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Report No. 9144

STUDY OF THE VAPOR PENETRATION OF CHEMICAL AGENT DROPLETS THROUGH FABRIC SYSTEMS

First Quarterly Progress Report
(June 1969 to September 1969)

by

S. S. Brody

December 1969

DEPARTMENT OF THE ARMY
Edgewood Arsenal
Defense Development and Engineering Laboratories
Physical Protection Laboratory
Edgewood Arsenal, Maryland 21010

Contract No. DAAA15-69-C-0715

MELPAR
An American-Standard Company
7700 Arlington Boulevard
Falls Church, Virginia 22046

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FOREWORD

The work reported herein was started in June and completed in September 1969.

DIGEST

The objective of the effort in the first quarter of the Study of the Vapor Penetration of Chemical Agent Droplets through Fabric Systems was to prepare tne analytical technique and to assemble the necessary equipment.

Several solvents were tested against the criteria of: evaporation rate; separation from agent using gas chromatography; and rapid analysis time. Two solvents met the criteria and were selected for use in the system. All the samples of fabric have been obtained from Edgewood Arsenal and prepared for testing. A means for disseminating the agents as monodispersed particles in the specified size and quantity has been set up; the metal test cups for holding the fabric have been fabricated; a controlled temperature and relative humidity chamber for performing the permeation tests has been built; and an analytical technique for agent analysis using gas chromatography with the Melpar Flame Photometric Detector has been developed.

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STUDY OF THE VAPOR PENETRATION OF CHEMICAL AGENT DROPLETS THROUGH FABRIC SYSTEMS

1. INTRODUCTION

The objective of the effort in the first quarter of the Study of the Vapor Penetration of Chemical Agent Droplets through Fabric Systems was to prepare the analytical technique and to assemble the necessary equipment.

Several solvents were tested against the criteria of: evaporation rate: separation from agent using gas chromatography; and rapid analysis time. Two solvents met the criteria and were selected for use in the system. All the samples of fabric have been obtained from Edgewood Arsenal and prepared for testing. A means for disseminating the agents as monodispersed particles in the specified size and quantity has been set up; the metal test cups for holding the fabric have been fabricated; a controlled temperature and relative humidity chamber for performing the permeation tests has been built; and an analytical technique for agent analysis using gas chromatography with the Melpar Flame Photometric Detector has been developed.

2. WORK PERFORMED

2.1 Fabric Materials and Systems

Materials

a. Sateen, Cotton, Nylon, Quarpel Treated 8.9-9.0 oz (R)

b. Serge Wool, 18.0 oz (W)

c. Nylon Oxford, 2.9 oz (N)

d. Poplin, Cotton, 6.0 oz (P)

e. Charcoal-Coated Poplin (CCP)

f. Sateen, Cotton, 8.2 oz (S)

g. CB Protective Overgarment 1969

h. Cotton-Nylon, 50-50 Twill, 5.5 oz

Systems for Test

The above materials are to be used in the following manner for the tests.

System	Materials
1	soiled a* over b over Baggie**
2	f over b over Baggie**
3	c over d over Baggie**
4	c over e over Baggie**
5	h over g over Baggie**

*Soiling procedure is shown in the appendix.
**0.5 mil polyethylene, Colgate-Palmolive Company.

2.2 Agent Dissemination

Figures 1 and 2 show the spray apparatus, including the motor driven syringe and the arrangement of concentric needles for delivering monodispersed particles of agent.

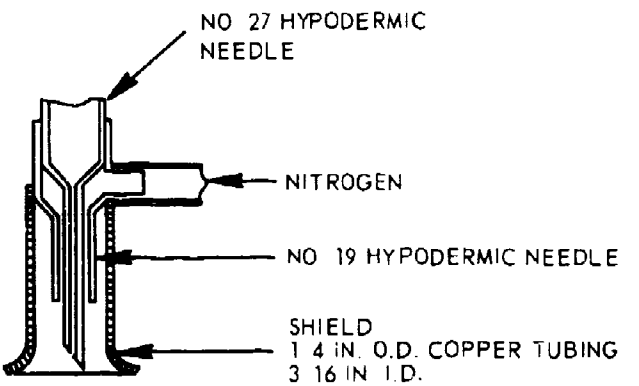

Figure 1. Needle Arrangement for Agent Dissemination Apparatus

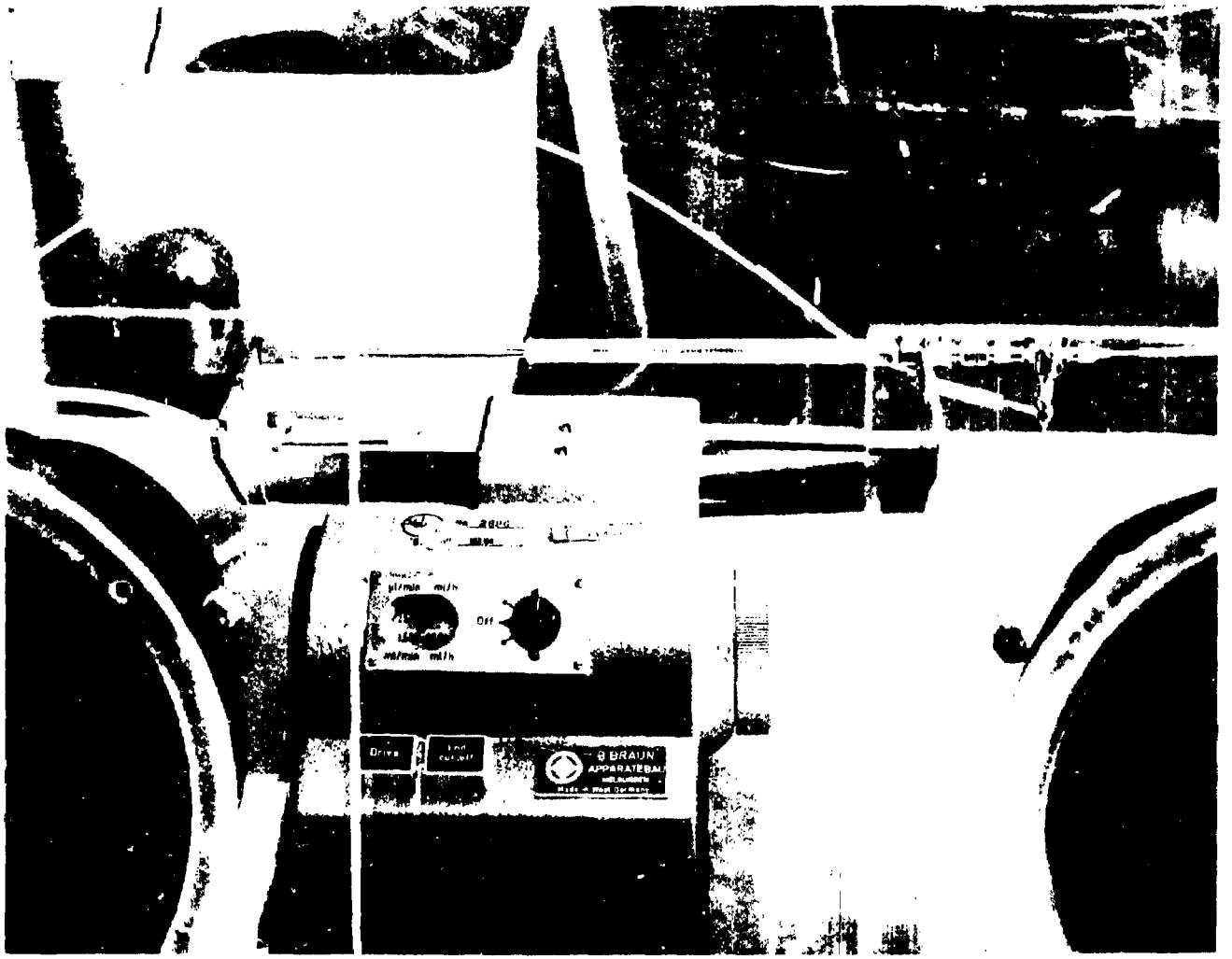

Figure 2. Photograph of Toxic Liquid Spray Apparatus

The syringe drive is capable of 24 different speeds and can operate with either a 50-ml or 5-ml syringe, giving 39 possible dissemination rates. With the nitrogen flowing at 650 ml/min, the agent rate at 0.0075 ml/min, a 16-sec spray will produce 2.0 mg of agent (equivalent to 2.0 g/m^2 on a 10 cm^2 surface) with a 600μ drop diameter. Table 1 shows setting for various disseminating rates.

TABLE 1

CONDITIONS FOR AGENT DISSEMINATION WITH 5-ML SYRINGE

Quantity (g/m^2)	Drop Size (μ)	Nitrogen (ml/min)	Delivery Rate (ml/min)	Spraying Time (sec)
2.0	600	650	0.0075	16
2.0	1200	400	0.0075	16
10.0	600	650	0.02	30
10.0	1200	400	0.02	30
20.0	600	650	0.02	60
20.0	1200	400	0.02	60

2.3 Sample Test Cups

Twenty-four sample test cups have been fabricated. The cups have been altered slightly from those utilized at Melpar on Contract No. DAAA15-68-C-0625; however, the spacing and all critical parameters have been kept the same. Swagelok quick disconnects are utilized on the top half of the cup and Swagelok fittings connected with O-ring seals are used on the bottom.

2.4 Test Chamber for Controlled Temperature and Relative Humidity

A test chamber and ancillary equipment was designed and fabricated for carrying out the work on this contract. This test apparatus can be divided into three subsystems, which are (1) air supply, (2) temperature and relative humidity (RH) control, and (3) sampling system. The environmental chamber is constructed of polypropylene and is 24 in. high, 36 in. wide, and 18 in. deep. Access to the interior is by way of a hinged Plexiglas door.

The basic parameters for conducting these tests dictate that the air passing through the sample cups be free of particulate and organic matter, and be maintained at a controlled temperature, humidity, and selected specific velocity. The air passing through the top and bottom of the cups is drawn through a set of bubbler sample collectors for analyses.

The program requirements establish specific test conditions including air temperature at 25° C and 50% relative humidity and air velocities through the cup of 0.5, 1.5, and 7.0 mph. The system, however, has considerable flexibility and capacity to conduct studies under a variable set of conditions in temperature, humidity, and airflows.

As denoted in figure 3, the air supply passes through a filter (1) for the removal of water, organic, and particulates. The air is pressure regulated (2) before branching off to the heat exchanger and RH generator subsystems. These independent air flows are controlled and regulated by needle valves (3) and rotameters (4 and 5). They are used to set up the test program dependent on test stations in operation and air flow requirements for the sampling system. The RH generator (6) consists of a 3-necked flask containing a coarse fitted disc, thermometer, and exhaust line outlet. The heating mantle temperature is controlled by a Variac. The outlet of the humidifier air enters the test chamber as indicated in figure 3.

The sample air passes through a preliminary heat exchanger coil (7) of 20 ft, 3/8 in. OD copper tubing. This coil which is considered the first stage is equilibrated at the operating temperature of the chamber. The air then flows by a second (flowing-water) heat exchanger (11). The air is drawn across a Nichrome wire heating element (12) before being exhausted by the fan (8) and recirculated throughout the controlled chamber. Test operating conditions are set such that inlet air volumetric capacity is always greater than the air exhaust through the sample cups. This is to insure that a slight positive pressure differential exists in the interior as against the external chamber pressure; thus, precluding any possibility of external air entering the chamber and being drawn through the sample cups.

The second system is the method of maintaining temperature control. This system is controllable within the range of 0° to 50° C. Under test conditions below 0° C, the cooler (10) can operate in a closed-loop system using a recirculating pump and a mixture of water and ethyl alcohol. Presently a tap water supply is utilized for 25° C conditions as shown in figure 3. Needle valves (3) are used to balance the water flow through the cooler (10) depending on ambient temperature conditions. The water is circulated through the heat exchanger radiator (11) to drain or, as previously stated, recirculated through the cooler. The combination of heat exchanger (11) and heating element (12)

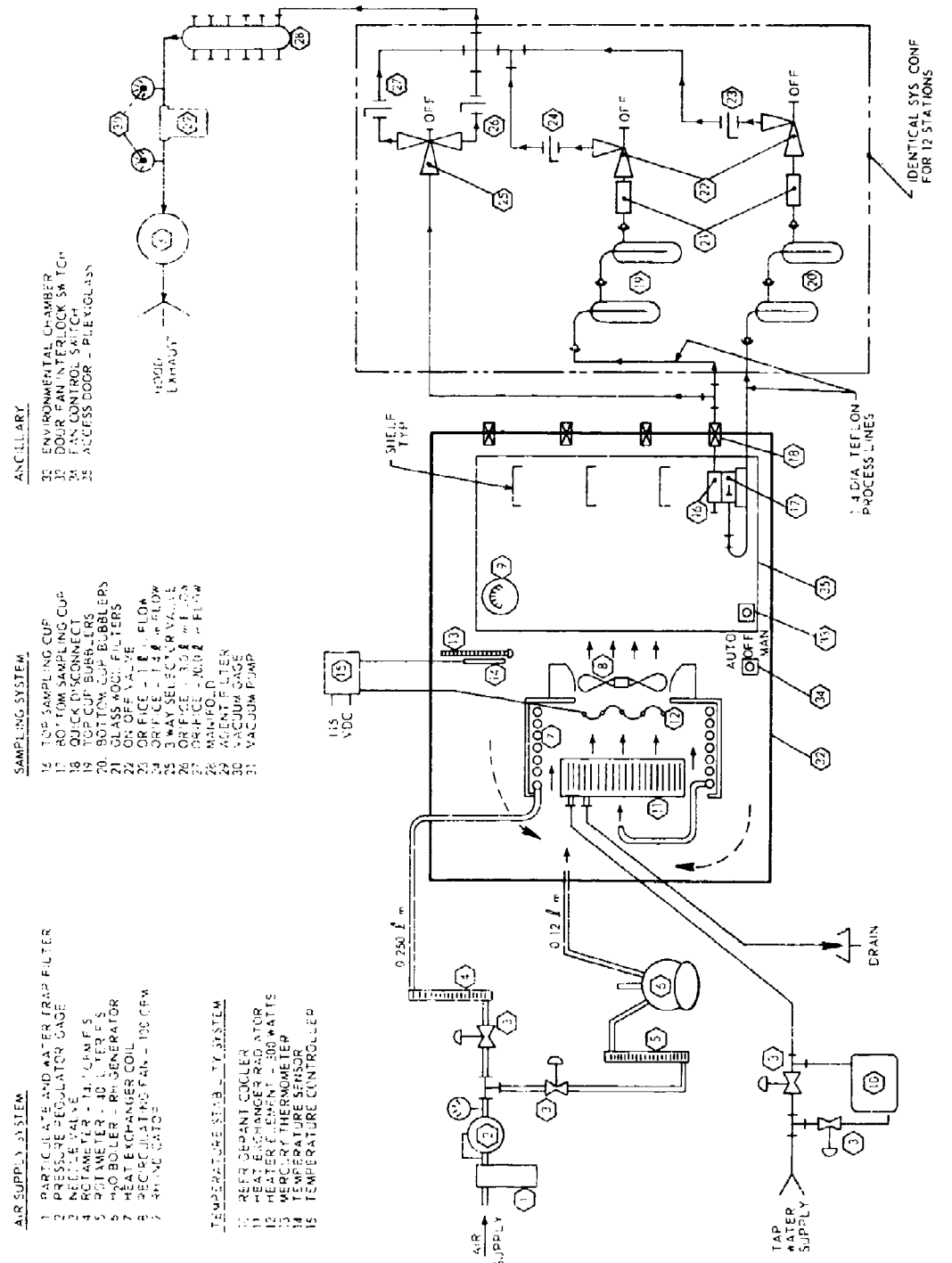

Figure 3. Test Apparatus for Vapor Penetration Studies

assures close temperature control. Temperature sensor (14) and controller (15) are referenced and visually monitored by a calibrated mercury thermometer (13). Relative humidity is monitored by indicator (9).

The air sampling system comprises twelve identical sampling stations with the capability of selecting three different airflow rates for present test purposes. This arrangement can be easily adapted to different flow rates by changing the orifices which set the airflow requirements. The system now provides nominal flow rates of 1.0 l/min for the bottom sample cup and 1.4, 3.0, and 20.0 l/min for the top sample cup. Air velocities of 0.35 mph for the cup are obtained at 1.0 l/min. The following wind speeds are obtained with the indicated flow rates:

0.5 mph = 1.4 l/min

1.5 mph = 4.4 l/min

7.0 mph = 21.4 l/min

The sample cups are prepared and assembled external of the chamber. The assembled cup is then installed, the top sample cup (16) connected to the sampling system by a quick-disconnect (18) and the bottom cup (17) by a Swage-lok nut. The conditioned air is drawn through the bottom cup and passed through a set of sample collection glass bubblers (20). The sample air then is passed through a polyethylene tube containing glass wool (21) to the on/off valve (22), through the flow control orifice (23) into the manifold (28).

Similarly, the conditioned air passes through the top sample cup, through its own set of bubblers (19), filter (21), on/off valve (22), flow orifice (24) into the manifold (28). Since the sample collection glass bubblers (19), however, are arbitrarily limited to a maximum volumetric flow of 1.4 l/min the process line is split at the disconnect junction. Only part of the top cup sample flow goes through the bubblers. This additive flow sets up test conditions for flow studies beyond the 1.4 l/min limitation. The system incorporates a 3-way selector valve (25), which channels the air through selective orifices (26) or (27).

All 12 stations terminate in a manifold (28), which operates at a pressure of less than 380 mm Hg ABS to maintain critical flow of the orifices. Sample contamination of the exhaust air, which leaves the hood to reach the vacuum pump (31), is removed by an MSA filter (29) before the expended air being returned to the exhaust hood by the vacuum pump (31). A safety factor is exercised at the filter which is monitored by two vacuum gages (30). These gages indicate the pressure drop across the critical orifices and the pressure

drop across the MSA filter. Thus, a visual indication is discernible if the pressure drop on the filter becomes excessive.

It should be pointed out at this time that all fittings and sample lines from the sample cups to the bubblers are stainless steel and Teflon. Lengths of all sample lines have been equalized and kept to a minimum.

An additional safety feature which is pertinent in the test apparatus is the interlock which controls the recirculating fan. The automatic interlock door switch (33) turns off the fan when the access door (35) is opened. This prevents the operator from inadvertently being subjected to a blast of air when installing the sample cups.

Photographs of various views of the chamber and test setup are shown in figures 4 through 7. As seen in the photographs the equipment is set up in a hood.

The data provided in table 2 indicates the actual flows obtained at the various stations of the test setup. This data is to be kept up to date during the future sample runs. The actual flow rates will be utilized in performing the calculations during the test runs.

2.5 Analytical Procedures

In this study it is required to collect the vapor from the top and bottom of the sample test cup in solvent. The solvent is then to be analyzed by gas chromatography, using the Melpar Flame Photometric Detector. It was necessary, therefore, to select a solvent through which air can be bubbled for 24 hours at room temperature without a significant loss of solvent by evaporation. In addition, the solvent must be such that it is adequately separated from tripropyl phosphate by gas chromatography (GC) and the GC analysis time must be held to less than three minutes per sample. After a number of solvents were tried, two proved to be satisfactory.

These two were ethyl phthalate and 2-(2-ethoxyethoxy) ethyl acetate. Table 3 shows evaporation data for these two solvents.

The 2-(2-ethoxyethoxy) ethyl acetate proved to be the more efficient solvent for both handling (viscosity) and analysis. Using the gas chromatograph and the Melpar Flame Photometric Detector, a calibration curve for tripropyl phosphate ranging from 1.0 γ/ml to 500 γ/ml was made (figure 8).

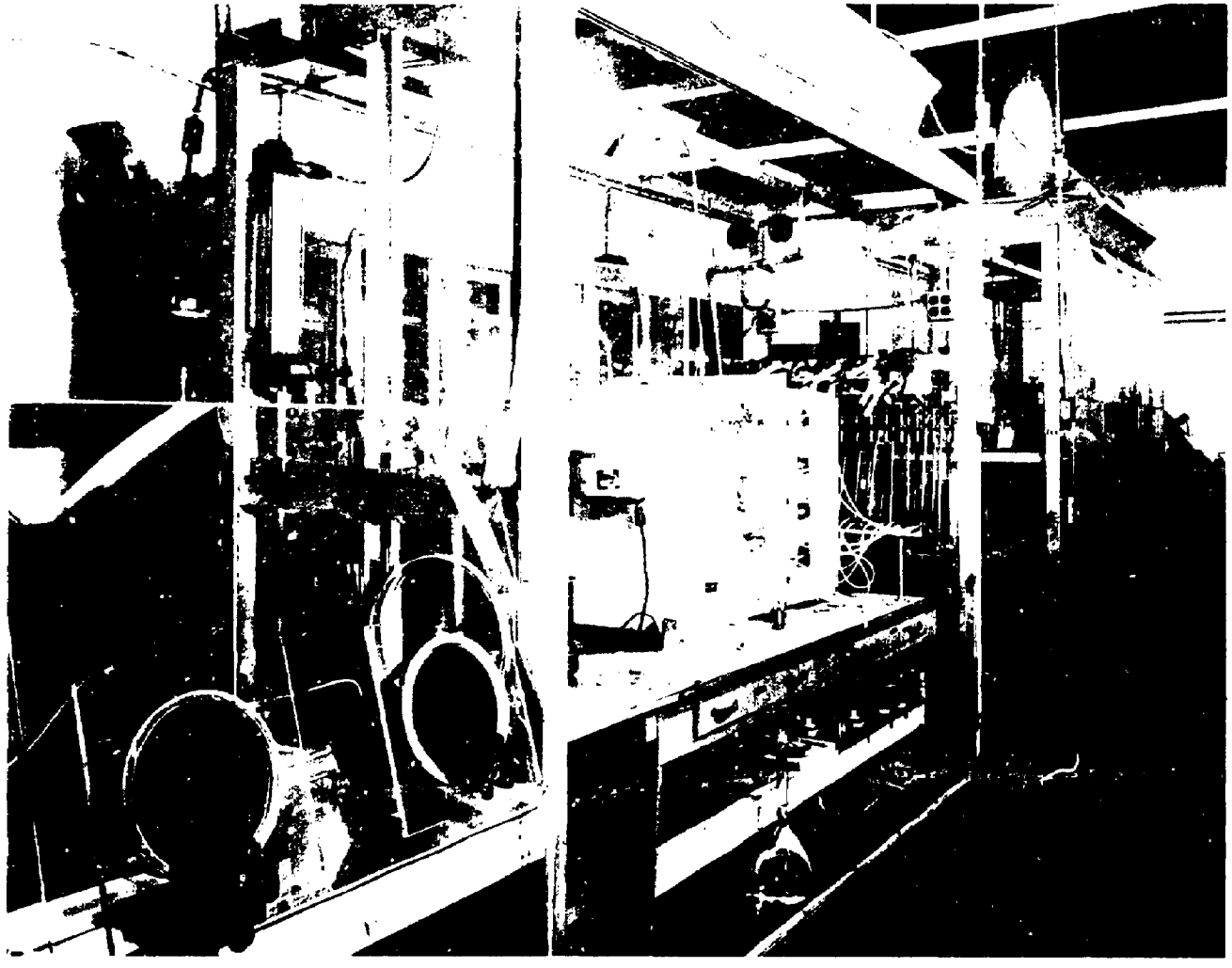

Figure 4. Photograph of Controlled Temperature, Relative Humidity Chamber and Test Setup

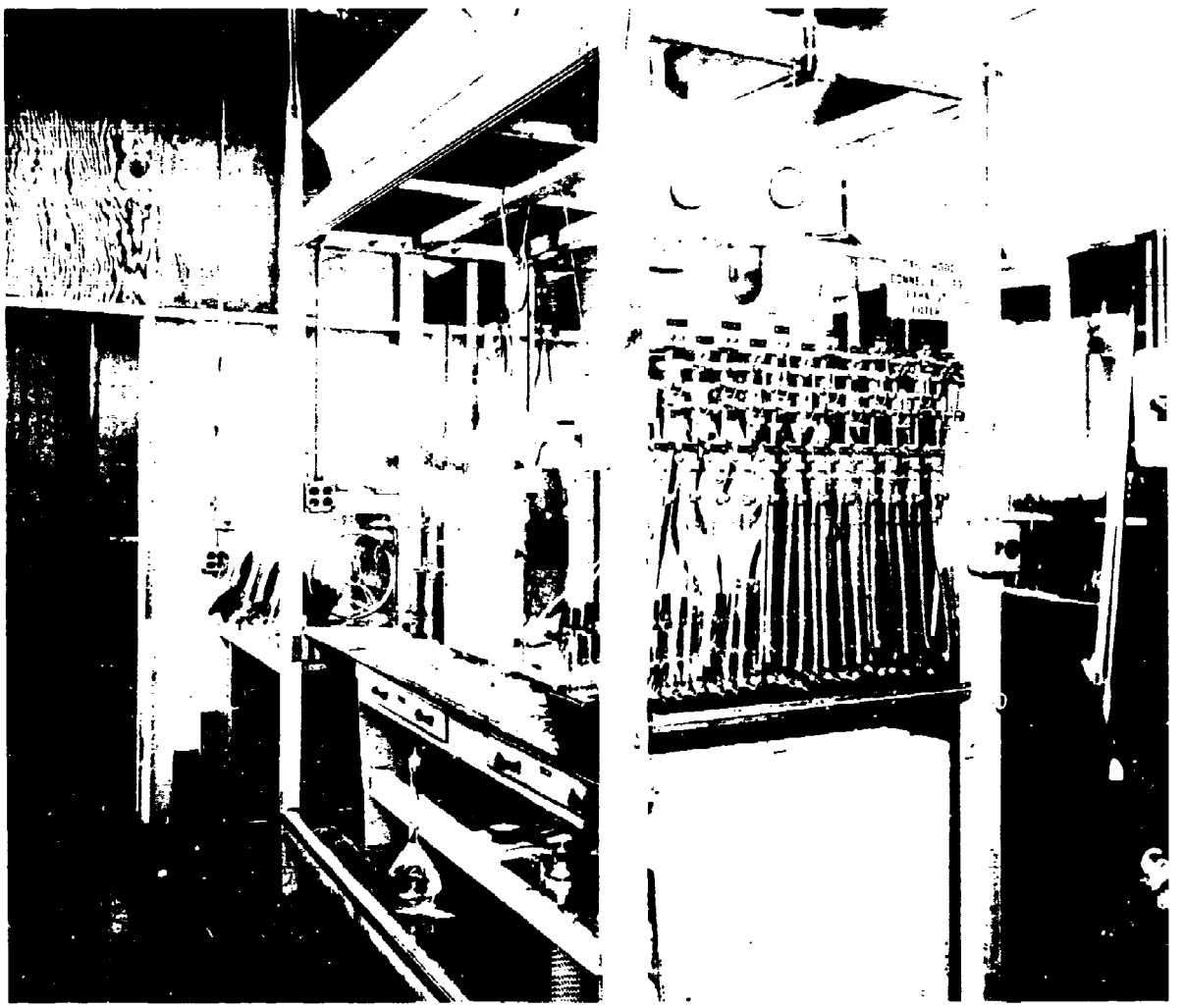

Figure 5. Photograph Showing Valve Assembly

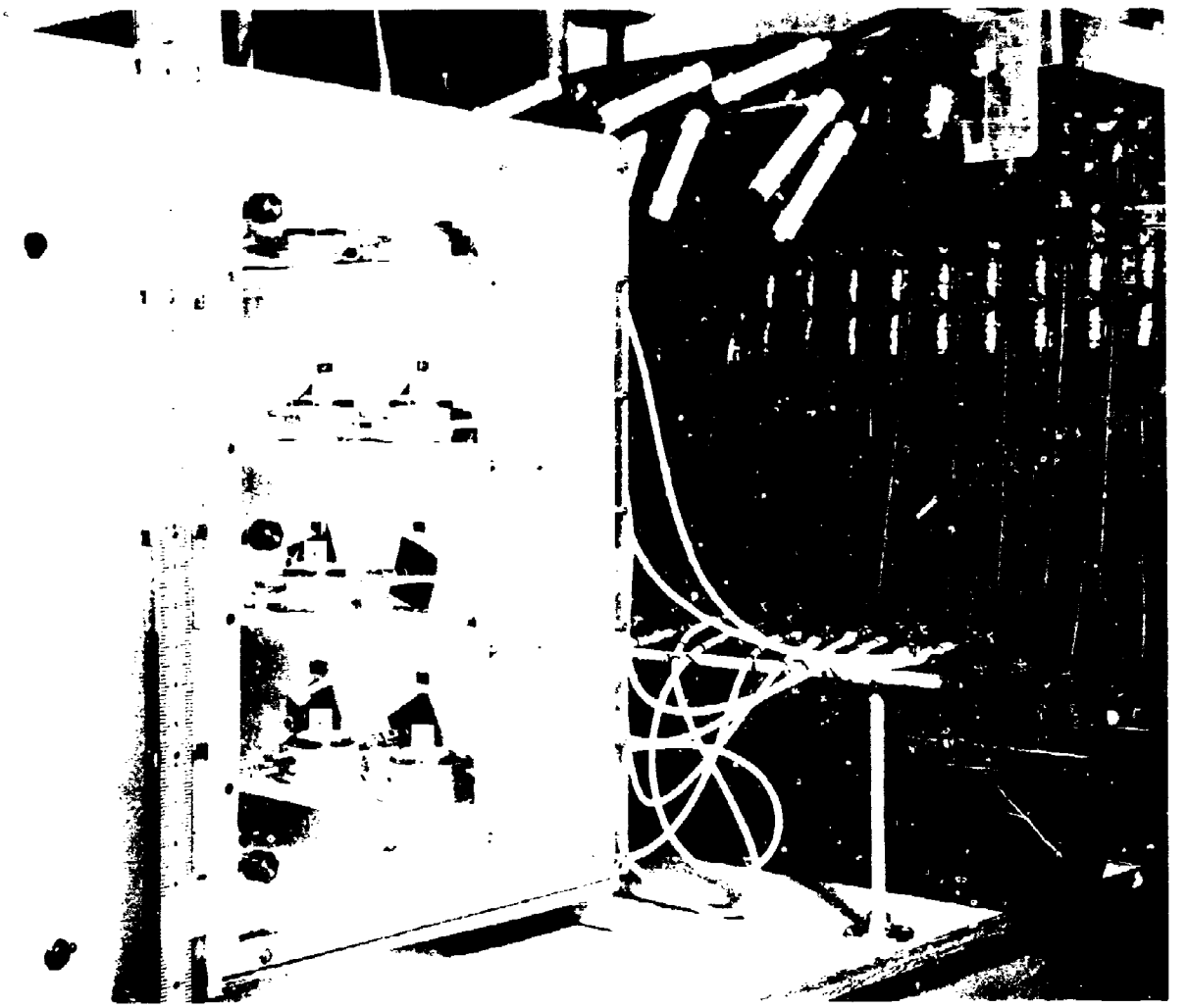

Figure 6. Photograph Showing Cup Placement in Test Chamber with Bubbler Setup

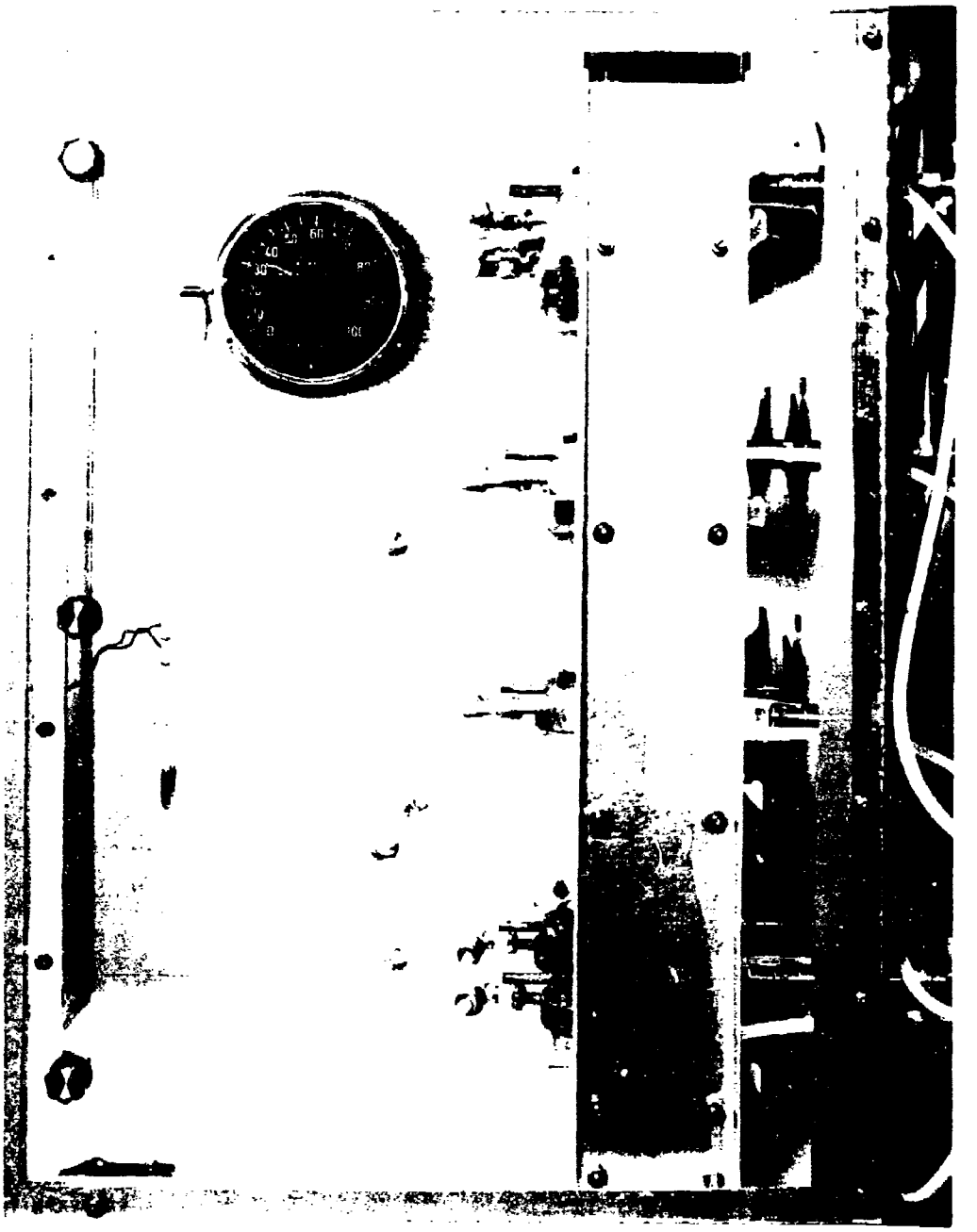

Figure 7. Photograph Showing Temperature Controller and Relative Humidity Gage

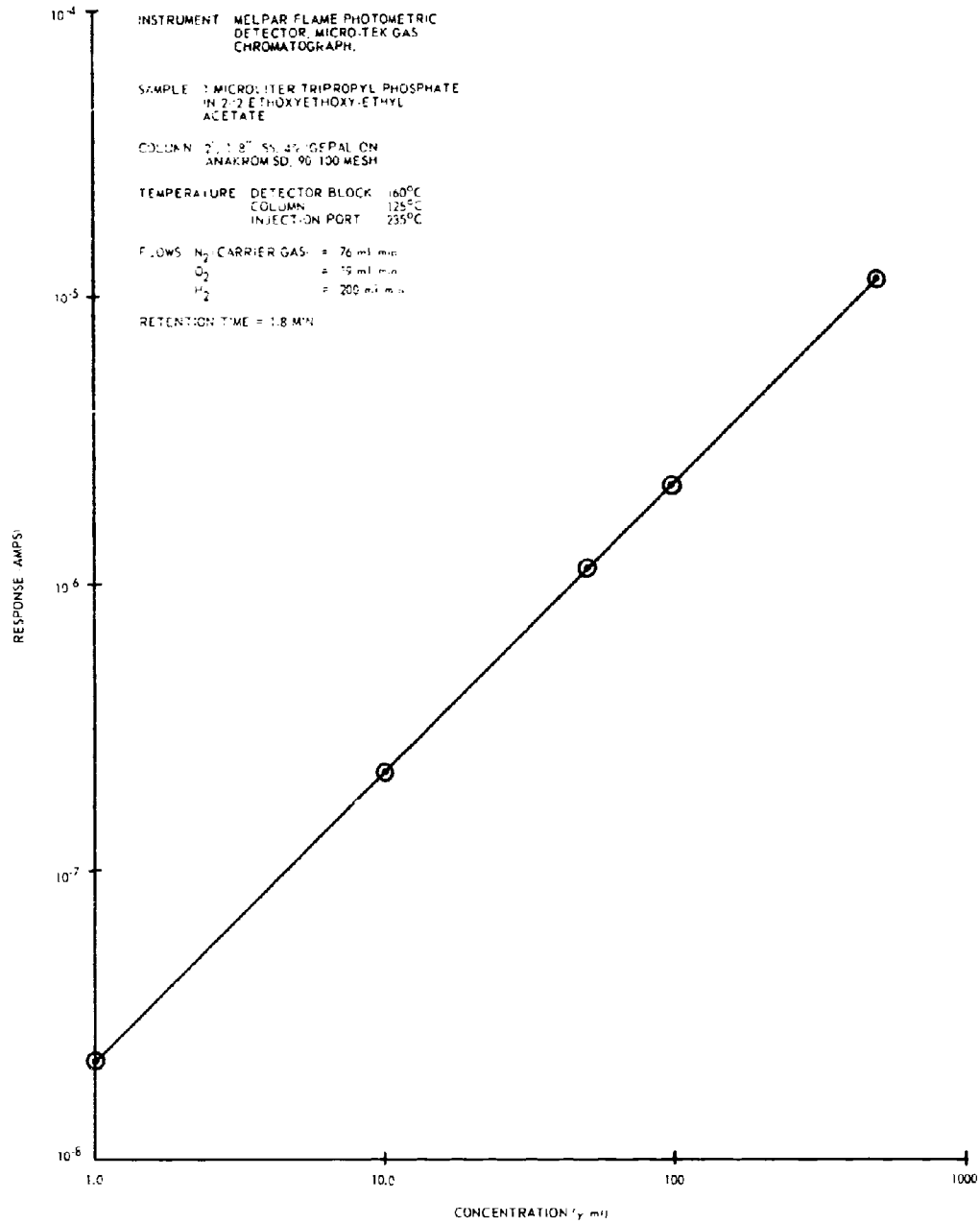

Figure 8. Calibration Curve for Tripropyl Phosphate

TABLE 2

FLOW RATES FOR BUBBLER STATIONS
(In Liters Per Minute)

	Bottom	Top		
Nominal	1.0	1.4	4.4	21.4
Station				
1	1.0	1.4	4.2	16.6
2	1.0	1.3	4.2	17.0
3	1.0	1.3	4.3	17.0
4	1.1	1.3	4.2	16.8
5	1.0	1.3	4.2	17.1
6	1.0	1.3	4.3	16.5
7	1.0	1.4	4.2	16.8
8	1.1	1.3	4.3	17.1
9	1.0	1.3	4.3	17.2
10	1.0	1.2	4.2	17.0
11	1.1	1.3	4.3	16.6
12	1.0	1.3	4.3	17.2

TABLE 3

EVAPORATION RATES OF ETHYL PHTHALATE AND
2-(2-ETHOXYETHOXY) ETHYL ACETATE

Ethyl Phthalate: 16.5-hr. test					
Front Bubbler			Back Bubbler		
Gross wt at start g	Gross wt at finish g	loss	Gross wt at start g	Gross wt at finish g	loss
57.7	56.8	0.9	59.0	57.2	1.8
54.5	54.5	0.0	63.0	63.0	0
2-(2-Ethoxyethoxy) Ethyl Acetate: 12-hr test					
55.2	54.4	0.8	57.7	57.4	0.3
*61.0	60.2	0.8			

Bubbling rate = 1.4 l/min
Solvent in bubblers, 5 ml
*Single bubbler test (10 ml of solvent)

3. PROGRAM FOR NEXT QUARTER

During the next quarter of the program, permeation studies using tripropyl phosphate will be initiated. Tests utilizing five different fabric systems, two drop sizes, two coverages, and the three different wind speeds are planned.

APPENDIX

PROCEDURE FOR SOILING REPELLENT FABRICS

1. Preparation of Soil

a. Soil Formulation*

Component	% by weight	Amount used in 1 gal ball mill
		g
Michigan Bacti Peat Moss (66% moisture) (Michigan Peat Co.)	51	800
Cement, Portland, Atlas Brand (United States Gypsum Co.)	16.2	255
Silica (sand)	16.2	255
Kaolin Clay, Bakers NF (J.T. Baker & Co.)	16.2	255
Ferric Oxide, red, anhydrous (Fisher Scientific Co.)	0.4	7

b. Preparation of Soil Mixture

All material was used as received with the exception of the sand which was sifted through normal window screen and then ball milled dry for 24 hr (1 gal ball mill, 5 lb of sand, 75 each, 20-mm diameter ceramic balls).

The materials, as received, and the ground sand were blended by hand in the proportions given above and placed in a shallow metal pan (blend was approximately a 1/2-inch layer) in a circulating oven at 50°C for 8 hr. The mixture was then ball milled for 24 hr (1 gal ball mill, 1572 g of mixture, 75 each, 20-mm diameter ceramic balls).

* Modification of formula contained in American Cyanamid Co. Textile Finishing Bulletin No. 148-A, Testing Sheet 1, dated December 28, 1955, titled: Juvenon Soil Retardant R, Accelerated Soiling Test.

2. Fabric Soiling Procedure

After washing, the fabric was cut into 1-yd lengths (for ease of handling). Each piece was weighed before being tacked down on a plywood table of sufficient size to accommodate the fabric. The fabric was secured to the plywood table using thumbtacks in the selvages only. This was done by hand and in such a manner so as to remove wrinkles, but not to cause any distortion of the fabric weave.

Five-hundred grams of the synthetic soil was spread out on the fabric in a random manner. This soil was then spread out as evenly as possible over the entire piece of fabric by hand, at the same time using a rubbing action and hand pressure to force to soil beneath the surface of the fabric. Once this was accomplished the fabric was brushed by hand using a scrubbing brush (FSN 7920-282-2470). The direction of scrubbing was back and forth on a line from the selvages to the center of the fabric with an emphasis on moving the excess soil to the center of the fabric. The soil was then rerubbed into the fabric by hand as before to produce a uniform covering. It was brushed again, but this time, starting at the center and working to the selvages. This complete cycle of spreading and rubbing by hand and brushing was repeated for the cut edges, and the complete process was repeated until the soil was worked into the fabric for a minimum of 6 min. The fabric was then brushed from selvage to selvage and as much of the excess soil as possible was collected. This was normally in the range 200 to 250 g.

The material was then vacuumed using a standard floor attachment without a brush of any type to remove excess soil. This was accomplished by making only single passes over the material from selvage to selvage. The fabric was then removed from the table and, while being held at two corners, was shaken once or twice, then folded and reweighed. An increase of 6% to 8% in total weight in combination with a reasonably uniform appearance was considered acceptable.

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13. ABSTRACT

The objective of the effort in the first quarter of the Study of the Vapor Penetration of Chemical Agent Droplets through Fabric Systems was to prepare the analytical technique and to assemble the necessary equipment. Several solvents were tested against the criteria of evaporation rate, separation from agent using gas chromatography, and rapid analysis time. Two solvents met the criteria and were selected for use in the system. All the samples of fabric have been obtained from Edgewood Arsenal and prepared for testing. A means for disseminating the agents as monodispersed particles in the specified size and quantity has been set up; the metal test cups for holding the fabric have been fabricated; a controlled temperature and relative humidity chamber for performing the permeation tests has been built; and an analytical technique for agent analysis using gas chromatography with a Flame Photometric Detector has been developed.

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Clothing						
Agent						
Monodispersed agent						
Relative humidity control						
Temperature control						
Clothing ensemble						
Permeation						
Permeation test cup						
Agent analyses						
Gas chromatography						
Melpar Flame Photometric Detector						