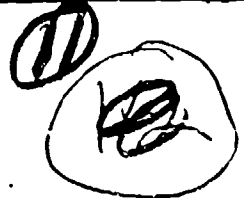


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U.S. ARMY CHEMICAL AND BIOLOGICAL DEFENSE COMMAND

ERDEC-TR-191

**ADHESIVE STUDY
FOR THE M40A1 CHEMICAL-BIOLOGICAL PROTECTIVE MASK'S
QUICK DOFF HOOD**



William M. Fritch

PM NBC DEFENSE SYSTEMS

August 1994

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PREFACE

The work described in this report was authorized under Project No. 10464806D019, Chemical/Biological Individual Materiel. This work was started in June 1993 and completed in November 1993.

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ADHESIVE STUDY FOR THE M40A1 CHEMICAL-BIOLOGICAL PROTECTIVE MASK'S
QUICK DOFF HOOD

1. INTRODUCTION

The current design of the M40A1 quick doff hood has a sewn and taped seam construction, which requires using an adhesive to keep the tapes bonded to the seams. The 3M 1357 adhesive was used in assembling the development prototypes evaluated in the User Test/Technical Test. During the development effort, the contractor expressed some concern over the adhesion strength of the 3M 1357 adhesive because the tapes peeled from the hood with only moderate force. This was perceived to be the weak link in the durability of the hood going into testing. Although testing showed no deficiency in the tape adhesion, efforts continued to identify adhesives to increase seam adhesion and eliminate the sole source adhesive requirement.

In constructing the hood, two panels of butyl-coated nylon are sewn together. A 1/4-in.-wide, butyl-coated nylon strip is glued over the seam to cover holes caused by the needle penetrating the material during the sewing operation. The needle holes must be sealed to prevent liquid agent from passing through them and contaminating bare skin. In the gluing operation, two coats of adhesive are applied to the seam and the tape. This design was chosen to eliminate the use of luting to cover the holes. Concerns with using luting include possible inadequate covering of needle holes, minimal resistance to agent, and surfacing or opening of holes after hood use by a soldier. The benefits of luting include its ability to prevent the direct pass-through of liquid agent and to increase soldiers' confidence that the hood is hole free. On the other hand, adhesive application to the tape and seam is labor intensive, and improved application techniques or alternative adhesives could improve these operations in hood production. This study investigated alternative adhesives and procedures, which could alter the technical data package of the hood to enhance its producibility.

2. TESTING

The first step in the investigation was to define the critical performance criteria for the sewn and taped seam. The main criterion is that the tape remain bonded to the seam through the mask's operational/environmental requirements. These requirements include cold, hot, and wet environmental conditions. Other criteria are as follows: the adhesive must not cause the hood to stick to itself (blocking) after assembly, and it must not damage the butyl rubber (evaluated using hydrostatic resistance testing). From review of existing documents and past test scenarios, several tests were determined as critical in testing the performance of a sewn and taped seam. The main document used in setting test requirements and criteria was the purchase description (PD-EA-H-1349) for the hood assembly of the M43/M43A1 masks. Based on this information, the following tests were adopted into the test plan:

▪ Hydrostatic resistance of adhesive-coated cloth. The hydrostatic resistance of the adhesive-coated cloth shall not be <90% of the actual hydrostatic resistance of the butyl-coated cloth prior to cementing, when tested as specified in Section 2.2.

▪ Cold crack. The adhesive, when applied and tested as specified in Section 2.1, shall not crack or flake.

▪ Strapping adhesion. Strapped seams shall meet the adhesion requirements specified in Table 1.

Table 1. Strapping Adhesion

Adhesion	Pounds/Inch of Width, Minimum	Test Paragraph
Initial	3.0	2.3.1
After water immersion	2.0	2.3.2
After heat aging	3.0	2.3.3
After cold storage	3.0	2.3.4

▪ Blocking. The strapped seams shall receive a maximum rating of No. 2 when tested as specified in Section 2.4.

The specific test procedures will be conducted in accordance with the following paragraphs.

2.1 Cold Crack of Adhesive-Coated Cloth.

Test specimens, measuring 8 by 8 in. minimum, were randomly cut from the coated-cloth sample. Test specimens were cleaned on one side only by wiping them with a suitable cleaning agent (isopropyl alcohol), which did not damage the butyl coating. The cleaned areas of three specimens were coated with the specified adhesive. Three samples coated with each adhesive were required. The test specimens were conditioned at room temperature and dusted until tack free. They were then tested as specified in Method 5874 of Federal Test Method Standard No. 191,¹ except the testing temperature was -20 °F, and the specimens were exposed to this temperature for not <4 hr before testing. Method 5874 specifies a temperature of 20 °F. The lower temperature was selected to be more representational of the operational environment. When performing these tests, the adhesive side was considered the heavier coated side.

2.2 Hydrostatic Resistance of Adhesive-Coated Cloth.

For hydrostatic resistance testing, three coated specimens (prepared as discussed in Section 2.1) and three specimens without an adhesive coating

were tested for hydrostatic resistance as specified in Method 5512 of Federal Test Method Standard No. 191.² The side without the adhesive coating was exposed to the water. Three coated specimens for each of the adhesives were required.

2.3 Strapping Adhesion.

Seam specimens, 5 + 1 in. long by 1 + 1 in. wide, were tested for adhesion as described in the following sections:

2.3.1 Initial Adhesion.

Three to five strapped seam specimens, depending on adhesive availability, were tested for seam adhesion in accordance with Federal Test Method Standard No. 191 (Method 5962³ for sewn and strapped seams).

2.3.2 Adhesion After Water Immersion.

Three to five strapped seam specimens, depending on adhesive availability, were immersed for not <2 hr in boiling water and removed. After being removed from the boiling water, the specimens were immersed for not <15 min in water at 75 ± 5 °F and removed from the water. While the specimens were still dripping wet, they were tested for adhesion in accordance with Federal Test Method Standard No. 191 (Method 5962³ for sewn and strapped seams).

2.3.3 Adhesion After Heat Aging.

Three to five strapped seam specimens, depending on adhesive availability, were exposed for not <7 days in a circulating air oven at 158 ± 2 °F, removed from the oven, conditioned for not <24 hr at 70 ± 2 °F and $64 \pm 2\%$ relative humidity, and then tested for adhesion in accordance with Federal Test Method Standard No. 191 (Method 5962³ for sewn and strapped seams).

2.3.4 Adhesion After Cold Storage.

Two test samples were exposed for 4 hr at a testing temperature of -20 ± 2 °F. The samples were tested for adhesion in accordance with Federal Test Method Standard No. 191 (Method 5962³ for sewn and strapped seams). This test was added because of the lack of Clemson adhesive samples for cold-crack testing. In addition, this test provided added informational data on the cold weather performance of the various adhesives.

2.4 Blocking.

Three strapped seams were tested for blocking in accordance with Federal Test Method Standard No. 191 (Method 5872⁴ for high temperature effect on cloth blocking). In folding the samples, the first fold was from seam to seam such that the seam was back to back.

3. TEST VARIABLES

The following list of variables was used in conducting the evaluation:

• Adhesives. The following adhesives were evaluated in this study. A listing of the company names and addresses is provided in Appendix A.

- 3M 1357 - high performance, rubber-based adhesive
- Clifton FA 1013 - MIL-A-5540³ - two-part adhesive
- Clifton FA 1040 - Neoprene-based, two-part adhesive
- 3M 950 - pressure-sensitive, acrylic adhesive
- 3M 9485 - pressure-sensitive, acrylic adhesive
- Clemson Apparel - adhesive-backed butyl, pressure-sensitive adhesive, Types IV and V

• Other variables.

- Single coating was used for adhesives requiring two coatings
- Varied tape widths of $\frac{1}{4}$, 1, and $1\frac{1}{2}$ in.
- Biased and nonbiased tapes

4. TEST SAMPLES

Samples for the seam adhesion were constructed as follows: Two strips, $4\frac{1}{4}$ -in. wide by 48-in. long, were sewn as indicated in the quick doff hood assembly drawing, 5-1-2701. The various width straps were applied in accordance with the test variables listed in Table 2.

Samples were cleaned with isopropyl alcohol prior to adhesive applications. The 3M pressure-sensitive tapes were the exception. They were cleaned with the alcohol; additional talc was removed with transparent tape. The adhesive was applied to the light side of the tapes and to the heavy side of the sewn seam section.

5. RESULTS AND DISCUSSION

The following limiting factors in data generation must be considered when reviewing the data. The samples were not assembled in production quality facilities. Marginal sewing equipment, lack of experienced assembly personnel, and isopropyl alcohol cleaning methods may have yielded lower results than would be expected on a production line with a defined and controlled process. In addition, sample sizes were restricted due to adhesive availability, as most of the adhesives were provided as distributor samples. Additionally, the large number of samples may have contributed to some confusion that resulted in some misidentified adhesion test samples, specifically with the Clifton 1013 adhesive. These limitations did not affect the conclusions drawn, and the study provides insight to the use of adhesives in hood assembly.

Table 2. Test Matrix

	One Coat	Two Coats	$\frac{1}{4}$ Tape	1 Tape	$1\frac{1}{2}$ Tape	Biased
3M 1357	X				X	X
3M 1357		X			X	X
Clifton 1013	X				X	X
Clifton 1013		X	X			X
Clifton 1013		X		X		X
Clifton 1013		X			X	X
Clifton 1013		X			X	
Clifton F71040		X			X	X
3M 950	X			X		X
3M 9485	X			X		X
Clemson IV	X			X		
Clemson V	X			X		

The data for the testing are provided in Appendix B. One other factor must be considered in reviewing this data. The adhesion peel strength is also dependent on the butyl rubber's adhesion to the nylon. Because the adhesion requirement for the butyl coating is 4 lb per 2 in. width, some samples could exhibit butyl-rubber separation from the nylon-base fabric before adhesive separation. Although not formally recorded in the test results, evidence of this phenomena was seen during sample assembly. This occurred when samples were improperly assembled and recovery was attempted. In these instances, there was evidence of some of the adhesives pulling the butyl rubber off of the nylon-base fabric.

The 3M 1357 adhesive, used in the development prototypes, consistently provided lower results than the requirement. This adhesive performed well in the M40A1 program Technical Test/User Test. Thus, the requirements were more stringent than what may be required in an operational environment. However, keeping the requirements high does ensure a better quality product, and the Technical Test/User Test did show minimal signs of the tape beginning to peel.

The Clifton 1013, MIL-A-5540,⁵ adhesive performed well in all configurations except that of the two-coat, $1\frac{1}{2}$ -in. biased tape. The problem with this tape was that two coats of adhesive on the tape caused it to curl and begin sticking to itself. As a result, several tapes were ruined, and assembly of those that were not ruined was less than ideal (e.g., wrinkles in the taped seams). At first, this was written off as inexperience of the assemblers. However, later discussions with industry revealed that they

preferred a 1-in. tape such as that used on the M43 mask and the special purpose hood for the M40 mask. The nonbiased tape did not have the same assembly problem. The one-coat, 1½-in. biased tape met the requirements, and the two-coat, 1-in. biased tape consistently had the best results.

The Clifton 1040 adhesive did well except in the cold storage test. This adhesive had the same problem the Clifton 1013 adhesive had when two coats of the adhesive were applied to the tape. Results with a 1-in. biased tape might show an improvement.

The 3M 950, pressure-sensitive, acrylic adhesive, only had one result lower than the requirements. This was a value of 2.94 lb versus a requirement of 3 lb on the initial adhesion requirement. The problem with this adhesive was that the talc had to be thoroughly removed to meet the requirements. This required using transparent tape to remove talc that the alcohol could not remove.

The 3M 9485, pressure-sensitive, acrylic adhesive, was consistently lower than the requirements. This adhesive would not be a good candidate to use on the hood.

Clemson V and Clemson hood samples performed well except in the water immersion test. The tapes tended to separate from the seam. Also, the Clemson V adhesive had the worst results with the blocking test.

Clemson IV adhesive did not perform well except in the heat aging test where the numbers improved substantially. This may indicate that heat curing/sealing may improve the results of this adhesive and, possibly, the other adhesives that Clemson is investigating. It would be interesting to see water immersion results after heat curing/sealing.

The Clemson IV and V adhesives were, by far, the easiest adhesives to use in sample assembly. However, no samples were prepared for the cold crack or hydrostatic resistance tests because the adhesive was delivered with the adhesive already applied to the tape. No adhesive was available for application to 8-in. by 8-in. samples.

Cold crack, blocking, and hydrostatic resistance test results are provided in Appendix C. None of the adhesives had problems with the cold-crack or hydrostatic resistance tests, and only the Clemson V adhesive had problems with the blocking requirement.

6. RECOMMENDATIONS

The following recommendations are provided based on the study conducted:

- Pursue the ultimate goal of permitting the use of any adhesive that meets the hood application by including all of these specific adhesive requirements in the hood assembly purchase description. The basis for this recommendation is that there are other adhesives that could eventually be used

in the hood assemblies that do not conform to MIL-A-5540.⁵ A less specific adhesive requirement could eventually lead to reduced hood costs and improved performance. Prior to adopting this approach, investigators need to conduct further tests to identify adhesives that conform to all of the requirements. The interim solution is to replace the sole source adhesive (3M 1357) with a MIL-A-5540⁵-approved adhesive, a more comprehensive adhesive specification. This should be done until more adhesive candidates can be evaluated and requirements be developed for incorporation into the hood assembly purchase description.

There are several other adhesives that require further evaluation. One adhesive to be considered or investigated further is the Clifton 1040 adhesive with the 1-in. biased tape, which could be an alternative to MIL-A-5540.⁵ In addition, heat curing of the Clemsco adhesives or improved water resistance could improve their performance. Improved application procedures for the 3M 950 adhesive could increase the usefulness of this adhesive.

Based on data established in this study, the following actions have been implemented through engineering change proposals:

- The 3M 1357 adhesive has been replaced with the MIL-A-5540⁵-approved adhesive specification.

- Since procedures are as critical as the adhesive selected, the cold-crack, hydrostatic resistance, and strapping adhesion tests (initial, water immersion, and heat aging, respectively) were added to the purchase description to ensure a quality product.

- The size of the tape for the hoods has been reduced from 1½ in. to 1 in. as this will improve hood producibility and lower hood cost. Taped seams for the M43 mask and other chemical-biological hoods use a 1-in. tape.

- The number of coatings to be applied to the tape and seam will not be specified. One coat could possibly provide adequate performance.

As a result of the assembly experience gained from this study and its data, an optimum configuration would be to use a MIL-A-5540⁵-approved adhesive with a 1-in. biased tape. The tape would have one coat of adhesive, and the seam would have two coats. This should provide improved adhesion and producibility over the previous design.

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LITERATURE CITED

1. Federal Standard for Textile Test Methods, Federal Test Method Standard No. 191, Method 5874, Department of the Army, 20 July 1978, UNCLASSIFIED Federal Standard.
2. Federal Standard for Textile Test Methods, Federal Test Method Standard No. 191, Method 5512, Department of the Army, 20 July 1978, UNCLASSIFIED Federal Standard.
3. Federal Standard for Textile Test Methods, Federal Test Method Standard No. 191, Method 5962, Department of the Army, 20 July 1978, UNCLASSIFIED Federal Standard.
4. Federal Standard for Textile Test Methods, Federal Test Method Standard No. 191, Method 5872, Department of the Army, 20 July 1978, UNCLASSIFIED Federal Standard.
5. Military Specification for Adhesive, Polychloroprene, MIL-A-5540, Department of the Navy, 28 December 1973, UNCLASSIFIED Military Specification.

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APPENDIX A

MANUFACTURER LISTINGS

<u>Company</u>	<u>Address</u>	<u>Phone</u>
Clifton Adhesives	Burgess Place Wayne, NJ 07470	(201) 694-0845
3M Industrial Specialties Div.	3M Center Building 220-7E-01 St. Paul, MN 55144-1000	1-800-227-5085
College of Commerce and Industry School of Textiles Clemson Apparel Research	500 Lebanon Road Pendleton, SC 29670-1957	(803) 646-8454

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APPENDIX B

ADHESION TEST DATA

INITIAL ADHESION (lbs/inch of width)

	ONE COAT 1 1/2" TAPE BIASED	TWO COATS 1 1/2" TAPE BIASED	TWO COATS 3/4" TAPE BIASED	TWO COATS 1" TAPE BIASED	TWO COATS 1 1/2" TAPE NON-BIASED	ONE COAT 1" TAPE
3M 1357	1.33 1.553 3.48	1.167 1.687 2.093				
CLIFTON 1013	4.067 4.973 6.3	12.93 2.667 4.50	3.32 3.267 15.60	10 12.54 10.53	7.073 9.633 10.687	
CLIFTON 1040		10.42 10.0 7.34				
3M 950						2.94 3.55 3.41
3M 9485						2.14 2.5 2.40 2.19
CLEMSON IV						2.23 2.54 2.36 3.08
CLEMSON V						7.48 7.49 7.17 7.41 7.40 7.72
CLEMSON HOOD						5.99 7.06 6.36 7.81 5.46

WATER IMMERSION ADHESION (lbs/inch of width)

	ONE COAT 1 1/2" TAPE BIASED	TWO COATS 1 1/2" TAPE BIASED	TWO COATS 3/4" TAPE BIASED	TWO COATS 1" TAPE BIASED	TWO COATS 1 1/2" TAPE NON-BIASED	ONE COAT 1" TAPE
3M 1357	1.698 2.315 2.796 2.492	3.473 2.796 4.503				
CLIFTON 1013	2.484 2.747 2.615	7.587 2.48 3.547 4.809	2.19 2.178 13.653	5.854 4.383 4.466	4.494 3.415	
CLIFTON 1040		1.204 7.727 4.737 5.41 8.827				
3M 950						3.531 3.892 3.691
3M 9485						1.812 3.186 2.856 2.412
CLEMSON IV						.0984 .4506 .4381 2 NO TESTS*
CLEMSON V						.5067 .5684 .3496 .3148 2 NO TESTS*
CLEMSON HOOD						.9239 1.192 .318 3 NO TESTS*

* NO TESTS WERE THE RESULT OF TAPE SEPARATING FROM SEAM DURING BOILING.

HEAT AGING ADHESION (lbs/inch of width)

	ONE COAT 1 1/2" TAPE BIASED	TWO COATS 1 1/2" TAPE BIASED	TWO COATS 3/4" TAPE BIASED	TWO COATS 1" TAPE BIASED	TWO COATS 1 1/2" TAPE NON-BIASED	ONE COAT 1" TAPE
3M 1357	2.86 2.46 2.027 2.14	2.01 2.38 2.513 3.527				
CLIFTON 1013	3.927 4.653 3.693 3.613	2.532 5.157 2.129 2.905 1.824	4.991	6.636 5.707	4.493	
CLIFTON 1040		4.0 9.74 2.5 2.573 7.993				
3M 950						3.77 3.90 3.45 3.45
3M 9485						2.27 2.18 2.35 2.4
CLEMSON IV						4.99 6.37 5.29 7.08 5.58
CLEMSON V						8.36 6.20 7.35 6.22 6.88 6.60
CLEMSON HOOD						8.33 9.22 8.66 8.54 8.15 7.94

COLD STORAGE ADHESION (lbs/inch of width)

	ONE COAT 1 1/2" TAPE BIASED	TWO COATS 1 1/2" TAPE BIASED	TWO COATS 3/4" TAPE BIASED	TWO COATS 1" TAPE BIASED	TWO COATS 1 1/2" TAPE NON-BIASED	ONE COAT 1" TAPE
3M 1357	1.211 2.822	1.397 2.543				
CLIFTON 1013	4.785 3.207	5.171 5.105	3.144 3.101	10.56 9.088	6.561 6.613	
CLIFTON 1040		1.363 1.459				
3M 950						3.434 3.669
3M 9485						2.134 2.702 2.065
CLEMSON IV						2.276 2.833
CLEMSON V						6.282 7.051
CLEMSON HOOD						5.468 4.992

APPENDIX C

COLD CRACK, BLOCKING, AND HYDROSTATIC RESISTANCE TEST DATA

Adhesives
M40 Mask - Quick Doff Hood
Producibility Study

LAB REPORT NUMBER 7138
3 Sep 93

Blocking

1. 3M9485 - slight
2. 1040 - 2 coat seam - 1 coat tape - slight
3. 1040 HV - 2 coat seam - 1 coat tape - slight
4. 3M9485 - slight
5. 3M9485 - slight
6. 1040 - 2 coat seam - 1 coat tape - moderate
7. Clemson 55 V - moderate
8. Clemson 55 V - moderate
9. Clemson 55 V - moderate
10. 1013 - 2 coat - moderate
11. 1357 - 2 coat - slight
12. 1357 - 2 coat - slight
13. 1357 - 2 coat - slight
14. Clemson 55 IV - none
15. Clemson 55 IV - none
16. FA1013 - 1 coat tape - 2 coat seam - slight
17. Clemson 55 IV - none
18. 1013 - 1 coat - slight
19. 1013 - 1 coat - slight
20. 1357 - 1 coat - slight
21. 1357 - 1 coat - slight

NOTE: The amount of tackiness was directly related to the amount of glue spread outside the tape width. Where there was no blocking, there was no uncovered glue. All tackiness was glue to glue.

Adhesives
M40 Mask - Quick Doff Hood
Producibility Study

LAB REPORT NUMBER 7138
3 Sep 93

Hydrostatic Pressure - Requirement - not less than 90% of control

- Control
1. 100 psi
2. 102 psi
3. 108 psi

- FA 1040 coated
1. 106 psi
2. 106 psi
3. 103 psi

- FA 1013 coated
1. 107 psi
2. 108 psi
3. 107 psi

- 1013 coated (cold crack)
1. 110 psi
2. 108 psi
3. 109 psi
4. 110 psi
5. 107 psi
6. 110 psi

- 3M 950 coated (cold crack)
1. 111 psi
2. 104 psi
3. 110 psi

- FA 1013 coated (cold crack)
1. 106 psi
2. 107 psi
3. 101 psi

- FA 1040 coated (cold crack)
1. 108 psi
2. 109 psi
3. 108 psi

- 3M 9485 coated (cold crack)
1. 113 psi
2. 109 psi
3. 108 psi