step 3:

The inside wall temperature is calculated as follows:
$$TW1 = TOUT - QGAS/(TIME \times HI \times (AREA1 + AREA2))$$
$$TW1 = 120 - 4.9428 \times 10^6/(1 \times 3.9325 \times (17100 + 10500))$$
$$= 75.649 \text{ F}$$

Step 4:

The temperature profile in concrete is calculated as follows:

For the heat absorbed by metal in the building:

$$QMET = CPMET \times WMET \times (TW2_{CURRENT} - TW2_{PREVIOUS})/2 \times TIME$$

where

$$WMET = 10 \times 2000 + \frac{0.05*}{12} \times AREA2 \times 488 = 41350 \text{ lb}$$

$$QMET = 0.1^1 \ \ 1350 \times (70.01-70)/2 \times 1 = 23$$

For the first iteration the temperature increase of the concrete wall is calculated as follows:

$$TA = \frac{1}{12} \sum_{I=1}^{12} (T_{ave}(I+1) - T_{ave}(I))$$

$$TA = \frac{1}{12}((75.65 + 73.29)/2 - (70 + 70)/2) + ((73.29 - 71.91)/2$$

$$(70 + 70)/2) + \ldots\ldots + ((70.02 - 70.01)/2 - (70 + 70)/2)$$

$$TA = .89 \ F$$

Step 5:

The heat balances yield the following results:

For the heat absorbed by the concrete wall:

$$QABS = q_{abs} \times TIME = CMASS \times CPCON \times TA \times TIME$$

where   TA = .89 F

           CPCON = .21 BTU/lb/F

           CMASS = AREA1 × DEPTH × DENSC

           AREA1 = 17100 ft^2

           DEPTH = 1 ft

           DENSC = 144 lb/ft^3

           TIME = 1 hr

$$QABS = 17100 \times 1 \times 144 \times .21 \times .89 = 4.6022 \times 10^5 \text{ BTU}$$

---

* 18 gauge sheet metal is used to enclose the basement of Building 1.

For the heat absorbed by the insulation on the building exterior:

$$\text{QINS} = \text{CPINS} \times \text{WINS*} \, (\text{TW2}_{\text{CURRENT}} - \text{TW2}_{\text{PREVIOUS}})/2 \times \text{TIME}$$
$$= 0.2 \times 0.1667* \times (70.01 - 70.0) \times 1$$
$$\text{QINS} = 0$$

For the heat loss to the soil:

$$\text{QHL1} = q_{hl}\text{xTIME} = \text{HO} \times \text{AREA1} \, (\text{TW2} - \text{TO}) \times \text{TIME}$$

where $\text{AREA1} = 17100 \text{ ft}^2$

            $\text{TIME} = 1 \text{ hr}$

            $\text{TW2} = 70.012 \text{ F}$

            $\text{TO} = 70 \text{ F}$

            $\text{HO} = 2.0552 \text{ BTU/ft}^2\text{/hr/F}$

$$\text{QHL1} = 2.0552 \text{x} 17100 \text{x} (70.012 - 70) = 422 \text{ BTU}$$

For the heat loss due to air infiltration into the building through cracks and other mall openings:

$$\text{QINF} = q_{inf}\text{xTIME} = \text{mCp}(\text{TW1} - \text{TO}) \times \text{TIME}$$

where $\text{Cp} = .24 \text{ BTU/lb/F}$

            $\text{TW1} = 75.649 \text{ F}$

            $\text{TO} = 70 \text{ F}$

            $\text{m} = \text{FINF(SCFM)} \text{x} 60 (\text{min/hr}) \text{x} 29 (\text{lb/lb-mole})/359(\text{SCF/lb-mole})$

            $\text{FINF} = 100 \text{ SCFM}$

            $\text{TIME} = 1 \text{ hr}$

$$\text{QINF} = 100 \text{x} 60 \text{x} 1 \text{x} 29/359 \text{x} .24 \text{x} (75.649 - 70) = 657 \text{ BTU}$$

For the heat loss to the air:

$$\text{QHL2} = q_{hl}\text{xTIME} = \text{U} \times \text{AREA2} \times (\text{TW1} - \text{TO}) \times \text{TIME}$$

where $\text{AREA2} = 10500 \text{ ft}^2$

---

* For 2 inches of insulation.

$$TW1 = 75.649 \text{ F}$$
$$TO = 70 \text{ F}$$
$$U = 1/(DINS/CINS + 1/4.6)$$
$$DINS = .3333 \text{ ft}$$
$$CINS = .05 \text{ BTU/ft/hr/F}$$
$$TIME = 1 \text{ hr}$$
$$QHL2 = 1/(.3333/.05 + 1/4.6) \times 10500 \times 1 \times (75.649 - 70) = 8617 \text{ BTU}$$

The total heat absorbed/lost is:

$$QTOTAL = QABS + QINS + QMET + QHL1 + QHL2 + QINF$$
$$QTOTAL = 460,220 + 0 + 23 + 422 + 8617 + 657 = 469,939 \text{ BTU}$$

Step 6:

Next, QABS and QTOTAL are compared and TOUT is incremented QABS and QTOTAL converge (i.e., repeat steps 1-5).

Example Output

An example computer output is given in Figure D-1.

```
BUILDING HEAT TRANSFER CALCULATIONS:TASK 3-BLDG. #3

FLOW=2000.00 SCFM.  TIN= 2000.00 DEGREES F.  DINS=  .1667 FEET.

ITERATION NUMBER  5  TIME= 5.00 HOURS

THE TEMPERATURE PROFILE AT TIME  5.00 IS:
 397.44 332.80 277.30 230.89 193.01
 162.81 139.23 121.20 107.73  97.94
  91.10  86.66  84.22
TOUT= 438.99  HI= 6.0888  TW1= 397.44 .TW2=  84.22  HO=  .9191
QABS=.283E+07 QHL1=.653E+05 QHL2=.105E+07 QINF=.381E+05 QINS=.525E+05
QMET=.666E+05 QGAS=.406E+07 QTOTAL=.407E+07

ITERATION NUMBER  10  TIME=10.00 HOURS

THE TEMPERATURE PROFILE AT TIME 10.00 IS:
 489.48 433.19 381.62 335.07 293.66
 257.41 226.20 199.83 178.05 160.56
 147.05 137.26 130.92
TOUT= 523.94  HI= 6.9654  TW1= 489.48  TW2= 130.92  HO=  .6499
QABS=.226E+07 QHL1=.198E+06 QHL2=.135E+07 QINF=.488E+05 QINS=.262E+05
QMET=.333E+05 QGAS=.386E+07 QTOTAL=.386E+07

ITERATION NUMBER  15  TIME=15.00 HOURS

THE TEMPERATURE PROFILE AT TIME 15.00 IS:
 545.42 495.31 448.44 405.08 365.41
 329.60 297.72 269.79 245.80 225.68
 209.35 196.69 187.56
TOUT= 576.22  HI= 7.5299  TW1= 545.42  TW2= 187.56  HO=  .5307
QABS=.185E+07 QHL1=.312E+06 QHL2=.155E+07 QINF=.533E+05 QINS=.177E+05
QMET=.225E+05 QGAS=.373E+07 QTOTAL=.373E+07

ITERATION NUMBER  20  TIME=20.00 HOURS

THE TEMPERATURE PROFILE AT TIME 20.00 IS:
 586.64 541.37 498.51 458.60 421.52
 387.33 356.76 329.27 305.12 284.32
 266.95 252.68 241.72
TOUT= 615.02  HI= 7.9654  TW1= 586.64  TW2= 241.72  HO=  .4596
QABS=.155E+07 QHL1=.395E+06 QHL2=.166E+07 QINF=.601E+05 QINS=.139E+05
QMET=.176E+05 QGAS=.364E+07 QTOTAL=.364E+07

ITERATION NUMBER  25  TIME=25.00 HOURS

THE TEMPERATURE PROFILE AT TIME 25.00 IS:
 619.58 578.32 539.10 502.14 467.60
 435.62 406.34 379.94 356.18 335.40
 317.52 302.51 290.34
TOUT= 646.15  HI= 8.3238  TW1= 619.58  TW2= 290.34  HO=  .4110
QABS=.133E+07 QHL1=.453E+06 QHL2=.177E+07 QINF=.639E+05 QINS=.112E+05
QMET=.143E+05 QGAS=.356E+07 QTOTAL=.356E+07

ITERATION NUMBER  27  TIME=27.00 HOURS

THE TEMPERATURE PROFILE AT TIME 27.00 IS:
 631.10 591.26 553.32 517.47 483.88
 452.69 424.01 397.94 374.55 353.97
 335.92 320.69 308.16
TOUT= 657.07  HI= 8.4529  TW1= 631.10  TW2= 308.16  HO=  .3955
QABS=.124E+07 QHL1=.471E+06 QHL2=.180E+07 QINF=.653E+05 QINS=.103E+05
QMET=.130E+05 QGAS=.355E+07 QTOTAL=.354E+07
```

FIGURE D-1. EXAMPLE COMPUTER OUTPUT FOR THE
HOT GAS DECONTAMINATION HEAT
BALANCE CALCULATIONS

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