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17 May 1943 NRL Report No. P-[illegible]5

NAVY DEPARTMENT

Report on

The Resistance of Impermeable Materials to Penetration by Liquid Vesicant Agents

NAVAL RESEARCH LABORATORY
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WASHINGTON, D. C.

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ABSTRACT

This is a progress report of the study of the liquid vesicant resistance of impermeable materials, particularly coated fabrics. The test methods used and the conditions affecting the vesicant resistance are described. Special attention has been given to the evaluation of several types of coatings. Butyl rubber was found to be the most resistant satisfactory coating material. It is also recommended that alkyd resin type coatings be considered for fabrics used for cockpit and engine covers. These data have been assembled for the information of the Bureau.

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AUTHORIZATION

1. This work was authorized under Project 547/41, "Maintenance, Bureau of Ships," dated 16 December 1940. Problems requiring work utilizing the methods and procedures described in this report were received in Bureau of Ships letter C-F38-2(3688), "Protection Covers for Prevention of War-Gas Contamination," dated 17 October 1942; Bureau of Ships letter C-S77-2-(2)(3688)(350), "Individual Protective Co for Defense against Vesicant Spray," dated 8 February 1943; and Bureau of Ships letter C-S77-2-(1)(688), "Navy Gas Masks Mustard Resistance Test," dated 29 March 1943.

STATEMENT OF PROBLEM

2. This study was undertaken to determine the resist ance of various materials to penetration by liquid vesicant agents. This required the development of test methods and a study of the factors which affect the penetration of material by liquid vesicants.

KNOWN FACTS BEARING ON THE PROBLEM

3. The British developed the "Mirror Test" for liqui vesicant penetration tests which is described in Porton 580 (R.7177). Penetration by liquid mustard is tested by the Chemical Warfare Service as described in C.W.S. Directive No. 113, "Liquid Mustard Test on Impermeable Materials." A large number of coated fabrics have been examined at the C.W. Development Laboratory, Cambridge, Massachusetts. This group has cooperated with the Naval Research Laboratory, offering suggestions in regard to preferable types of coating materials, and furnishing several samples of coated fabric.

THEORETICAL CONSIDERATIONS

4. Materials to be resistant to penetration by vesicant agents must present a continuous film of a substance which does not dissolve the agent. Both of these factors are necessary. If the surface is not continuous, the liquid agent will seep through the pores to the other side. If the surface of the material is continuous and does not dissolve the agent, a very thin film can resist penetration by the agent indefinitely. Thin films of cellophane or PVA, for example, have been demonstrated to resist penetration by liquid mustard for more than 40 hours.

5. However, if the material has a continuous surface but will dissolve the vesicant, penetration will occur more

[redacted]

or less rapidly. For example, ordinary natural rubber compositions allow penetration by liquid mustard after 100 to 150 minutes per millimeter of thickness of rubber. On the other hand, sheets of butyl rubber of corresponding thickness were not penetrated by liquid mustard after ten days' exposure.

PREVIOUS WORK DONE AT THIS LABORATORY

6. Part of the data contained in this report has been transmitted from time to time directly to the Bureau.

7. A Memorandum to the Director, "Recoating Pantasot Fabric to Improve the Resistance to Mustard Gas" dated 16 Mar 1943, contains a discussion of the attempts made to improve t mustard resistance of airplane cockpit cover fabric now on hand. This includes a discussion of the work done with an ai drying paint which can be applied to cockpit covers and simil materials in the field. Also included are the attempts made to apply a factory coating to Pantasote to make it mustard resistant. This information for the most part has not been included in this report.

EXPERIMENTAL PART I. METHODS AND PROCEDURES

A. General Considerations

8. The general method of testing for penetration of materials by liquid HS, M-1 and 1130 is the same in principle as the British "Mirror Test" for vesicant penetration described in Porton 580 (R.7177). Several changes in the procedure were made during the course of this investigation. As a consequence of a study of the effect of temperature on the penetration by liquid HS, all determinations are now made a constant temperature box at 30° ± 0.5°C. The humidity of the atmosphere during the storage of materials previous to the penetration test was found to be an important factor. As a result of this observation all samples are kept at 75°F and 50% relative humidity before the penetration tests are made for comparative purposes.

9. The procedure is essentially the same for all three vesicants except that a different test paper is used in each case. The test paper for mustard is a modified form of the S.D. (Spotted Dick) paper described in Porton Report No. 2264A. In this same report is described the DT (diphenyl thiocarbazone) paper used for M-1. The 1130 detector paper is the DB3 paper as developed at this Laboratory. For HS and 1130 a sodium carbonate filter paper is used between the detector paper and the material being tested, but no carbonate paper is used with the DT paper for M-1. The details of the preparation of these test papers are given in Appendix A.

[redacted]

B. Test Procedure

10. The material to be tested is conditioned for at least 24 hours at 75°F and 50% relative humidity. Three 3x3-inch squares are cut from the sample of material. The arrangement of the test assembly is given in Plate I. On a 2.5-inch square of plate glass (A), 0.375 inch thick, are placed successively the detector paper (B), the carbonate filter paper (C) if one is to be used, the test specimen (D), and the brass washer (E). The brass washer is 0.125 inch thick, 0.75 inch inside diameter and 2.0 inches outside diameter. A drop of the vesicant (ca. 0.025 g.) is placed on the fabric in the center of the washer, and another square of plate glass (F) is placed over this to keep the various parts in place and to decrease the rate of evaporation of the vesicant. The entire set-up is placed in a box maintained at 30°C. The test paper is observed by means of a mirror set at an angle below the box. The average time elapsing until the first appearance of a positive test is recorded as the penetr tion time (HPT).

C. Factors Affecting the Liquid Vesicant Penetration Time

(1) Temperature of the Test.

11. Samples of six different fabrics which were stored at 75°F and 50% relative humidity were tested for penetration by liquid mustard at three different temperatures 25°C, 30°C and 35°C. The other conditions of the test were held constant. The results of these tests are given in Table I. Each value given for the liquid mustard penetration time (HPT) is the average of three determinations.

Table I

Liquid Mustard Penetrations at Different Temperatures

NRL #	Sample	*HPT, Minutes			HPT Rat
		25°C	30°C	35°C	25°C/35
4-7	Stedfast CZ	305	180	120	2.5
8-10	Congoleum-Nairn WSOD	380	280	180	2.1
9-2	Nylon on cotton duck	460	315	195	2.4
9-3	Alkyd on bandolier cloth	64	43	34	1.9
4-18	U.S. Rubber L-8734	305	195	125	2.4
8-17	Jones-Dabney cloth	165	120	75	2.2

*HPT denotes liquid mustard penetration time

[redacted]

12. The values given in the last column of Table I were obtained by taking the ratio of the HPT values at 25°C and 35°C. This ratio varies from 1.9 to 2.5, which means that mustard penetration will occur at 35°C about twice as fast as at 25°C. From these data it is evident that the temperature of the test is an important factor. To get comparative HPT values for different materials it is necessary to make the penetration tests at controlled temperatures. 30°C was chosen as the standard test temperature because it is more easily maintained than lower temperatures, especially during the summer months.

(2) Storage Conditions Previous to the Penetration Test.

13. Samples of seven different fabric materials were stored under several different conditions. Those marked (CT) were stored in a constant temperature room at 75°F and 50% relative humidity. The (RT) samples were kept in the laboratory at a temperature of approximately 85°F and a relative humidity of 80-90%. Those marked (Des.) were kept at 75°F in a desiccator containing Anhydrone. In each case the fabrics were stored for at least 24 hours under these respective conditions before the tests were made. The HPT values given in Table II are the average of three determinations made at 35°C.

Table II

Mustard Penetrations of Fabrics Stored under Different Conditions

		HPT, Min. after storage at		
NRL #	Sample	CT	RT	Des.
4-7	Stedfast CZ	120	150	93
8-10	Congoleum WSOD	180	150	150
9-2	Nylon on cotton duck	195	90	190
4-18	U.S. Rubber L-8734	125	120	90
8-17	Jones-Dabney cloth	75	60	
8-13	Acme T-102-B-132	>475	220	
9-3	Alkyd on bandolier cloth	34	31	

14. Although the effect of humidity varied with each fabric, it is evident that the mustard resistance of the fabrics is affected by its previous treatment. Consequently, subsequent tests were made on the materials after storage for at least 24 hours at 75°F and 50% relative humidity for purposes of comparison. A given material should be examined over a wide range of conditions of temperature and relative humidity for complete evaluation.

[redacted]

(3) Amount of Vesicant Used.

15. Various coated fabrics were tested for penetration by liquid vesicants, using different amounts of vesicant. For example, HPT values were obtained using 1, 3 and 5 drops of mustard. Each drop weighed approximately 0.021 grams. The same procedure was used for M-1 and 1130, the weight of each drop being about 0.023 g. and 0.025 g. respectively. The data obtained in these tests are given in Table III. Each value given is the average of three determinations.

Table III

Liquid Vesicant Penetration Times Using Different Amounts of Vesicant

Agent	Sample	Penetration Time, Min.		
		1 drop	3 drops	5 drops
HS	Pantasote	16	12	12
"	Alkyd on bandolier	30	28	28
"	Alkyd on rayon	138	148	171
"	Fairprene 5033	50	55	56
"	Pyroxylin 25-243	31	31	34
M-1	Pantasote	< 2	< 2	< 2
"	Alkyd on bandolier	17	17	17
"	Alkyd on rayon	103	123	108
"	Pyroxylin 25-243	20	21	21
"	Newmarket J-8528	60	25	28
1130	Pantasote	11	10	8
"	Alkyd on bandolier	28	30	23
"	Fairprene 5033	67	59	63
"	25-243	42	41	37
"	Hodgeman Saflex	51	49	47

16. Examination of the data in Table III indicates that in general the penetration time of the vesicants through such materials is independent of the amount of vesicant used within the limits of the experiment. In one case, the penetration of Newmarket J-8528 by M-1, a marked drop in penetration time is observed for three and five drops of vesicant as compared to one drop. It was noted, while performing the test, that the M-1 spread rapidly on the surface of the Newmarket fabric. The fabric was heavy and may have absorbed most of the M-1 in one drop, prolonging the time required for penetration. In none of the other cases is there any significant decrease in penetration time with increase in the amount of vesicant used.

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EXPERIMENTAL PART II. DATA AND RESULTS.

A. Penetration of Materials by Liquid Mustard

17. In the course of the study of several problems at this Laboratory a large number of materials was examined for resistance to penetration by liquid mustard. Most of the materials tested may be classified under four headings: paper films, rubbers and synthetics, and coated fabrics. These dat are contained in Appendix B so that comparisons of the mustar resistance of various materials might be facilitated.

(1) Coated and Treated Papers.

18. Table I of Appendix B gives the mustard penetration times (HPT) of coated and treated papers. Untreated papers will allow penetration by liquid HS in less than one minute. Most of the papers tested were coated with material containing "Mazein," a protein prepared from corn by the Corn Products Refining Company. Many of the papers coated with this material have good mustard resistance. The exact composition of many of the coating was not obtained. At least one of the papers, Dixie Wax Paper Company # 2V018, was coated with a paraffin mixture which was also very resistant to mustard.

(2) Thin Films.

19. Table II of Appendix B contains the HPT values for thin films of various materials. In almost every case, cellophane, PVA, and nylon gave good resistance to penetratio by liquid HS.

(3) Rubber and Synthetic Materials.

20. Table III of Appendix B contains the mustard pene tration values of rubber materials. The thickness of each sample in millimeters is recorded also. The last column lists the penetration time in minutes per millimeter calculat from the above data. Cellulose acetate, butyl rubber, nylon on neoprene and chemigum with nylon interface were extremely resistant to penetration by liquid mustard. The latter two materials may owe their resistivity to the nylon film present Saflex, butvar and acryloid coated neoprene were more resista than the ordinary rubber sample. Thiokol, neoprene and vinyl were less resistant than rubber.

(4) Coated Fabrics.

21. Table IV of Appendix B contains the penetration time for a large number of coated fabrics. No attempt is made to discuss all of these materials. In a later section a group of these fabrics is discussed in more detail.

[redacted]

B. Comparison of Penetration of Impermeable Materials by the Liquid Vesicants, HS, M-1 and 1130.

22. From time to time a number of materials were teste for penetration by M-1 and 1130 in addition to HS. It was fel that a compilation of this information might be of value in choosing materials for protection against these vesicants. The penetration time in minutes for each vesicant for the mate rials tested is given in Tables IV, V and VI. Each value in the tables is an average of three determinations. Table IV contains the data for films and papers, Table V the data for synthetic rubber-like materials, and Table VI the data for coated fabrics.

23. A number of materials were not penetrated by any of the vesicants during the time of the test. Some thin films of material such as PVA and cellophane, waxed papers, and gelatin impregnated fabrics resist penetration by the vesicants indefinitely. CR-39 laminated cloth was not penetrated by HS and 1130 and doubtless would not allow penetration by M-1.

24. On the other hand, some materials such as vinylite rubber from rubber gloves and some lightweight fabrics, such a the thiokol-coated fabric, offered little resistance to any of the vesicants. In the case of certain fabrics, this is due to the lack of continuity of the coating. In other cases, such as vinylite and rubber, this lack of resistance is a characteristic of the material itself.

25. In general, M-1 penetrates various coated fabrics and other materials more quickly than do 1130 and HS. An extreme example of this is the penetration of nylon film and nylon-coated duck by M-1 within a few minutes. Nylon gives good protection against HS and 1130. Polyvinyl butyral coated fabrics have higher resistance to M-1 than to the other two vesicants. Butyl rubber coated fabrics also have good resistance to M-1.

26. Generally, 1130 penetrates fabrics in about the same time as does HS. There are some exceptions. Ordinary cellophane has poor resistance to 1130, but the laminated cellophane has good resistance. This laminated cellophane is the same type of material as used in the cellophane individual protective covers.

[redacted]

Table IV

Penetration of Films and Papers by Liquid Vesicants

NRL #	Description	Penetration Time (Min.) for HS	1130	M-[illegible]
2-14	Nylon film	>180	>120	[illegible]
9-2	Nylon film laminated on cotton duck	320	225	1[illegible]
8-19	Sylvania cellophane - clear	> 200	90	>20[illegible]
2-19	PVA film, K-2	>2400	>120	
2-11	Cellophane PA, water-soluble	960	>120	> 24[illegible]
2-18	PVA film, K-1	>2400	>120	>138[illegible]
10-1	Cellophane MSAT laminated to duck	>1200	>1500	> 24[illegible]
10-2	Cellophane MSAT laminated to duck	>1200	> 1500	>24[illegible]
13-6	Laminated cellophane, clear	> 300	> 300	>30[illegible]
13-7	Laminated cellophane, olive drab	> 300	> 300	> 30[illegible]
5-16	Riegel 50# MPHS paper	> 240	> 300	> 42[illegible]
5-5	Dixie Wax Paper #4M	>1280	> 120	>24[illegible]

Table V

Penetration of Rubbers and Synthetic Materials

NRL #	Description	Penetration Time (Min.) for HS	1130	M-[illegible]
10-19	Vinylite X	96	92	11[illegible]
11-1	Cellulose acetate, 0.015" thick	> 1200	>1200	27[illegible]
4-16	Vinylite, white	4	4	[illegible]
4-17	Vinylite, green	8	8	[illegible]
	Rubber from gloves	12	18	1[illegible]
	Facepiece rubber, NC Mark I gas mask	192	275	> 25[illegible]

[redacted]

Table VI

Penetration of Coated Fabrics by Liquid Vesicants

NRL #	Description	Penetration Time (Min.) fo		
		HS	1130	M-
10-5	Cloth impregnated with CR-39	>1200	> 1200	
10-18	Gelatin-laminated fabric	>1200	> 1200	> 10
10-20	Nitrocellulose-castor oil coated cloth	14	20	
9-16	Pantasote M729	27	17	
10-6	Pyroxylin-coated fabric	26	32	
10-7	Pyroxylin-coated fabric	48	29	
9-11	Thiokol coated fabric	10	14	<
11-6	Urea formaldehyde coated fabric	54	58	
11-7	Urea formaldehyde coated fabric	56	50	
9-13	Butyral coated duck	47	45	> 1
9-6	Butyral coated duck	80	59	> 4
12-12	Butyral coated duck	85		> 4
9-10	Saflex coated cloth	15	20	
10-12	Alkyd coated fabric, rayon	132	126	
9-3	Alkyd coated fabric, cotton	43	51	
9-12	Alkyd coated fabric, rayon	131	201	
	Pantasote coated with alkyd paint	84	28	
7-18	Vistanex-neoprene impregnated fabric	172	77	
4-7	Vistanex-neoprene impregnated fabric	212	84	
8-10	Congoleum-Nairn WSOD	298	> 256	
11-9	Cellulose acetate coated fabric	23	35	
11-12	Butyl rubber coated fabric	13	5	<
11-13	Butyl rubber coated fabric	56	26	
12-13	Butyl rubber coated fabric	150	90	2
13-4	Butyl rubber coated fabric	290	360	> 6

[redacted]

C. Fabrics Examined for Cockpit Covers.

(1) Introduction.

27. A large number of fabrics were examined in an attempt to find materials which would be suitable for cockpit and engine covers. These materials were desired to conform to Navy Aeronautical Specification 24D4C, and at the same time have good resistance to mustard gas. Most of the fabrics tested are listed in Table IV, Appendix B. Many of them obviously would not qualify according to Spec. 24D4C but were tested for resistance to mustard in an attempt to learn what types of coatings would be of value. Many of them had very poor resistance to liquid mustard and will not be discussed in this report. Table VII lists the fabrics of particular interest.

28. Although particular attention was given to the study of the mustard resistance of these candidates, the other properties required in a suitable fabric were not ignored. For example, the balance between the minimum tensile strength and maximum weight of the finished product is of interest and importance. The minimum weight of base fabric which will give the required tensile strength is between 14 and 15 ounces per square yard. Since the maximum allowable weight of the coated fabric is 22.0 ounces per square yard, not more than about seven ounces per square yard of coating is allowable. For coatings such as nylon, cellophane and butyl rubber which are extremely impervious to penetration by mustard, this weight can be maintained easily. For materials which dissolve mustard more or less rapidly, such as polyvinyl butyral, it may be difficult to get a fabric of satisfactory weight having sufficient mustard resistance.

29. Another important consideration is the flexibility requirement, especially at -30°F. Many materials such as polyvinyl butyral become stiff and are apt to crack at low temperatures although the flexibility at 70°F and 150°F is satisfactory. Materials like butyl rubber have good flexibility at all temperatures. Where heavy coatings are necessary for mustard protection, the flexibility requirement is likely to be difficult to meet. Alkyd coated fabrics are an example of this situation. In order to build a coating on heavy duck which will be continuous and thick enough to give satisfactory mustard resistance, the net weight of coating will necessarily be near the maximum. To obtain flexibility, modified alkyds are necessary, but unfortunately their addition tends to decrease the mustard resistance.

[redacted]

(2) Examination of Fabrics.

30. Table VII lists the fabrics which will be discusse in more detail. The first three were representative of materials which had been purchased under Specification 24D4C. The Pantasote fabric had a HPT of 25 minutes which was the best of the three. However, it was found that under one month of mild weathering the HPT of Pantasote drops to about two minutes. Consequently the mustard resistance of all three fabrics may be considered to be quite low.

31. The mazein impregnated fabrics were stiff and attacked by water. Therefore, in spite of their extremely high mustard resistance, there was little possibility that a satisfactory cockpit fabric would result from the use of mazein as a coating material. The gelatin impregnated fabric became brittle and cracked easily after immersion in water at room temperature for several hours. The CR-39 fabric was also very stiff and representatives of Columbia Chemical Division of Pittsburgh Plate Glass Company offered no hope that CR-39 could be plasticized to make it more flexible.

32. Nylon film was found to be extremely resistant to penetration by HS. For this reason the Fabrikoid Division of E. I. DuPont de Nemours & Company prepared several samples of cotton fabrics with a laminated nylon film. Most of these laminated fabrics had high HPT values, but all suffered from the fault that the nylon film was too easily damaged by abrasion. Since these fabrics owed their resistance to mustard to the nylon film, this advantage was lost when the nylon was torn. The cellophane laminated fabrics were also unsatisfactory because of low resistance to abrasion. The adhesion of the cellophane film to the base fabric was not as good as the adhesion of the nylon film.

33. The Stedfast Rubber Company sample #CZ is a two-ply fabric which is impregnated with vistanex-neoprene. It was submitted to NAF (Naval Aircraft Factory, Philadelphia, Pennsylvania) and was rejected because its tensile strength and tear resistance were low, the coating was basic to litmus and gasoline caused bubbles to appear in the coating. Sample #CY likewise contained a neoprene-vistanex mixture. NAF tests showed that its flexibility was above the allowable maximum and its weight (33.8 oz./yd.^{2}) was far above the 22.0 oz./yd.^{2} limit. Stedfast #DA was not tested further because of its extreme weight, 36.8 oz./yd.^{2}

34. The U.S. Rubber Company sample L-8734 is also a vistanex-neoprene impregnated fabric. It had a mustard resistance of more than three hours but NAF tests showed that the tear resistance and tensile strength were low.

[redacted]

Table VII

Liquid Mustard Penetration of Coated Fabrics

NRL #	Description	Weight oz./yd.^{2}	HPT Minutes
1-1	Valentine cockpit fabric		12
1-2	Pantasote cockpit fabric		25
1-3	Unidentified cockpit fabric		10
5-4	Laminated mazein, 2 cloth layers		>1080
7-15	Mazein on Arnzen		> 240
10-18	Gelatin-laminated fabric		>1200
10-5	CR-39 laminated duck and sheeting	34.8	>1200
7-16	Nylon on duck, NCR 7684-SS-819		> 960
8-9	Nylon on duck, VCR 7733-SS-828		93
9-2	Nylon on duck	21.0	320
9-7	#538, nylon on duck	29.9	3120
9-8	#537, nylon on duck	21.5	615
10-1	Cellophane MSAT laminated on Pantasote, one side	19.8	>1200
10-2	Cellophane MSAT on Pantasote, two sides	20.9	> 1200
10-3	Cellophane CA48 laminated on Pantasote, one side	19.9	36
10-4	Cellophane CA48 laminated on Pantasote, two sides	20.6	98
4-7	Stedfast CZ, vistanex-neoprene impregnated	23.4	180
4-6	Stedfast CY	33.8	92
4-8	Stedfast DA	36.8	> 360
4-18	U. S. Rubber L-8734	17.4	193
8-10	Congoleum-Nairn WSOD	17.6	150
10-6	Pyroxylin-coated cotton		26
10-7	Pyroxylin-coated cotton		48
9-6	Butyral-coated duck, cotton	34.2	80
12-12	Polyvinyl butyral-coated rayon	25.0	85
9-13	Laminated butvar		47
9-3	Alkyd-coated bandolier sheeting	9.4	43
9-12	Alkyd-coated rayon	14.2	71
8-17	Devoe-Reynolds gasproof cloth, alkyd coating		120
11-4	Alkyd-coated cotton, O2I-1	24.0	236

[redacted]

(Cont'd.)

Table VII (Cont'd.)

NRL #	Description	Weight oz./$yd.^2$	HPT Minutes
12-6	Alkyd-coated rayon , 02I-9	19.2	112
12-7	Alkyd-coated cotton, 02I-8	25.5	181
8-13	Laminated alkyd fabric T-102-B		>1200
11-12	Butyl rubber-coated cotton		13
11-13	Butyl rubber-coated rayon		56
12-3	Butyl rubber-coated rayon	14.1	150
13-4	Butyl rubber-coated rayon	21.6	290

[redacted]

Congoleum-Nairn sample WSOD is a laminated material with mustard resistance of 150 minutes. NAF tests showed that the tear resistance was low, the coating was acid, the sewability of the fabric was poor and it lacked flexibility at all test temperatures.

35. The two samples of cotton cloth with pyroxylin coating were obtained from the CWS Development Laboratory, Cambridge, Massachusetts. They were materials purchased under C.W.S. Specification No. 197-51-171. This specification requires a minimum of 100 minutes resistance to liquid mustard as tested under C.W.S. Directive No. 113. When tested by the NRL method, these fabrics did not have good mustard resistance. These fabrics were lightweight and not suitable for the heavy duty required of Navy cockpit covers. Pyroxylin coatings have fairly good mustard resistance but this resistance goes down markedly when plasticizers are added to give flexibility to the fabric.

36. Fabrics coated with polyvinyl butyral were suggested by the C.W.S. Development Laboratory. It was cautioned by C.W.S. that such coatings were found to stiffen when cold. Several samples of such materials prepared by the Hodgeman Rubber Company were examined. One of the samples (9-6) had only 80 minutes' protection against mustard although it had an extremely heavy coating (total weight 34.2 oz./yd.^{2}). A second sample (12-12) had mustard resistance of 85 minutes. It also was overweight (25.0 oz./yd.^{2}) and NAF tests showed that the adhesion of the coating to the base fabric was poor and the coating split open at -30°F. Another sample of fabric (9-13) was laminated with polyvinyl butyral by Hodgeman Rubber Company, but the mustard resistance was low (47 minutes).

37. From several sources it was learned that coated fabrics employing an alkyd resin material had been found to give good mustard resistance. The unmodified alkyd resins are mustard resistant, but are hard and brittle and require the addition of modified alkyds to increase the flexibility. This addition of plasticizing alkyd resins tends to decrease the mustard resistance of the alkyd coating. Several compani which had prepared alkyd-coated fabrics were requested to attempt the preparation of a fabric to conform to the require ments of Specification 24D4C.

38. Two samples were obtained from the CWS Developmen Laboratory. The alkyd coated bandolier sheeting (9-3) was a lightweight fabric (9.4 oz./yd.^{2}). The alkyd-coated rayon material (9-12) had an initial mustard resistance of 71 minut which gradually increased to more than 120 minutes after thre months. The tensile strength of this material was low after

[redacted]

immersion in water. The Devoe-Reynolds "Gas-proof cloth" was a lightweight alkyd-coated cotton fabric. Congoleum Nairn, Inc., samples 02I-1, 02I-8 and 02I-9 were alkyd-coated materials having HPT values greater than 100 minutes. They appeared to be good possibilities and were sent to NAF for additional tests. It was found that all three samples cracked badly when tested at -30°F in accordance with the method of paragraph F-4j of Navy Aeronautical Specification 24D4C. No further tests were run on these materials.

39. The Acme Protection Equipment Company sample T-102-B (8-13) was a laminated fabric with an alkyd sandwich material. This fabric was extremely resistant to mustard penetration and a sample was sent to NAF. The resistance to tear was low, the flexibility poor and the water resistance before and after abrasion was unsatisfactory. American Cyanamid Company submitted several samples but the coating was faulty and further attempts are in progress to supply a satisfactory fabric.

40. Several samples of fabrics coated with butyl rubber have been tested for mustard resistance. Four of these prepared by the Stedfast Rubber Company and received through NAF show the possibilities of this material. Two of these, one on cotton and the other on rayon base fabric, had HPT values of 13 and 56 minutes, respectively. Two other butyl rubber-coated rayon fabrics had 150 and 290 minutes' protection against mustard. The latter one was found by NAF to pass all the requirements of 24D4C also. This fabric is the best material examined thus far as a mustard resistant cockpit cover fabric.

(3) Discussion.

41. With the exception of the butyl rubber-coated rayon (13-4), none of the fabrics which have passed all the qualifying tests at NAF would meet any but an extremely low mustard resistance requirement. However, it is unlikely that butyl rubber will be available for coating fabrics. For this reason the next best material should be considered. The next best material seems to be alkyd-coated fabric. Such fabrics have been prepared with more than 100 minutes' mustard protection, but none has been shown to have satisfactory low temperature flexibility when tested according to the method of paragraph F-2e of Navy Aeronautical Specification 24D4C.

42. If alkyd coated fabrics are to be considered for cockpit fabrics some compromise seems to be necessary. Perha the temperature of the flexibility test could be raised from -30°F. It may be valid to relax this requirement because vesicants will be unlikely to be troublesome in Arctic regio

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The damage from vesicants will be much worse at ordinary or tropical temperatures, and at such temperatures alkyd coated fabrics are flexible enough. The present cockpit fabric materials might be used in cold regions, and an alkyd coated fabric be used in temperate and tropical zones, especially in view of the fact that a separate specification has been set up for the HS resistant fabrics.

(4) Conclusions.

43. The materials now being used to make cockpit covers have low resistance to liquid mustard. Pantasote fabric is the best of these but its mustard resistance is soon lost on outdoor weathering.

44. Of all the fabrics tested for use as cockpit covers the butyl rubber coated rayon is the best. It has good mustard resistance (290 minutes) and meets all the other qualifying tests for cockpit fabrics under Navy Aeronautical Specification 24D4C.

45. The next best materials appear to be alkyd coated fabrics. The difficulty with this coating material is that it tends to crack at -30°F.

46. Lamination of thin films of materials such as nylon and cellophane to heavy fabrics is impractical. The film is too easily torn, and as soon as torn its coating value is gone.

47. Polyvinyl butyral coatings thus far have not been found to very mustard resistant. The low temperature properties are poor also.

(5) Recommendations.

48. Due to the unavailability of butyl rubber, it is recommended that alkyd coated fabrics be considered for use as cockpit covers. It may be necessary to amend the present specifications to allow for the low temperature limitations of an alkyd coated fabric.

49. Larger experimental runs of alkyd-coated fabrics should be made so that the possibilities of this material might be examined under plant conditions.

DISCUSSION OF RESULTS

50. A test procedure has been set up for the determination of the liquid vesicant penetration time when HS, 1130 and M-1 are used.

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51. It was observed that the previous storage conditions of material and the temperature of the penetration test have a great effect on the vesicant penetration time. No significant difference in time of penetration was observed when the amount of vesicant used for each test was varied between about 20 mg. and 100 mg.

52. It was found that a number of materials are not penetrated by liquid vesicants even after a number of hours' exposure. Such materials as cellophane, nylon, PVA, certain waxed papers, gelatin-impregnated fabrics and mazein-impregnat fabrics and papers come in this class.

53. With some exceptions, it was found that M-1 penetrates coated fabrics and other materials generally faster than does HS. The penetration time for HS and 1130 is generally about the same.

54. A large number of coated fabrics were examined for possible use as cockpit cover material. Butyl rubber was found to be the best coating material of those tested. Alkyd resin coatings were next best.

CONCLUSIONS AND RECOMMENDATIONS

55. The best material for coating cockpit cover fabrics is butyl rubber.

56. Because butyl rubber may not be available, it is recommended that alkyd coated fabrics be considered for use as cockpit cover material. This may require revision of existing specifications or preparation of a separate specification for a mustard-resistant fabric.

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APPENDIX A

Preparation of Detector Papers

1. S.D. - 328 paper for HS. Whatman #44 filter paper is dipped into a 0.03% aqueous solution of Congo Red and allowed to dry. This Congo Red paper may be kept indefinitely. On the day the paper is to be used, it is spotted with a fresh 5% solution of S-328 in tetrachloroethane. A stencil is made by making a number of small holes in a square of cellophane with the point of a pin. The stencil is laid on the Congo Red paper with the projecting sides of the holes downward. Then a cotton swab moistened with the S-328 solution is rubbed over the cellophane wetting the Congo Red paper in spots. As soon as dry, this paper is ready for use. The sensitivity of the S-328 paper to HS decreases slowly from day to day after it is prepared; consequently it should be made fresh each day. The appearance of blue spots on the red paper is a positive test for HS.

2. DT paper for M-1. Whatman #44 filter paper is dipped into a 0.1% solution of diphenylthiocarbazone (dithizone) in chloroform and allowed to dry. While wet, the paper is colored green, but upon drying, it turns dark gray. If kept in a closed box out of the light, the paper does not lose its sensitivity very rapidly and may be kept for several weeks. In use, no filter paper is placed between the fabric and the test paper. The appearance of a red or pink color is recorded as a positive test.

3. DB3 paper for 1130. Whatman #44 filter paper is dipped into a 10% aqueous solution of sodium carbonate and allowed to dry. This carbonate paper may be kept indefinitely. Shortly before the test is made, the carbonate paper is spotted with the DB3 solution in a manner similar to that described in spotting the S.D.-328 paper. The solution is made by dissolving 0.4 g. of pure DB-3 in 10 ml. of acetone. As soon as dry, the paper is ready for use. The paper is white before use, and the appearance of blue spots indicates a positive test.

4. Sodium carbonate filter paper. Whatman #44 filter paper is dipped into a 10% aqueous solution of sodium carbonate and dried. This paper may be kept indefinitely. This paper is used in conjunction with the S.D.-328 paper for HS and the DB3 paper for 1130.

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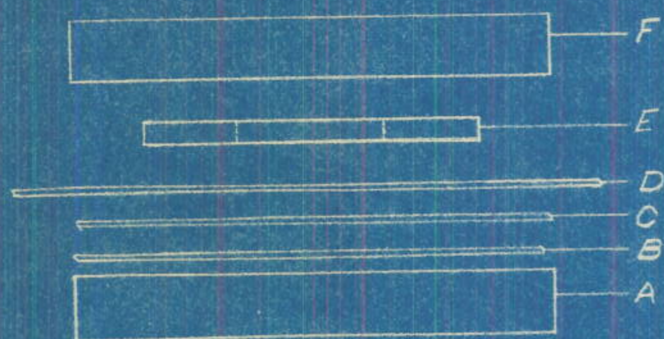

LIQUID VESICANT PENETRATION-TEST ASSEMBLY

PLATE

APPENDIX B

Penetration of Materials by Liquid Mustard

1. Penetration of Treated Papers. These data are given in Table I. In each case the source of the material is given where known. The values for the mustard penetration time written as > 1080 indicate that the test was discontinued after 1080 minutes and no penetration had occurred during that time.

2. Penetration of Thin Films. These data are given in Table II. The source of the material as well as the designation are given. Where the symbol > is used, such as > 960, the test was discontinued before penetration occurred.

3. Penetration of Rubber and Synthetic Rubber. These data are contained in Table III. The thickness in millimeters was measured for most of the materials. The rate of penetration in minutes per millimeter calculated from these data is given in the last column.

4. Penetration of Coated Fabrics. These data are given in Table IV.

Table I

Penetration of Treated Papers by Liquid Mustard

NRL #	Source	Designation	HPT Minut
1-10	Corn Products Co.	Mazein-coated paper	4
2-3	" " "	Heavy brown paper	69
2-4	" " "	Thin paper	2
2-5	Hazen Paper Co.	Mazein paper - CB-55	6
2-6	" " "	" " - CH-Manila	11
2-7	" " "	" " - M.K. Grade	10
2-8	Hazen Paper Co.	Mazein paper - C.W. - 47-1/2	5
2-9	" " "	" " - Lavender tissue	8
2-10	" " "	" " - Flexible Liner	22
4-15	" " "	" "	10
5-5	Dixie Wax Paper Co.	#4M	>1080
5-6	" " " "	#5M	>1080
5-7	Dixie Wax Paper Co.	#2V018	>1440
5-8	" " " "	#4M, water leached	>1440
5-9	" " " "	#5M, water leached	>1440
5-14	Riegel Paper Co.	25# Glassine	5
5-15	" " "	35# Vegetable Parchment	5
5-16	" " "	50# MP HS	> 240
5-17	Riegel Paper Co.	43# MPHS	60
5-18	" " "	28# MPHS	36
5-19	" " "	45# Glassine	>1440
6-13	Dixie Wax Paper Co.	Sample A	28
6-14	" " " "	" B	6
6-15	" " " "	" C	5
7-1	Hazen Paper Co.	A-55CB-GHS	230
7-2	" " "	A-60WK-GHS-CIS	340
7-6	" " "	A-30CB-GHS	200
7-9	" " "	A-60WK-GHS	265
8-2	" " "	Wall Paper	< 1
9-14	Corn Products Co.	MG-7	113
9-9	" " "	Mazein Nevillac paper	16

[redacted]

Table II

Penetration of Thin Films by Liquid Mustard

NRL #	Source	Material	HPT Minutes
2-11	DuPont	Cellophane PA-water soluble	> 960
2-12	"	" + PVA	>1020
2-13	"	PVA-Ketal type	> 180
2-14	"	Nylon film	> 180
2-15	"	MSAT cellophane	> 180
2-16	"	PVA	> 180
2-17	DuPont	Cellophane PA - moistureproof	> 180
2-18	"	PVA-K-1	>2400
2-19	"	" -K-2	>2400
2-20	"	" -Sample #1	> 180
3-1	"	" " #2	> 180
3-2	"	" " #3	> 180
3-3	DuPont	PVA-Sample #4	> 180
3-4	"	" " #5	9
7-17	"	Cellophane - 200 Type B, PA, MSAT	> 2400
8-19	Sylvania Industrial Corp.	" - #750 PVMS, white	170
8-20	Sylvania Industrial Corp.	" - #750 PVMS, olive drab	60
13-6	Prot. cover, individual	Laminated, cellophane, clear	> 300
13-7	Prot. cover, individual	" " , olive drab	> 300

[redacted]

Table III

Penetration of Rubber and Synthetic Materials by Liquid Mustard

NRL #	Sample	Thickness, mm.	HPT Min.	Min./mm.
3-5	Nylon on neoprene	0.79	>1080	>1370
3-6	Thiokol FA	0.80	13	16
3-7	" M	1.09	25	23
3-11	Saflex F-1	0.58	112	193
3-12	" F-2	0.62	247	400
3-13	Butvar	0.52	99	190
3-14	Acryloid C-10 on neoprene	0.54	90	167
3-16	Neoprene	1.90	50	26
3-17	Rubber	1.66	235	141
3-20	Chemigum with nylon interface		> 1200	
4-16	Vinylite - white	0.11	4	36
4-17	" - green	0.20	8	39
7-19	Vinylite X	0.79	96	121
11-1	Cellulose acetate	0.38	> 1200	> 3160
10-17	Butyl rubber	1.29	> 1440	> 1115

[redacted]

Table IV

Penetration of Coated Fabrics by Liquid Mustard

NRL #	Source	Material	HPT Min
1-1	Valentine	Cockpit fabric	12
1-2	Pantasote	" "	25
1-3	Unidentified	" "	10
1-4		Brunsene C	6
1-5		" H	5
1-6		" I	5
1-7		2002-1	18
1-8		2002-2	30
1-9		Mazein-coated fabric	12
1-12		Krasoid #1	4
1-13		" #2	10
1-14		" #3	5
1-15		Krasoid #4	4
1-16		" #5	3
1-17		"	6
1-18		Airplane fabric nitrocellulose dope	31
1-19		Cellulose nitrate fabric	5
3-8		Thiokol Airplane Fabric	172
3-10		Ameripol on airplane fabric	46
3-19		Lignin coated fabric	75
4-1		Mazein coated Arnzen cloth	4
4-2		PVA cement on fabric	39
4-3		Sawyer-green	38
4-4		" gray	47
4-5	Stedfast Rubber Co.	#CX	72
4-6	" " "	#CY	92
4-7	" " "	#CZ	180
4-8	" " "	#DA	>360
4-18	U.S. Rubber Co.	L-8734	193
4-10	U.S. Rubber Co.	L-8292	147
4-11		Mazein on Arnzen	34
4-12	Bureau of Aero.	Dark back cloth	3
4-13	" " "	Light back cloth	3
4-14	" " "	Striped cloth	4
4-19		Thiokol coated fabric	60
4-20		Mazein coated fabric	95

(Cont'd.)

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Table IV (Cont'd.)

NRL #	Source	Material	HPT Min.
5-1		Mazein-coated celanese	47
5-4		Laminated mazein, 2 cloth layers	>1080
5-10	Congoleum-Nairn	#28A-Z	148
5-12	DuPont	Fairprene #5033	78
5-13	"	" #5056	28
6-1	Montsanto	#25960B	262
6-2	Montsanto	#25960A	> 300
6-3	"	#550 Kendall	79
6-4	"	#25960C	70
6-5	"	#550 Balloon	69
6-7	Stedfast Rubber Co.	#DG	236
6-8	Lincoln Bleachery	Nylon on balloon	163
6-9	Lincoln Bleachery	Nylon on fabric	162
6-10	Congoleum-Nairn	Aluminum foil laminated fabric	>1440
6-12	Sawyer	Slicker	104
6-19	Lincoln Bleachery	Koroseal	15
6-20	Atlas Powder Co.	Cellulose nitrate coated fabric	10
7-15		Mazein on Arnzen	> 240
7-16	DuPont	NCR 7684-SS-819, nylon	>1440
7-19	Atlas Powder Co.	Zapon XO-340	> 240
7-20	" " "	" XO-285	53
8-3	" " "	" XO-351, single	5
8-4	" " "	" XO-351, double	10
8-5	" " "	" XO-350, "	10
8-6	Stedfast Rubber Co.	Plasted	10
8-7		Vulcanized Mazein	> 1440
8-9	DuPont	VCR-7733-SS-828	93
8-10	Congoleum-Nairn	WSOD	280
8-11	Acme Prot.Equip.Co.	T-102	> 1200
8-12	" " " "	T-102-B (Rebaked 120)	> 240
8-13	Acme Prot.Equip.Co.	T-102-B (Rebaked 132)	> 1200
8-15	DuPont	Resin impregnated fabrikoid	30
8-17	Devoe-Reynolds	Gasproof cloth	120
9-2	DuPont	Nylon on duck	320
9-3	CWS (MIT)	Alkyd-coated cotton	43
9-6	Hodgeman Rubber Co.	Butyral-coated duck	80

(Cont'd.)

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Table IV (Cont'd.)

NRL #	Source	Material	H M
9-7	DuPont	#538, nylon on duck	31
9-8	"	#537 " " "	6
9-10	Hodgeman Rubber Co.	Saflex-coated fabric	
9-11	R-E-H Dispersions	Thiokol-coated cloth	
9-12	CWS (MIT)	Alkyd-coated rayon	
9-13	Hodgeman Rubber Co.	Laminated butvar	
9-15	Goodyear Rubber Co.	Hycar OR, PVA laminated	> 12
9-16	Pantasote Co.	M-729, non-specular blue-gray	
9-17	NDRC	CR-7896	
9-18	"	CR-7822	<
9-19	"	CR-7719	<
9-20	"	PS-42.247	
10-1	DuPont	Pantasote-cellophane MSAT laminated	> 12
10-2	"	" " " "	> 12
10-3	"	" " CA48 "	
10-4	"	" " " "	
10-5	Pittsburgh Plate Glass Co.	CR-39 impregnated duck	> 12
10-6	CWS (MIT)	Pyroxylin-coated cotton cloth	
10-7	CWS (MIT)	Pyroxylin-coated cotton cloth	
10-8	DuPont	Fairprene 5007	1
10-9	"	" 5006	
10-12	Newmarket Mfg.Co.	J-8528, coated rayon	1
10-13	Congoleum-Nairn	08I-1, alkyd-coated Pantasote	
10-14	" "	08I-2, " " "	
10-18	NDRC	Gelatin-laminated fabric	> 12
10-20	"	Nitrocellulose, castor oil coating	
11-2	Dunnflex	Gelatin impregnated glass cloth	> 12
11-13	"	Gelatin impregnated cotton cloth	> 12
11-4	Congoleum-Nairn	02I-1, alkyd coated cotton	2
11-6	NDRC	F-1255	
11-7	NDRC	CR 8050	
11-8	Hodgeman Rubber Co.	Butyl rubber on rayon	
11-9	NDRC	Cellulose acetate coated fabric	
11-12	NAF, Phila.,Pa.	#1, butyl rubber on cotton	
11-13	" " "	#2, " " " rayon	
11-14	American Cyanamid	T-1, alkyd-coated Pantasote	1

(Cont'd.)

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Table IV (Cont'd.)

NRL #	Source	Material	HP Mi
11-15	American Cyanamid	T-2, alkyd-coated Pantasote	6
11-16	" "	T-3, " " "	5
11-17	Electrotechnical Prod. Co.	Alkyd-coated rayon	2
11-18	Electrotechnical Prod. Co.	" " cotton	2
11-20	NAF, Phila., Pa.	#3	<
12-1	" " "	#4	<
12-2	NAF, Phila., Pa.	#5	
12-3	" " "	#6, butyl rubber on rayon	15
12-6	Congoleum-Nairn	02I-9	11
12-7	" "	02I-8	18
12-12	NAF, Phila., Pa.	#8, polyvinyl butyral on rayon	8
12-13	" " "	#9, Fairprene on cotton	2
12-19	American Cyanamid	#2, alkyd on duck	
12-20	" "	#4, " " "	2
13-1	" "	#5, " " rayon	<
13-2	" "	#7, " " "	2
13-3	NAF, Phila., Pa.	#7	2
13-4	" " "	#10 butyl rubber on rayon	29
13-5	Devoe-Reynolds	Alkyd on cotton	3

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