

# REPORT DOCUMENTATION PAGE

Form Approved  
OMB NO. 0704-0188

Public Reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave Blank)		2. REPORT DATE <b>01/11/02</b>	3. REPORT TYPE AND DATES COVERED <b>Final Report 02-12-01-12-11-01</b>	
4. TITLE AND SUBTITLE <b>Decontamination Using a One Atmosphere Uniform Glow Discharge Cold Plasma</b>		5. FUNDING NUMBERS <b>DAAD19-01-C-0033</b> <del>DAAD19-01-C-0035</del> <b>P00001</b>		
6. AUTHOR(S) <b>Kimberly Kelly - Wintenberg, Ph.D.</b>		8. PERFORMING ORGANIZATION REPORT NUMBER <b>42002.1-CH</b> <b>A00-020-AGT-1999 581</b>		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) <b>Atmospheric Glow Technologies, 2342 Stock Creek Blvd., Rockford, TN 37853-3014</b>		9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) <b>U. S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211</b>		
11. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.				
12 a. DISTRIBUTION / AVAILABILITY STATEMENT <b>Approved for public release; distribution unlimited.</b>		12 b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) <b>Atmospheric Glow Technologies is developing an innovative mobile atmospheric plasma decon system based upon the patented one atmosphere uniform glow discharge plasma (OAUGDP) technology. This advanced system will be a deployable military protection system that will decontaminate highly toxic chemicals and microorganisms on sensitive equipment, personnel protective equipment, and aircraft, vehicle, and shelter interiors. Phase I efforts demonstrated feasibility of the system in the neutralization of chemical warfare simulants (99.3% reduction). Phase II efforts will extend studies to biological warfare agents including spores, building upon the proven efficacy of the OAUGDP against a wide range of microorganisms. Future work will involve optimizing the system for recirculation of the airflow and air cooling. Studies are currently underway to determine the concentration of atmospheric plasma produced active species as well as the extent of chemical and biological degradation achieved. The system will also be tested for materials and device compatibility as well as neutralization studies of actual chemical and biological warfare agents.</b>				
14. SUBJECT TERMS <b>atmospheric glow plasma cold plasma decontamination</b>		15. NUMBER OF PAGES <b>28</b>		
17. SECURITY CLASSIFICATION OR REPORT <b>UNCLASSIFIED</b>		18. SECURITY CLASSIFICATION ON THIS PAGE <b>UNCLASSIFIED</b>	19. SECURITY CLASSIFICATION OF ABSTRACT <b>UNCLASSIFIED</b>	20. LIMITATION OF ABSTRACT <b>UL</b>
16. PRICE CODE				

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)  
Prescribed by ANSI Std. Z39-18  
298-102

20021017 049

**Final Report  
Decontamination Using a One Atmosphere Uniform Glow  
Discharge Cold Plasma  
A002-0497**

**Contractor:  
Atmospheric Glow Technologies  
2342 Stock Creek Blvd.  
Rockford, TN 37853-3044**

**Prepared by:  
Kimberly Kelly-Wintenberg, Ph.D.**

**January 11, 2002**

## Table of Contents

Statement of Work.....	3
Summary of Results	3
Summary of Task 1	7
Summary of Task 2	8
Summary of Task 3	10
Summary of Task 4	16
Summary of Task 5	18
Summary of Option Phase Studies	18
Listing of all technical reports supported under this contract	25
Listing of all scientific personnel involved in the project	25
Listing of inventions	25

### Statement of Problem

The specific aim of this Phase I contract was to assess the feasibility of the One Atmosphere Uniform Glow Discharge Plasma (OAUGDP) process as an innovative deployable military protection system that decontaminates highly toxic chemicals and microorganisms on military materiel. Each of the tasks described in the Phase I proposal have been addressed and data collected using a newly designed atmospheric plasma reactor and power supply. The Phase I technical objectives are listed below and **Table 1** lists the personnel involved.

### Summary of Results

#### Phase 1 Technical Objectives

- ◆ *Acquiring and characterizing simulants.*
- ◆ *Assessing plasma efficacy against selected simulants on non-porous surfaces*
- ◆ *Assessing plasma efficacy against selected simulants on porous surfaces*
- ◆ *Preparing a comprehensive plan.*
- ◆ *Designing a field portable OAUGDP*
- ◆ *Delivering a final report*

Table 1. Personnel involved in Phase I study

Name	Affiliation	Role on project
Kimberly Kelly-Wintenberg	Atmospheric Glow Technologies	Principal Investigator
Suzanne South	Atmospheric Glow Technologies	Director of Biological/Chemical Research
Daniel Sherman	Atmospheric Glow Technologies	Director of Plasma Technology
Michael Wimpee	Microbial Insights	Subcontractor
J. Reece Roth	University of Tennessee	Consultant

---

---

### Phase I engineering of atmospheric plasma reactor prototypes

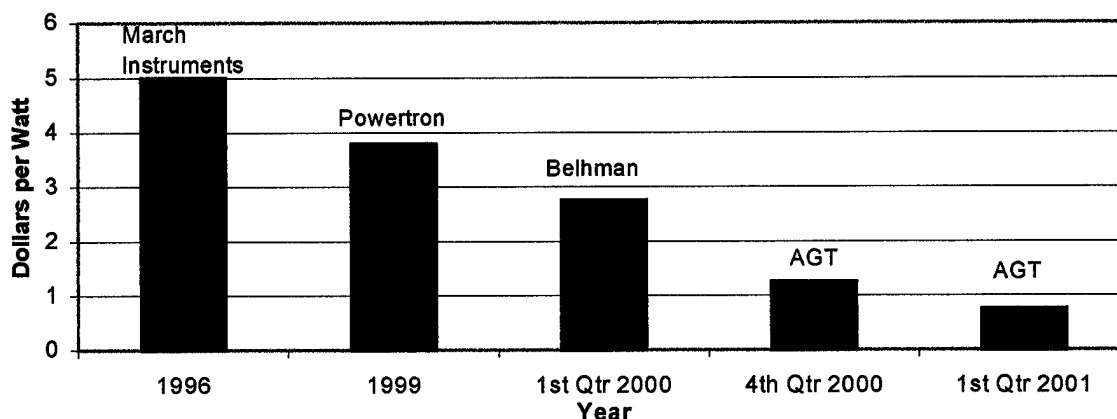
---

---

Although engineering of prototypes was not addressed as an individual task in the Phase I proposal, this section details this work. The time between submission of this research proposal and the initiation of the work plan, the power supply used to drive the atmospheric cold plasma was redesigned and fabricated using off-the-shelf components. This has led to

increased power efficiency and significantly reduced cost of the power supplies. The prototype reactors used in this study were designed by Atmospheric Glow Technologies' engineers and fabricated at our facility or locally. Upon completion of the first quarter of 2000, the direct manufacturing cost of an atmospheric plasma reactor and power supply was \$15,000 such that it was cost prohibitive. As shown in Table 2, Atmospheric Glow Technologies has significantly reduced the reactor and power supply cost thereby making it a very competitive technology with direct manufacturing costs of the power supply and plasma reactor estimated to be less than \$8,000.

**Table 2. Power Supply Cost (Direct Manufacturing Costs)**



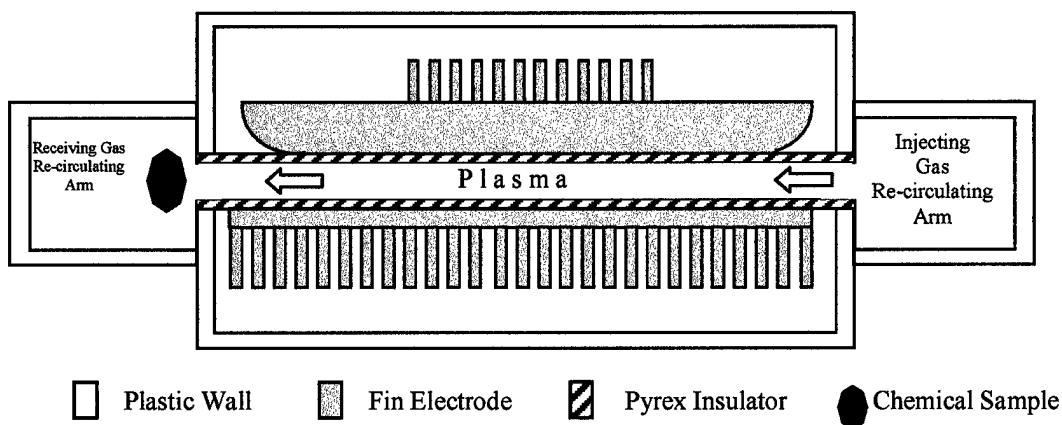
To ensure proof of concept of chemical neutralization based upon the OAUGDP technology was demonstrated we chose to concurrently develop and use two different plasma reactors. The first reactor used was the **atmospheric plasma chamber reactor**. We took advantage of its design qualities to demonstrate the effects of remote plasma exposure of samples in a recirculating mode with an airflow face velocity of 750 fpm (8mph). The second prototype employed was the **Atmospheric Plasma Decon 210 unit (APD-210)** a portable atmospheric plasma blower using single pass air with an airflow face velocity of 3,000fpm (30mph). The APD-210 design was proposed in the Phase I proposal.

**Based upon the experimental data discussed below, the APD-210 prototype will be preferentially used as the basis for succeeding generations of decontamination devices.**

**Atmospheric Plasma Chamber.** Samples were typically exposed in the arm of the atmospheric plasma chamber reactor. A diagram of the reactor is shown in Figure 1.

This reactor is based upon the parallel plate electrode configuration that has been studied for ten years. The plasma exposure reactor is comprised of two gapped insulated parallel plate electrodes, a re-circulating air supply system, an air electrode cooling system, and an enclosure to contain any leaks. For these experiments plasma is generated in the gap between the two 15 x 15 cm pieces of 2 mm thick Pyrex which insulated the parallel plate electrodes. The typical operating conditions of the atm plasma were 11kV and 9 kHz, however for certain experiments other operating parameters were employed and are described in the report. The chemical samples were placed 6cm downstream of the plasma in the recirculating arm as shown in Figure 1.

**Figure 1. The Atmospheric Plasma Chamber.**

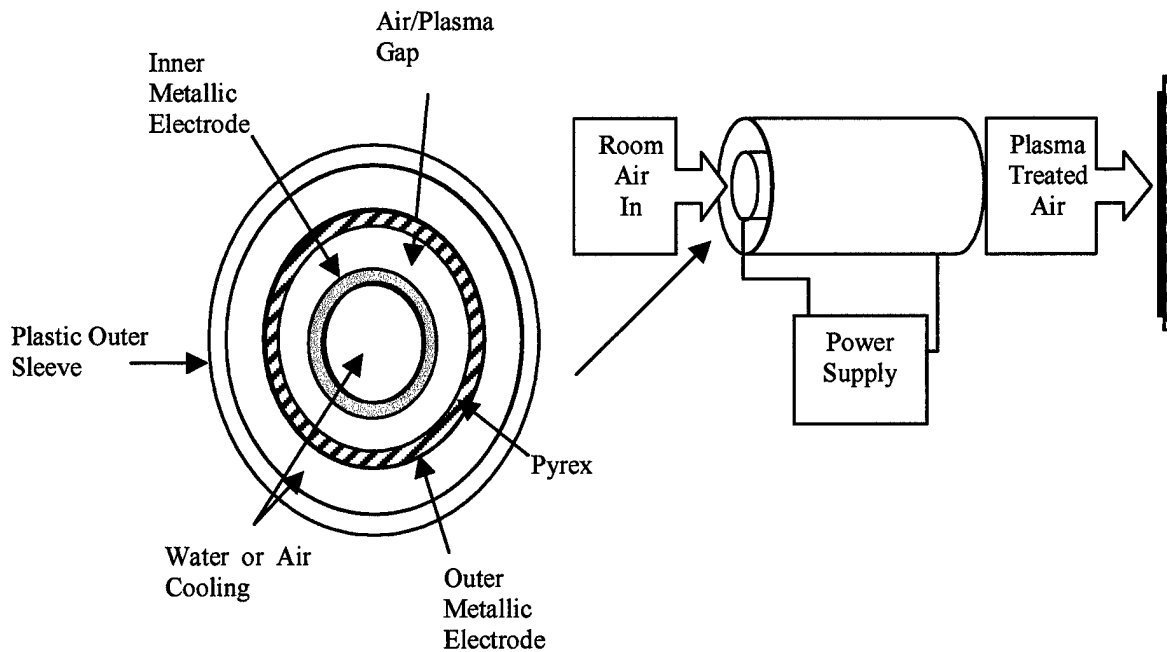


The Atmospheric Plasma Decon 210 Unit (APD-210). As shown in Figure 2, this prototype was developed specifically for this Phase I contract work. During the proof of concept experiments described below, the APD-210 operated in a single pass air circulation mode with 80% of the surface area covered with plasma with operating conditions of 8kV and 8.5kHz. The APD-210 requires recirculated water for cooling the plasma generation area and subsequently the exhaust flow. The device utilized 0.25 in pipes with a water flow rate of 0.735 L/Min. The water was recirculated at temperatures of 60-60°F. This parameter adaptation on the APD-210 allowed samples to be plasma exposed for at least 12 min and did not exceed 38°C. Table 3 describes the physical parameters of the two reactors used in these studies.

Both of these first generation devices are capable of the required chemical decontamination and future devices will be designed from their successes.

Below is a brief description of each aim and the work performed in Phase I and the subsequent bridging period.

- Task 1: Simulant acquisition and characterization**
- Task 2: Plasma reaction with selected simulants on non-porous surfaces**
- Task 3: Plasma reaction with selected simulants on porous surfaces**
- Task 4. Preparation of a comprehensive plan.**
- Task 5. Design of a field portable OAUGDP.**



**Figure 2. Atmospheric Plasma Decon 210 (APD-210) unit**

**Table 3. Physical description of the atmospheric plasma reactors used in Phase I studies.**

Plasma Devices	Physical Parameters	
	Weight (lbs)	Size (LxWxH) in
<b>APD-210</b>		
Amplifier	14	12 x 9 x 9
HV Transformer	38	13 x 8 x 6
HV Transformer	38	13 x 8 x 6
Ring Compressor	32	11 x 10 x 11
Hand held Blower	16	58 x 2.25 D
<b>Remote Chamber</b>		
Amplifier	5	11 x 6 x 6
HV Transformer	4	5 x 5 x 4.5
Reactor	15	11.5 x 11 x 10

---



---

**Task 1: Simulant acquisition and characterization**

---



---

Literature searches for chemical simulants revealed that there is no single chemical used as a simulant standard. A number of chemicals were suggested from the searches, and the three chosen for this study were dimethyl methylphosphonate (DMMP) and methyl salicylate as simulants for GB, and butyl sulfide as a simulant for distilled mustard. Two organic chemists were called upon to verify our predictions of the chemical degradative products produced upon plasma exposure to the simulants. This safety precaution was done to ensure there was no release of toxic compounds during operation. The MSDS sheets for these materials are kept in each laboratory and in the main Atmospheric Glow Technologies' office. In addition, a list of chemicals are kept in a nearby facility in the event of an emergency assess to the MSDS sheets could be done remotely using the Internet. The predicted products are described below along with the chemical's properties.

**Dimethyl methylphosphonate** is widely recognized as the simulant of choice for the volatile G series of chemical nerve agents. It is a liquid at room temperature with a boiling point of 181°C and a density of 1.145. Exposure to plasma based oxidative species is expected to result in degradation of the DMMP through attack at the P=O bond.

**Butyl sulfide** is a liquid at room temperature with a boiling point of 118°C and a density of 0.838. This chemical is similar in structure to mustard gas. The sulfur atom in butyl sulfide is expected to be chemically equivalent to the sulfur atom in mustard gas and will undergo the same type of chemistry upon interaction with plasma. This is predicted to be an initial oxidative addition to the sulfur atom to give a sulfone and eventual oxidative degradation to a sulfate.

**Methyl salicylate** is commonly used by DoD as a CB agent simulant due to its non-toxic nature, its easily recognizable odor, and its ease of detection by GC/MS.

---

---

## **Task 2: Plasma reaction with selected simulants on non-porous surfaces**

---

---

This task was performed to examine the effectiveness of plasma on the destruction of chemical surrogates coated onto stainless steel coupons. This task simulated contamination of equipment such as sidearms, rifles, radios, and aircraft.

### **Methodology**

The method of preparation of a samples for testing was as follows. A 6ul sample of standard Dimethyl methylphosphonate,  $\text{CH}_3\text{P}(\text{O})(\text{OCH}_3)_2$ , CAS # 756-79-6, Fluka Chemical, Switzerland (DMMP) diluted with chloroform to a final concentration of 61.11 nmol/ul was spotted in the center of a sterile 25 x 55 mm stainless steel coupon and immediately placed in a sterile petri plate (chemical side up) until plasma exposure. These samples were exposed either directly in the plasma or remotely in the recirculating arm (**See Figure 1**) with the following operating parameters: Operating gas: room air, 11kV, 9kHz, recirculation speed 700fmp, 31.7°C.

The treated surrogate was removed from the coupon with chloroform and analyzed via Hewlett Packard 6890 Series Gas Chromatograph coupled to a Hewlett Packard 5973 Mass Selective Detector. Electron ionization gas chromatography-mass spectrometry (GC-MS) was carried out on a HP-5 capillary column (30m X 0.25mm X 0.1 $\mu\text{m}$  film thickness) programmed at 60°C using helium at a flow rate of 0.5 ml/min for the carrier gas. Area responses of prolonged exposure to atmospheric plasma conditions were determined at different time intervals. All fragmentation patterns of surrogate standards were verified via NBS 75K, National Bureau of Standards and NIST 1992.

## Results

Due to the accelerated submission of the Phase II proposed work only preliminary experiments for this task have been performed to date. Initial experiments indicated a loss of the compound due to volatility in the non-exposed samples. To circumvent this problem, the DMMP was mixed with methanol to a final concentration of 61.11 nmol/ul and inoculated onto the surface of a stainless steel coupon. These GC tracings showed DMMP being eluted at its standard elution time. (Figure 3) This data would suggest the volatile simulants must be mixed with thickening agents to increase their persistency.

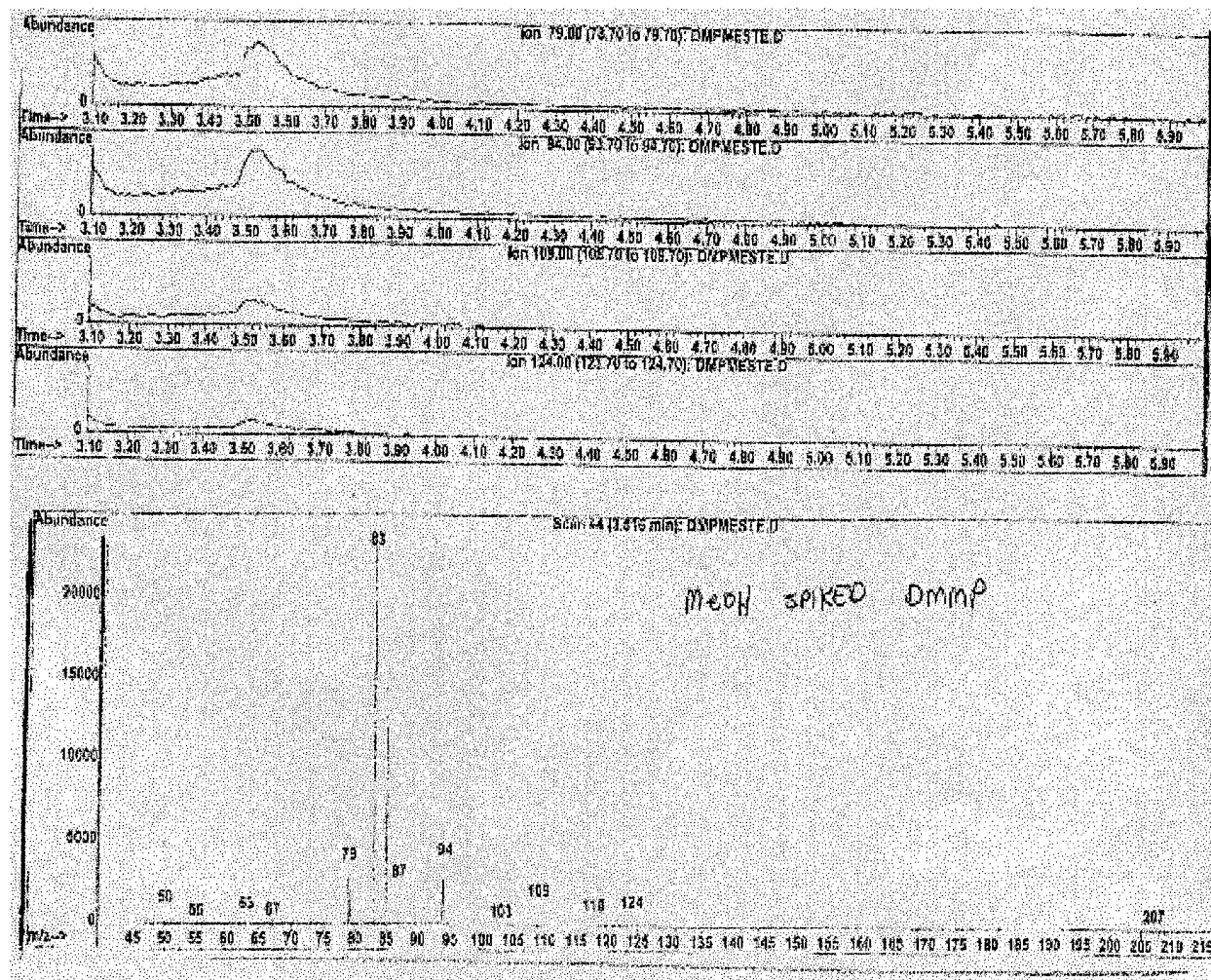
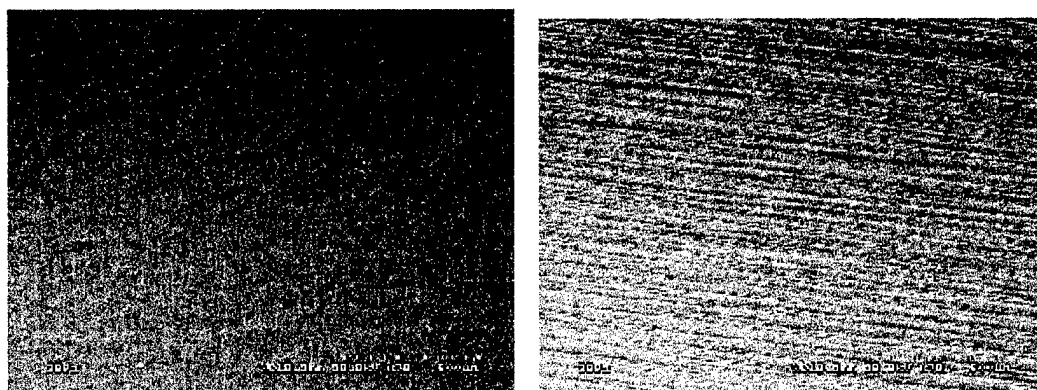


Figure 3. GC/MS tracings of DMMP diluted with methanol. The tracings represent the elution of DMMP without plasma exposure.

To compliment the work described above we chose to treat the stainless steel coupons in atmospheric plasma and look at the effects using variable pressure scanning electron microscopy (VP SEM). Shown below in **FIGURE 4** is VP-SEM digital images of two stainless steel coupons both prior to (A.), and after, two-minute exposure to the plasma (B.). In both cases the coupons were autoclaved prior to examination. In the untreated sample there are monolayers of attached hydrocarbons, i.e. oils, still attached to the surface. Two minutes of plasma exposure is sufficient time to significantly etch, i.e. remove these layers and enhance the visual appearance of the surface topography. The removal of these monolayers leaves the stainless steel, zinc coated steel, surface exposed. The etching process on the stainless creates the unlikely possibility of the formation of residues on the treated surface.



**A.**

**B.**

**Figure 4.** **A.** untreated coupon, depicts a clean metallic surface. The lineations in the surface topography are created during the rolling process used to create the stainless steel sheet from which the coupons were cut. **B.** treated coupon, depicts the lineation of the fabrication process as visually more pronounced

---

---

### **Task 3: Plasma reaction with selected simulants on porous surfaces**

---

---

To date the majority of contract time for experimentation has been spent on this task.

#### **Methodology**

Surrogate standards such as Methyl Salicylate,  $C_8H_8O_3$ , CAS # 119-36-8, Sigma Chemical Company, St. Louis, MO and Dimethyl methylphosphonate,  $CH_3P(O)(OCH_3)_2$ , CAS # 756-79-6, Fluka Chemical, Switzerland were used undiluted or diluted with chloroform to a final

concentration of 61.11 nmol/ul for DMMP and 77.05 nmol/ul for methyl salicylate. Glass Microfibre filters 47mm (Whatman Cat No 1821-047) were baked overnight then inoculated with three 2ul drops of simulant in a circular pattern in the center of the filter paper. The simulants absorbed quickly and were stored in sterile petri plates prior to plasma exposure.

As described in **Task 1** the samples were either exposed in the remote chamber reactor or the APD-210 unit for durations between 0 to 12 min. For each experimental run, a control of airflow only was performed to examine the effects of air at the face velocity running over a sample.

Chemical simulants were analyzed via Hewlett Packard 6890 Series Gas Chromatograph coupled to a Hewlett Packard 5973 Mass Selective Detector as described above. Electron ionization gas chromatography-mass spectrometry (GC-MS) was carried out on a HP-5 capillary column (30m X 0.25mm X 0.1µm film thickness) programmed at 60°C using helium at a flow rate of 0.5 ml/min for the carrier gas. Area responses of prolonged exposure to atmospheric plasma conditions were determined at different time intervals. All fragmentation patterns of surrogate standards were verified via NBS 75K, National Bureau of Standards and NIST 1992.

## **Results**

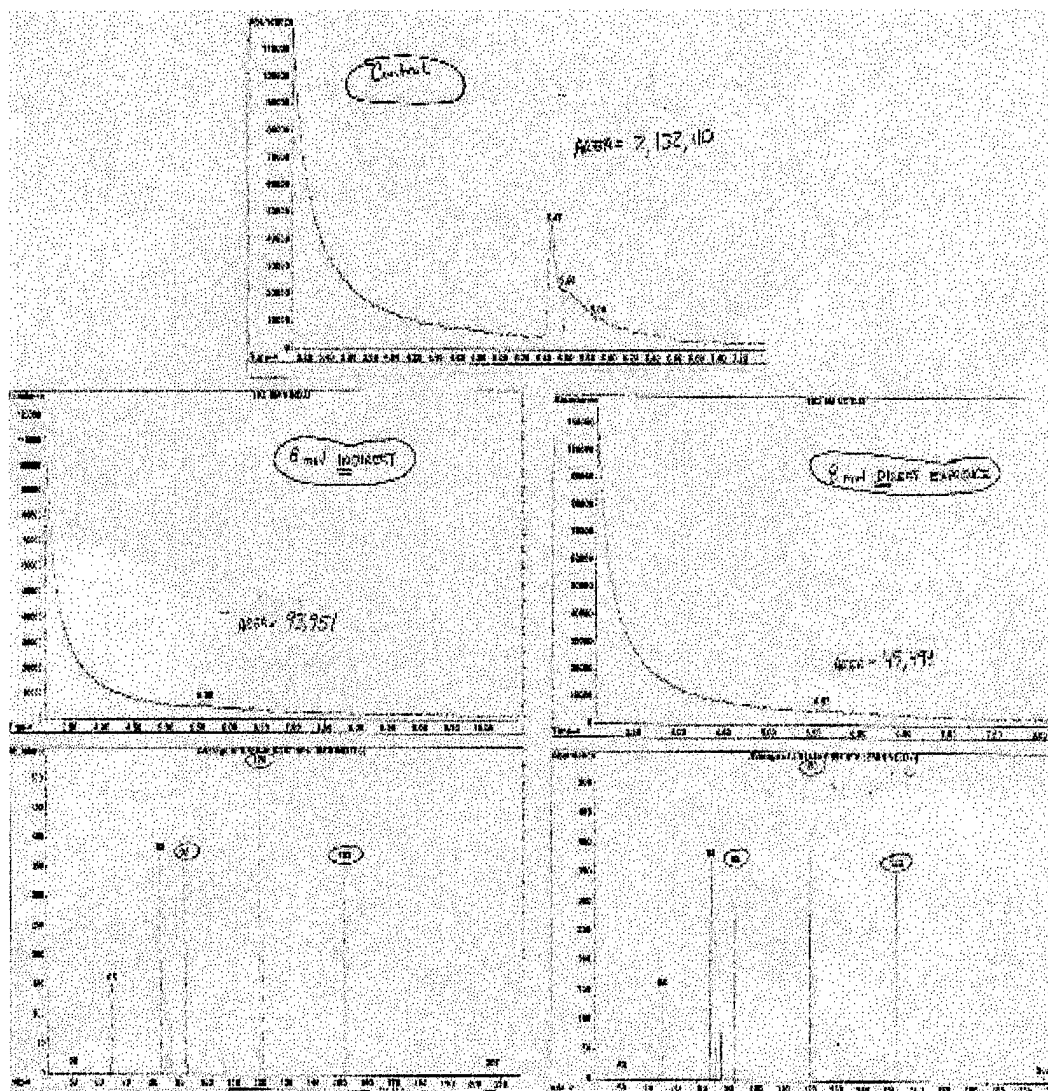
### **Methyl Salicylate:**

**Direct vs. Indirect Plasma Exposure.** Initially we wanted to compare the efficacy of exposing methyl salicylate (prepared as described above) in direct plasma exposure and indirect plasma exposure using the reactor shown in **Figure 1**. In the past, direct plasma exposure has been the most effective means of inactivation of biological or chemical agents; however, its mode of operation is not feasible for military decon since items larger than the 4 to 6 mm gap distance. After exposure the samples were analyzed by GC/MS and the results are seen in **Figure 5 (see next page)**. The remote chamber generated a 93.68% reduction in activity after 8 min of exposure while 8 min of direct exposure showed a 97.68% reduction.

The results from this experiment are summarized in **TABLE 4**. **These results demonstrated efficacy of exposing the REACTIVE OXYGEN SPECIES (ROS) generated by the plasma to a remote location and attaining chemical neutralization.**

**Table 4. Percent reduction of methyl salicylate after atmospheric cold plasma exposure.**

Direct Plasma Exposure		Indirect Plasma Exposure	
Time (min)	Percent Reduction	Time (min)	Percent Reduction
0	0	0	0
2	91.3	2	0
8	97.68	8	93.68
8 min Air Flow Only	0	8 min Air Flow Only	0



**Figure 5. GC/MS tracings of methyl salicylate following either direct or indirect plasma exposure for eight minutes. Control in no plasma exposure. Air flow only tracings showed no change from the control.**

Based upon the results described in Table 4, we choose to compare the efficacy of the APD-210 are the airflow patterns: single pass vs. recirculated air, respectively. Samples of methyl salicylate were prepared on the glass filter paper as described above expect the methyl salicylate was used at a 100% concentration and exposed in either the remote chamber (Figure 1) or the APD210 (Figure 2) for durations of 0 to 8 min. The operating parameters are described in TABLE 5.

**Table 5. Operating conditions of the plasma reactors.**

Operating parameters	Remote chamber	APD-210
Voltage	11.3 kV	8kV
Frequency	9kHz	8.6kHz
Working gas	Room air	Room air
Airflow face velocity	12.5 ft/sec recirculated	50 ft/sec, single pass
Temperature	43°C	48.9°C
Distance samples exposed from plasma source	2.36 in	3.25 in

Each experimental run always included an “airflow only” control corresponding to the longest exposure time to ensure the effects seen were due to ROS generated by the plasma and not volatilization by airflow alone. Samples were prepared and analyzed by GC/MS as described above and described below in Table 6.

**TABLE 6. GC/MS analyses of plasma exposed methyl salicylate using a recirculated or single pass airflow.**

Time(min)	Remote chamber		APD-210	
	Area under curve	Percent Reduction	Area under curve	Percent Reduction
0	7,829,494,6900	0	9,239,846,066	0
2	8,924,522,872	0	2	<10
6	3,859,708,310	50.7	ND	--
8	ND	--	7,330,965	99.93
10	64,166,480	91.8	ND	--
Air Flow Only	8,117,179,174	0	9,378,751,120	0

From the data collected and presented in **TABLE 6** the APD-210 has demonstrated its ability to neutralize methyl salicylate (99.93% reduction) and its feasibility for development into a robust portable decon unit for the military.

**Dimethyl methylphosphonate-DMMP**

**Remote chamber vs. APD-210 unit.** Experiments similar to the ones described for methyl salicylate was performed for DMMP. As previously described, the simulant (DMMP 61.11 nmol/ul) was inoculated onto glass microfibre filters (3-2ul drops) and exposed in either the arm of the recirculating chamber or the APD-210. The operating parameters are described in **Table 5**. Following plasma exposure the samples were analyzed by GC/MS and the results are reported as tracings in **Figure 6** and described in **Table 7** below.

**Table 7. GC/MS analyses of plasma exposed methyl salicylate using a recirculated or single pass airflow.**

Time(min)	Remote chamber		APD-210	
	Area under curve	Percent Reduction	Area under curve	Percent Reduction
0	13,321,361	0	13,321,361	0
8	2854305	78.58	890,816	93.31
12	ND		21,233	99.84
Air flow only	10,909,839	18.1	12,675,925	4.84

Further analyses of the ion ratio data for DMMP is shown in **Table 8**. This data shows no detectable levels of DMMP after 12 min of exposure in the APD-210 unit.

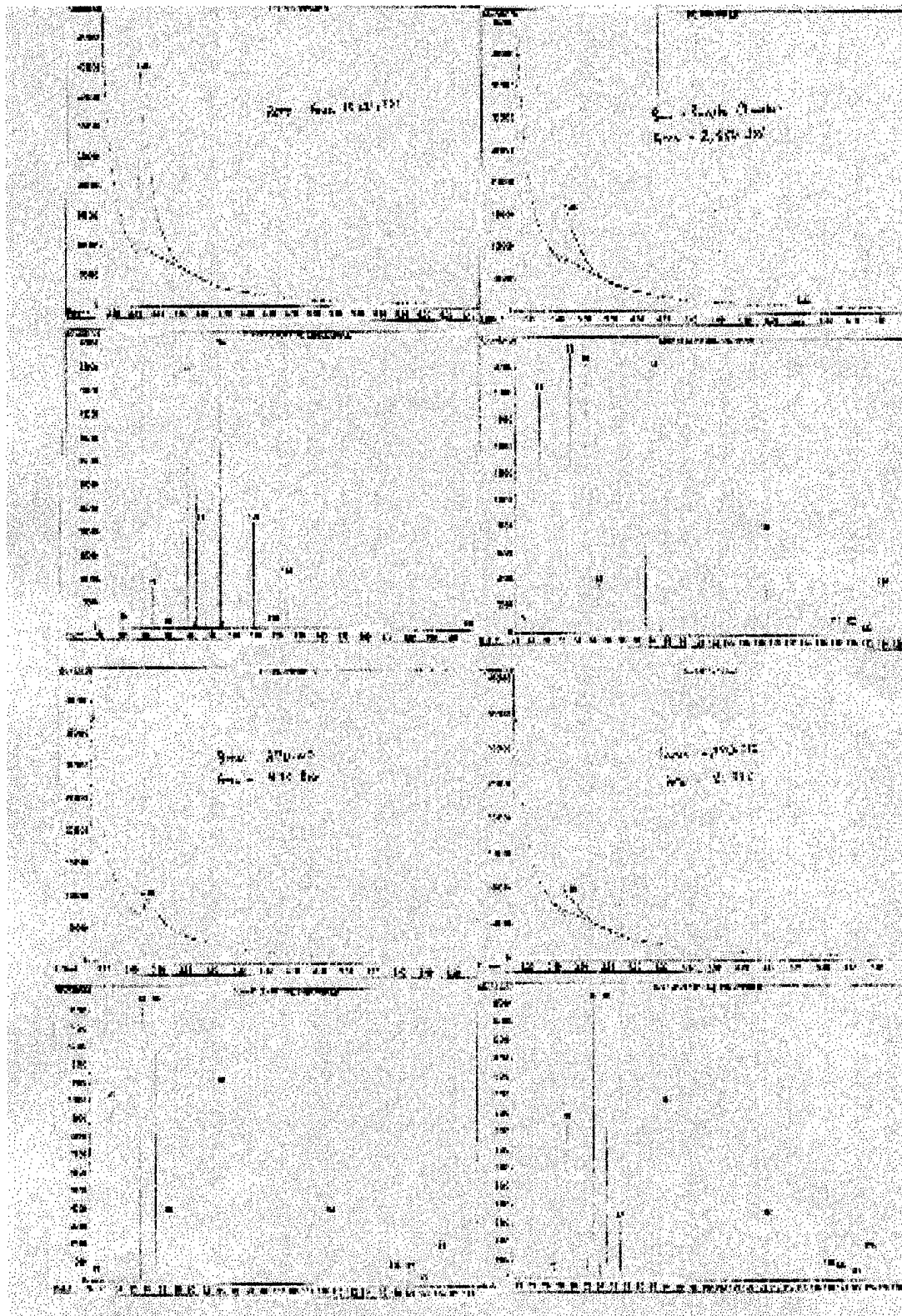


Figure 6. GC/MS tracings of DMMP exposed the APD210

**Table 8. Ion Ratio Data for DMMP**

<b>Sample</b>	<b>Ions</b>	<b>Response</b>	<b>Ion Ratio</b>	<b>+/- 20 Range</b>
<b>Zero control</b>	79	8,446,864	0.916	(0.733-1.1)
	94	9,226,007	1.000	NC
	109	3,066,130	0.322	(0.266-0.398)
	124	1,644,173	0.178	(0.142-0.214)
<b>Air Flow in APD-210</b>	79	7,298,349	0.907	(0.733-1.1)
	94	8,043,186	1	NC
	109	2,599,126	0.323	(0.266-0.398)
	124	1,143,826	0.142	(0.142-0.214)
<b>Air Flow in Remote Chamber Reactor</b>	79	3,749,640	0.856	(0.733-1.1)
	94	4,378,692	1	NC
	109	1,630,442	0.372	(0.266-0.398)
	124	469,258	0.107	(0.142-0.214)
<b>8 min exposure in APD-210</b>	79	852,484,	0.823	(0.733-1.1)
	94	1,035,588	1	NC
	109	336,726	0.325	(0.266-0.398)
	124	129,254	0.125	(0.142-0.214)
<b>12 min exposure in APD-210</b>	79	968,031	1.03	(0.733-1.1)
	94	941,024	1	NC
	109	73,914	0.078	(0.266-0.398)
	124	60,847	0.065	(0.142-0.214)

As seen with methyl salicylate, the APD-210 reduces over 93% of the chemical simulant within 8 min and no detectable levels remain after 12 min of exposure. This finding demonstrates the feasibility of using the APD-210 unit as a decon device and that it merits incorporation of design changes and improvements to reduce exposure times in a Phase II effort.

---



---

**Task 4. Preparation of a comprehensive plan**

---



---

A number of different parameters were examined during the course of this study to optimize the atmospheric plasma reactor and the power supply. At the time of writing the Phase I proposal, the direct manufacturing cost of an atmospheric plasma reactor and power supply was \$15,000 which made it cost prohibitive for commercialization. As shown in **Table 2**, Atmospheric Glow Technologies has significantly reduced the reactor and power supply cost thereby making it a very competitive technology with direct manufacturing costs of the power supply and reactor estimated to be less than \$ 8,000.

**Table 9** describes the weight and required electrical power of the reactor and cart equipment. In prototype testing a Veriac was used to adjust the input voltage to the power supply. **Table 10** describes specifications of the reactors used in Phase 1 studies and addresses system optimization of the prototype for potential military use.

**Table 9. Required power and weight of the prototypes.**

	APD-210	Remote Chamber
Weight of Generation Equipment (lbs)	122	9
Reactor Weight (lbs)	16	15
Input Voltage (volts)	208	120
Required Input Power (kVA)	2	0.22
Input Power Utilized (Watts)	850	100

**TABLE 10. Specs of the Remote Plasma Reactors**

Parameter	APD-210	Remote Chamber
Number of dielectrics	1	2
Dielectric thickness (mm)	2	2
Plasma gap (mm)	3	2.5
Electrode gap distance (mm)	5	6.5
Operating Voltage (kV)	8	11
Electric field strength (V/m)	1.6	1.7
Operating frequency (kHz)	8.5	9
Effective Source plasma area (cm <sup>2</sup> )	998	53.6
Effective source plasma volume (cm <sup>3</sup> )	325	13.4
Surface/volume ratio	3.1	4
Input power consumed	850	100
Input power density (watts/cm <sup>3</sup> )	2.6	7.5
Input power arial density (Watts/cm <sup>2</sup> )	0.9	1.9
Handheld weight (lbs)	16	N/A

Three parameters electric field strength, input power and the operating frequency discussed in **Table 10** dominate the OAUGDP chemistry. The electric field strength, determined by the applied voltage and the distance between the metallic electrodes, indicates chembio agent

fragmentation, and production rates of the active species. As the operating voltage is increased there will be a requirement for more power to the APD devices. As the operating frequency increases so does the filamentation (the presence miniature electrical arcs) throughout the plasma volume. This filamentation increases the etching rate of the dielectrics, and can result in sudden failure of the dielectric and subsequently the device. The other parameters in the table above are listed for reference.

---

---

**Task 5. Design of a field portable OAUGDP.**

---

---

Chemical neutralization using the atmospheric cold plasma was demonstrated with the APD-210. While this prototype has performed very well in initial studies, Atmospheric Glow Technologies proposes to optimize the device and it will be named the Atmospheric Plasma Decon-210A unit (**APD-210A**). It is our belief that this modification will allow more complete degradation of agents and increased operator safety. Atmospheric Glow Technologies can deliver this cost-effective prototype at the end of the Phase II contract. We are confident that the APD-210A can meet the military's need for inactivation of both microorganisms and chemicals but will not require water-cooling. The schematics for the APD-210A are shown in **Figure 7. Figure 7 depicts the APD-210 optimized unit with a weight of approximately 16 lbs. Figure 7b and Figure 7c are cutouts and detail key system requirements at the intake end and the exhaust end, respectively.**

In the Phase II work plan a new reactor, the APD-310 is being designed that encompasses all the advantages of the APD-210A and be air-cooled. Its design will be developed during the Phase II portion of the project.

**Phase I Option Activities**

The table shown below briefly summarizes the activities performed during the interim spanned by the Phase I Option. The performed activities are detailed in the subsequent pages.

**Table 11. Tasks performed during the Phase I Option period**

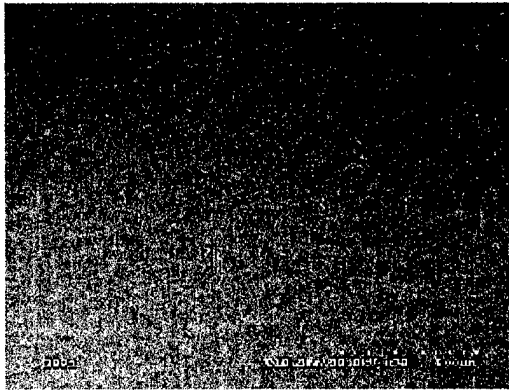
Description	Task Performed
Examining surface alterations of significant military materiel	<ul style="list-style-type: none"> <li>• SEM of stainless steel using direct exposure to the plasma.</li> <li>• Tearing strength testing of cloth substrates.</li> <li>• Color alteration test of cloth samples</li> </ul>
Characterization of chemical reactions	<ul style="list-style-type: none"> <li>• Have had discussions with ORNL scientist on potential candidates.</li> <li>• Have measured exhaust rates of ozone, and NO<sub>2</sub>, and NO generated by the prototype.</li> <li>• Are nearing completion of arrangements to sample airflow for gaseous breakdown by-products of the CW surrogates.</li> </ul>
Prototype development	<ul style="list-style-type: none"> <li>• Three revisions in the prototype design and power supply have been performed. The following improvements have been made:</li> <li>• Prototype size reduced</li> <li>• Enhanced the prototype cooling assembly</li> <li>• Modifications to the power supply and impedance matching system</li> <li>• Assessed the prototype effectiveness against biological agents.</li> </ul>

**Examination of Surface Changes**

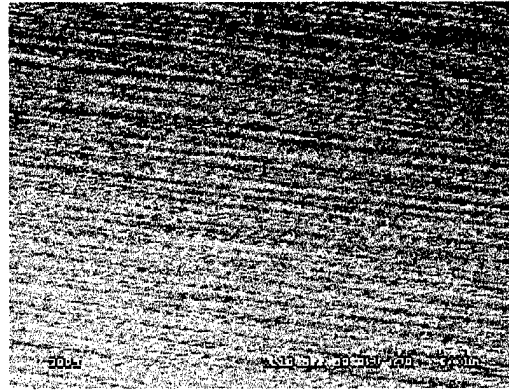
To examine changes on the surface of metals and soft plastics by atm plasma treatment, variable pressure scanning electron microscopy (VP-SEM) was performed by The University of Tennessee Electron Microscopy Facilities. VP-SEM micrographs shown in **Figure 8** demonstrate the changes in surfaces after atm plasma exposure. In order to assess the changes done to military grade fabric by the exhaust from the plasma, data for both tearing strength test and color alteration test is shown below. Follow-up tests will evaluate changes done to the surface by the exhaust plasma to the stainless steel coupons, soft plastic, and military fabric.

**Figure 8. VP-SEM data for Stainless Steel Coupons and PP Film**

Shown below are two vapor pressure scanning electron microscopy digital images of two stainless steel coupons both prior to, and after, two-minute exposure to the plasma. In both cases the coupons were autoclaved prior to examination.

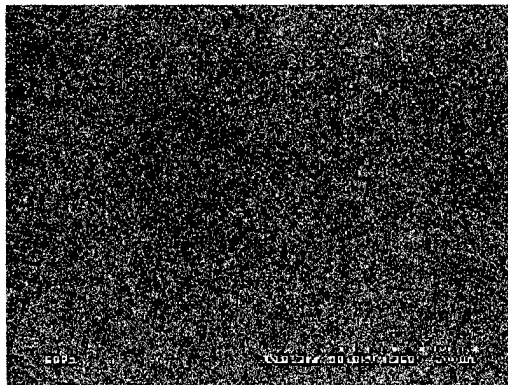


**Figure 8A.**



**Figure 8B.**

**Figure 8A**, the untreated coupon, depicts a clean metallic surface. The lineations in the surface topology are created during the rolling process used to create the stainless steel sheet from which the coupons were cut. **Figure 8B**, the treated coupon, depicts the lineation of the fabrication process as visually more pronounced. Even after autoclaving, there are monolayers of attached hydrocarbons, i.e. oils, still attached to the surface. Two minutes of plasma exposure is sufficient time to significantly etch, i.e. remove these layers and enhance the visual appearance of the surface topology. The removal of these monolayers leaves the stainless steel, zinc coated steel, surface exposed.



**Figure 8 C.**



**Figure 8 D.**

The following three images depict vapor pressure scanning electron microscopy digital images of a low-density polypropylene film for the untreated, 30 second, and 60 second plasma exposures. The PP film was treated with a 70 percent ethanol solution and dried prior to examination. The first image shows the surface topology of untreated PP film. The primary feature depicted is the overall smoothness of the film's surface. The second image, **Figure 8 D**, shows the plasma has etched of some of the film's soft plastic surface and is beginning to expose the hard plastic components of the film. The last image, **Figure 8 E**, continues the removal of the soft plastic components and appears to depict the film's underlying hard plastic components of PP film. For low-density plastics, the soft plastic components will begin to leach back to the surface from the bulk of the film. With several repeated direct plasma exposures, low-density plastics would tend to become brittle and harden and would thus have a limited amount of reusability.



**Figure 8 E.**

### **Tensile Strength Testing of Military Fabric**

Samples of military camouflage cloth were obtained from the Textiles and Nonwoven Developmental Center (TANDEC) located on the campus at The University of Tennessee and consisted of a pair of summer wear pants (50% cotton & 50 % rayon). Samples were initially cut by hand and then later using a die to minimize variations due to different sample widths. The samples were exposed to the exhaust airflow of the plasma using the APD 210 prototype decon device with operating parameters of 9kV and 8kHz. The samples were maintained at less than 140 degrees Fahrenheit during the four minute exposure to the exhaust. The samples were then taken to TANDEC where they were the tearing strength of the fabric was examined.

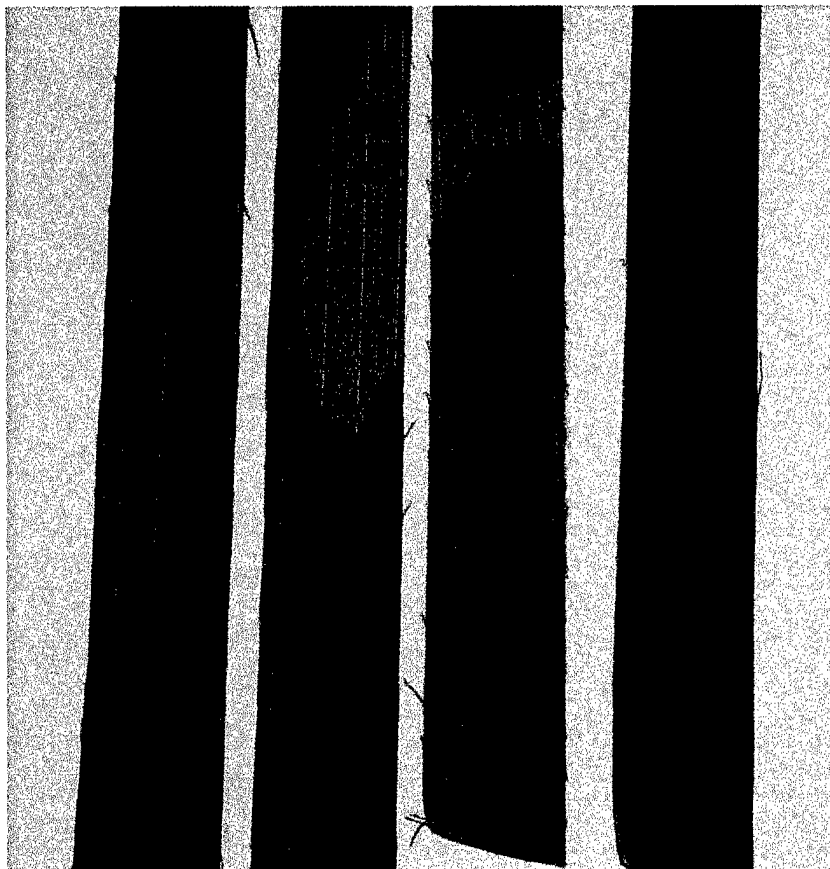
Using the ASTM D1117-80 protocol, an extensometer was used on 3 inch gauge samples for both the atm plasma treated and untreated samples. After minimizing the variations due to different sample widths, the control group of samples was found to vary slightly. Using the most repeatable data points the controls samples were found to have a mean peak tearing force of 34.25 kg while the samples treated for four minutes were found to have a mean peak tearing force of 32.56 kg. This difference amounts to approximately a 5 percent reduction in the tearing strength of the material after a four minute exposure to the exhaust of the plasma. According to personnel at TANDEC, a portion of the difference in tearing strength could be due to differences in the moisture content of the samples.

### **Color Alterations of Military Grade Fabric**

The same stock of military camouflage cloth was used to evaluate the changes in the color of the fabric. These samples were also maintained at a temperature less than 140 degree Fahrenheit during the four minute exposure to the exhaust of the plasma. After exposure both a control group and the exposed samples were taken to a testing facility where they were examined using a Data Color International CS-5 Spectrophotometer.

The color difference data was obtained by averaging four scans of each sample as compared to the control sample. Each scan was approximately 3.7 seconds in duration, at one nanometer increments from 400-700 nanometers spectral range. The light source was a quartz halogen cycle tungsten lamp which was filtered to eliminate infrared sample heating and to simulate D65 spectral distribution incident on the samples. A 10 degree observer was used to simulate the sample being held at arms length.

The military camouflage material is comprised of four different colors: green, brown, tan, and black. Color differences were observed for the green and brown colors. The black and tan color differences were not examined. For the green colored parts of the fabric, exposure to the exhaust from the prototype lightened the material between 8 to 11 percent. For the brown colored parts of the fabric, exposure lightened the material approximately 8 percent. Part of the color alteration could be due to the fabric being purchased new and not repeated washed to insure the color stability prior to testing. Shown on the following page is a photograph of the camouflaged fabric. The 1<sup>st</sup> and 3<sup>rd</sup> samples have been treated using the prototype, the 2<sup>nd</sup> and 4<sup>th</sup> were control samples.



**Figure 9.** Results of camouflage material testing. Samples 1 and 3 were treated to the exhaust airflow of the plasma for 4 minutes. Samples 2 and 4 were exposed to the airflow only.

### **Characterization of Chemical Reactions**

Discussions are still ongoing with potential candidates to act as the lead chemist in the analysis of the chemistry of the oxidative exposure effects of the exhaust of the APD-210 prototype. Atmospheric Glow Technologies is ideally located to have access not only to the staff of the University of Tennessee but also to the staff located at the Oak Ridge National Laboratory.

With the arrival of a Hiden Analytical HPR-60 (three-stage molecular beam triple-filter quadrupole mass spectrometer) scheduled for delivery in March of 2002, our chemical technicians will have access to one of the most sophisticated gas composition detection instruments commercially available.

In order to better understand the oxidative effects of the APD-210 the concentrations of ozone, NO<sub>2</sub>, and NO were examined for the current prototype. Using an Ozone Monitor that operates based on Beer's Law, the ozone concentration was observed to be approximately 1200 ppm. Using a chemiluminescence NOX detector, the concentration of NO<sub>2</sub> was observed to be between 71ppm and 79 ppm depending on distance from the exit nozzle of the device (1.5 inches for 79 ppm and 6 inches for 71 ppm). NO production was not observed using the standard operating conditions of the current blower prototype. The critical plasma active species responsible for the oxidation of CBW surrogates is thought to be monatomic neutral oxygen. The concentration and role of short-lived plasma generated active species will be examined upon arrival of the new Hiden mass spectrometer.

During the interim of the mass spectrometer arrival and installation, arrangements have been made to sample the exhaust gas during decontamination using a TO canister under vacuum. Thus far liquid or solid by-products from the oxidation of the simulants have not been observed. The TO canister will be used to determine the composition and concentration of aerosolized by-products. Once the sample is collected the canister will be taken to a nearby laboratory for analysis.

### **APD-210 Development**

The original APD-210 prototype has been greatly improved during the course of the Phase I Option period. These improvements allow the laboratory version of this device to operate for longer periods of time, at higher power levels, and lower temperatures. These improvements were made while also reducing the overall physical dimensions of the plasma generating elements nearly 50 percent.

The original plasma generating elements of the APD-210 were approximately 46 inches in length but are now only 23 inches in length. This not only allows the device to have a lower weight, but also lessens the current burden on the power supply. The previous prototype surface area was so large that the device was limited to an applied voltage of approximately 8 kVrms. By reducing the device length, the applied voltage has been raised to 14 kVrms.

The original prototype only had a limited amount of water-cooling capability. The new unit has an increased flow capacity allowing the device to operate for longer periods of time at a lower

temperature. Current operation protocols do not allow the temperature to increase above 170 degrees Fahrenheit (76°C). This restriction insures that development is relevant to heat sensitive items. Other enhances in the cooling system would lower the operating temperature further.

The power supply has been modified to have an improved impedance matching system. This allows the operation of the prototype to be more electrically efficient, while delivering more power to the prototype. Additionally, fewer electrical shutdowns have occurred since modifying the impedance matching and power supply characteristics.

In light of recent national events, the effectiveness of these prototype and power supply improvements have been assessed using biological simulants, in particular the spores *Bacillus subtilis*. var. Niger and *Bacillus anthracis* Stern F34 strain. The prototype developed during Phase One Option destroys log 6 (1 million) Niger spores in 4 minutes and log 5 Anthrax spores in 2-3 minutes at distances up to 4 inches from the exit nozzle of the device. The improvement in the effectiveness against chemical surrogates is under assessment in the phase II.

**Listing of Publications and Technical Reports**

No publications

Monthly Technical Reports were submitted for Feb-Aug 2001.

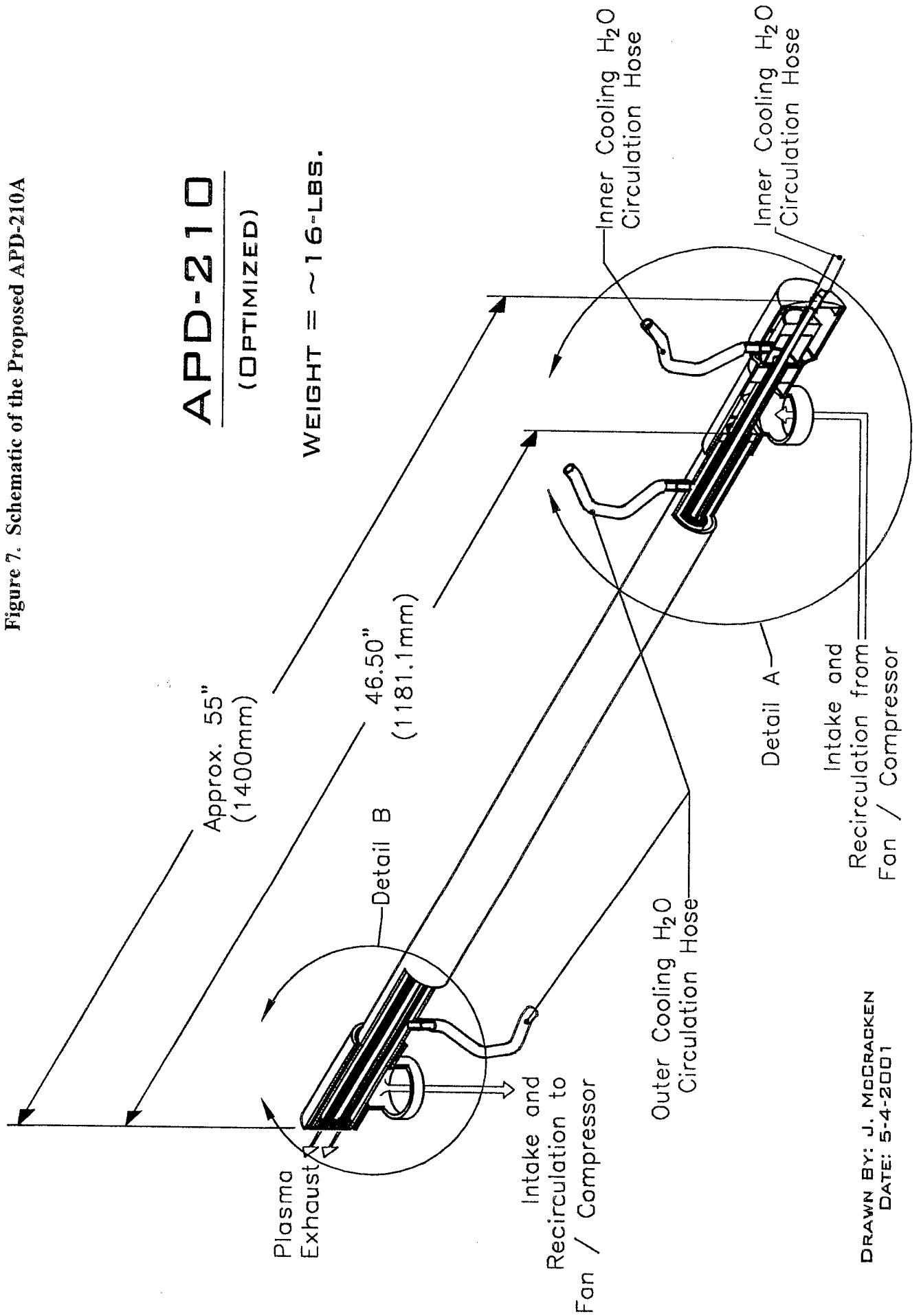
**Listing of Scientific Personnel Involved in the Project**

Personnel involved in Phase I study no advanced degrees were obtained during contract period

<b>Name</b>	<b>Affiliation</b>	<b>Role on project</b>
<b>Kimberly Kelly-Wintenberg, Ph.D.</b>	<b>Atmospheric Glow Technologies</b>	<b>Principal Investigator</b>
<b>Suzanne South</b>	<b>Atmospheric Glow Technologies</b>	<b>Director of Biological/Chemical Research</b>
<b>Daniel Sherman</b>	<b>Atmospheric Glow Technologies</b>	<b>Director of Plasma Technology</b>
<b>Michael Wimpee</b>	<b>Microbial Insights</b>	<b>Subcontractor</b>
<b>J. Reece Roth</b>	<b>University of Tennessee</b>	<b>Consultant</b>

List of Inventions- none

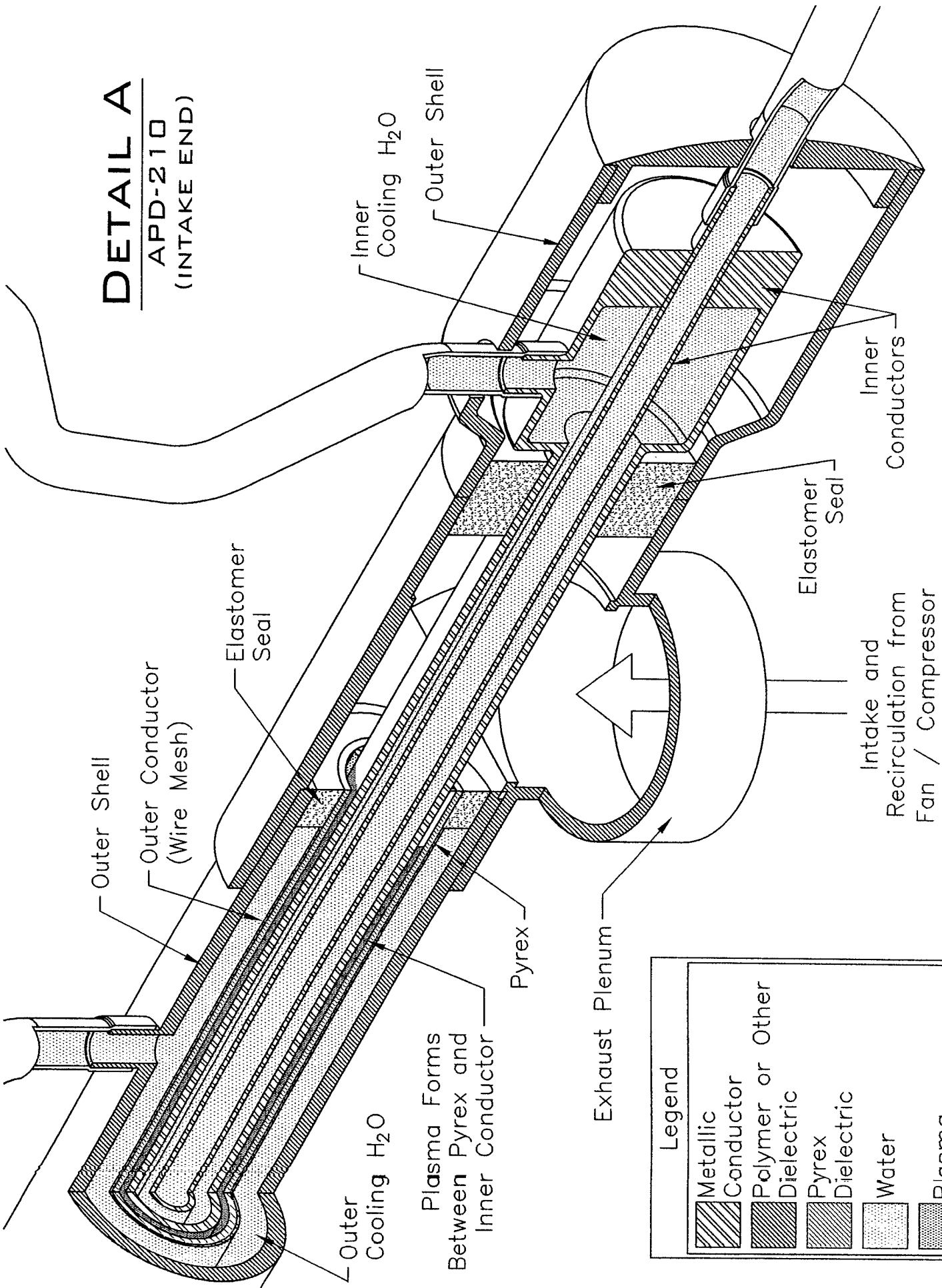
Figure 7. Schematic of the Proposed APD-210A



DRAWN BY: J. MCCRACKEN  
DATE: 5-4-2001

# DETAIL A

APD-210  
(INTAKE END)



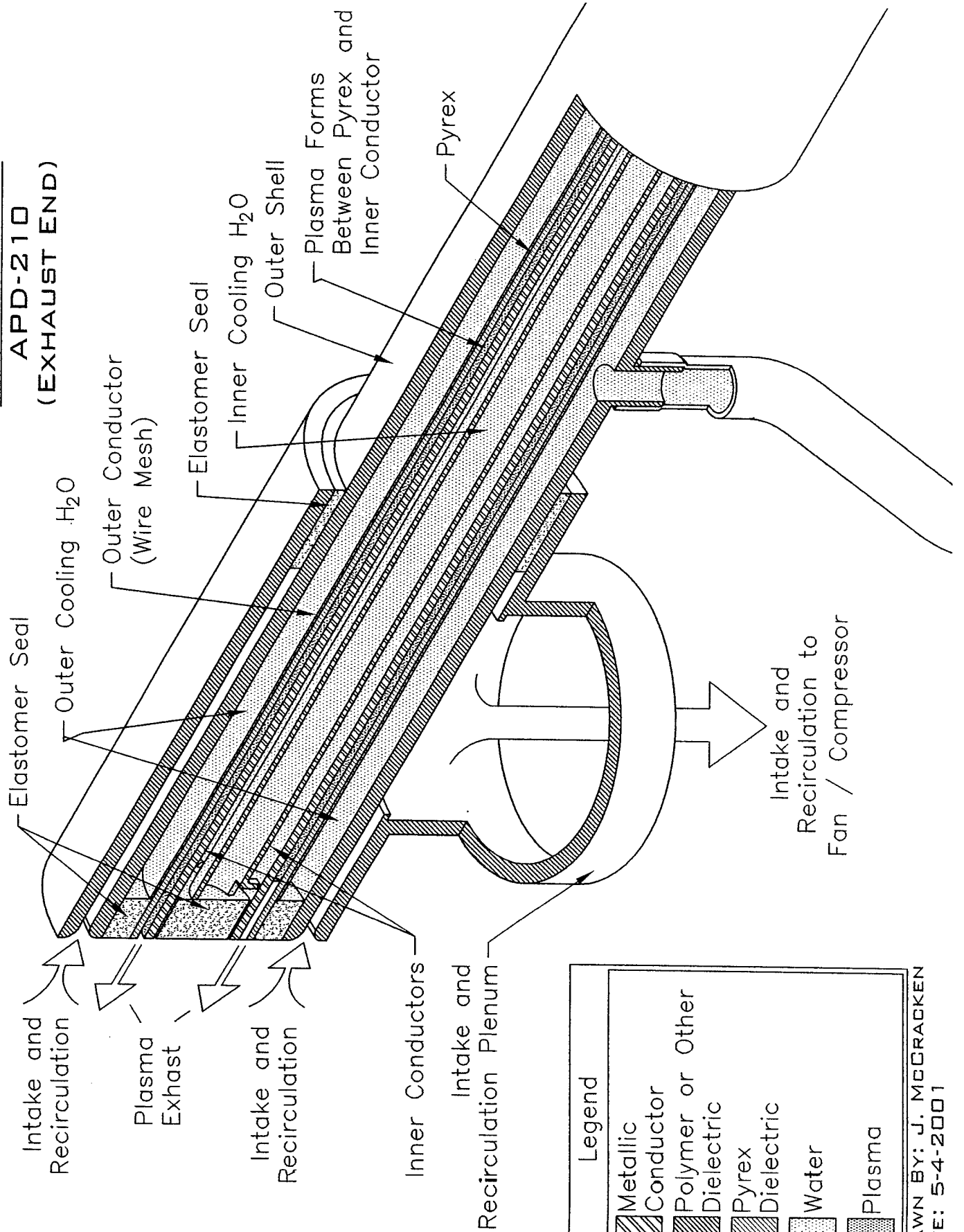
Legend

	Metallic Conductor
	Polymer or Other Dielectric
	Pyrex Dielectric
	Water
	Plasma

DRAWN BY: J. MCCracken  
DATE: 5-4-2001

# DETAIL B

APD-210  
(EXHAUST END)



Legend	
	Metallic Conductor
	Polymer or Other Dielectric
	Pyrex Dielectric
	Water
	Plasma

DRAWN BY: J. MCCracken  
DATE: 5-4-2001