

UNCLASSIFIED

CONFIDENTIAL

#674
CR

Division 9

NATIONAL DEFENSE RESEARCH COMMITTEE

of the

OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

Regraded **UNCLASSIFIED**
by authority of Secretary
of Defense Memo
2 AUG 1960
by ELDON V. MORGAN, Chairman,
Classified Documents Review Section
CRDL, A Cml C, Md.

OSRD No. 5286

Copy No. 25

UNCLASSIFIED

CONFIDENTIAL

Reg No 674431

Division 9
NATIONAL DEFENSE RESEARCH COMMITTEE
of the
OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

PERMEABLE PROTECTIVE FABRICS XL - FIELD
IMPREGNATION SYSTEM FOR S-461 - STATUS REPORT

to
October 1, 1943
by
Chemical Department, Experimental Station
E. I. du Pont de Nemours and Company

Report OSRD No. 5236
Copy No. 25
Date: June 30, 1945

Copy Nos.

1-7	Dr. Irvin Stewart
8-24	OSRD Liaison Office
25	Office of the Chief, Chemical Warfare Service Technical Division, Washington 25, D.C.
26-29	Commanding General, Chemical Warfare Service Technical Command, Edgewood Arsenal, Maryland
30	Director, Naval Research Laboratory
31-32	Commanding Officer, CWS Development Laboratory Massachusetts Institute of Technology Attention: (b) (6)
33	Office of the Quartermaster General Attention: Brig. Gen. G. F. Doriot
34	Commanding Officer, Dugway Proving Ground
35	Dr. A. C. Cope
36	Dr. Homer Adkins
37	Dr. P. L. Salzberg
38	Dr. W. R. Kirner

Total Number of Copies - 50

This document contains information affecting the national defense of the United States within the meaning of the Espionage Act, U.S.C. 50; 31 and 32. Its transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.

~~CONFIDENTIAL~~

Division 9
NATIONAL DEFENSE RESEARCH COMMITTEE
of the
OFFICE OF SCIENTIFIC RESEARCH AND DEVELOPMENT

OSRD No. 5286

PERMEABLE PROTECTIVE FABRICS XL - FIELD
IMPREGNATION SYSTEM FOR S-461 - STATUS REPORT

Service Directives: OWS-24; NL-B27

Endorsement (1) from Dr. Homer Adkins, Member, Division 9 to Dr. Walter R. Kirner, Chief, Division 9.

Forwarding report and noting:

"Field units have been developed for the emergency impregnation of clothing with S-461 and demonstrated under field conditions (OSRD 3362) in parallel with similar units employing CC-2. The present report reviews all of the development work on S-461 field set formulations up to November, 1943 at which time work was discontinued. S-461 impregnating baths can be prepared using polyvinyl alcohol as the dispersing agent and chloroparaffin as the binder, and, as in the case of the CC-2 field set, the field process is efficient under a wide range of conditions. Very satisfactory formulations employing Aresklene 400 (dibutyl phenyl phenol sodium disulfonate) as the dispersing agent have also been developed; these can be prepared very rapidly by a simplified procedure."

(2) From Dr. Walter R. Kirner, Chief, Division 9 to Dr. Irvin Stewart, Executive Secretary of the National Defense Research Committee.

Forwarding report and noting:

"OSRD Reports numbered 636, 1279, 3362, 3987, 4592, 4603, 4628, 4631, 4759, and 4910 comprise the reports already issued covering the work carried out by the du Pont Company on the use of S-461 in permeable protective fabrics. For other reports on the study of fabrics impregnated with S-461, consult NDRC Division B Formal Progress Reports Serial Numbers 309 (OSRD 794), 428 (OSRD 1010), 461 (OSRD 1118), and NDRC Division 9 Progress Reports, OSRD Numbers 1209, 1760, 1762, and 3942. OSRD Report 5276 should be consulted for the numbers of OSRD reports on the use of CC-2 in permeable protective fabrics."

This is a progress report under Contract 9-255, OMSr-361 with the E. I. du Pont de Nemours and Company.

~~CONFIDENTIAL~~

TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	1
OBJECTIVES	1
SUMMARY AND CONCLUSIONS	1
PROGRAM	3
EXPERIMENTAL SECTION	3
I. Development of the S-461 Field Procedure for Impregnating Clothing	3
A. Adapting the S-461 T of O Formula to Field Use	3
B. Large-Scale Trial of Field Process	4
C. Tentative Field Procedure	5
D. Demonstration of Process at Naval Research Laboratory	6
E. Field Trials at Edgewood Arsenal	6
F. Recommended Procedure for Future Use	7
II. Properties of Herringbone Twill Cloth Impregnated by the S-461 Field Process	8
A. Stability	8
B. Laundry Resistance	9
C. H Vapor Resistance	9
III. Factors Influencing the Field Performance of the S-461 Set	10
A. Storage Stability of the Field Set	10
B. Operation of the Field Process under Unfavorable Conditions	11
C. Comparison of S-461 Supplied by Commercial Solvents Corporation and Merck	12
IV. Simplified System Dispersed by Aresklene 400	13
APPENDIX	

INTRODUCTION

A method for impregnating clothing in the field was developed to fill the need for an emergency process which could serve when the usual supply of impregnated clothing could not be obtained. The field impregnating set has the additional advantage of reducing the demands on transportation facilities. The set will impregnate roughly four times its gross weight of clothing, and therefore eliminates shipping this amount of clothing to and from a central impregnating plant.

The first impregnation process utilized impregnite CC-2. This report describes preliminary work on a field process which utilizes impregnite S-461.

The work described here was carried out by Drs. H. H. Hoehn and I. F. Walker under the supervision of Dr. G. D. Patterson.

OBJECTIVE

The objective of this study has been the development of a field process for impregnation of clothing with S-461. The S-461 process was to be analogous to the CC-2 process insofar as this was consistent with satisfactory performance.

SUMMARY AND CONCLUSIONS

1. A method has been devised for impregnating clothing with S-461 without the use of mechanical equipment. The process has been operated successfully in two field trials at Edgewood Arsenal by inexperienced men operating from written directions only. Since a decision to use S-461 in clothing impregnation rests on general questions not yet answered, development of this field set has not been carried beyond the preliminary stage reported here. Improved stabilizing systems have not yet been adapted to it.

2. Preparation of an impregnating suspension requires four steps: (a) preparation of a polyvinyl alcohol (PVA) solution, (b) emulsification of chloroparaffin in the PVA solution, (c) dispersion of S-461 in the concentrated emulsion, and (d) addition of camouflage color and dilution of the suspension to impregnating strength. Clothing is dipped and wrung free of excess suspension by hand. Mixing is also done manually, using a paddle and canvas mixing bucket which are provided in the field impregnating set. About 1-1/4 hours are required to prepare the suspension.

3. The impregnating suspension contains 12.5% S-461, and its composition is S-461/chloroparaffin/ZnO/Daxad 11/polyvinyl alcohol/"Duponol" ME = 100/75/50/5/5/0.15. This is an adaptation of the ingredient formula originally used for mechanical impregnation in the T of O plant. Chloroparaffin has been cut from 100 to 75 parts to reduce the weight of the set, and "Duponol" ME is used as a wetting agent to assist in obtaining a PVA solution rapidly.

4. Clothing impregnated by the field method has been equivalent to clothing impregnated with S-461 in the T of O plant in stability, laundering, and H-vapor tests.

5. The S-461 field process is effective under a wide range of conditions. It has been operated at 0° and at 45°C., and both synthetic hard water and seawater have been used in preparing the suspension. Results were satisfactory in all cases. In preliminary aging tests, the components of the field impregnating set have shown no evidence of serious instability.

6. The original field process has been changed in one minor respect to insure stability when stored at high temperatures. Originally, the PVA in the field set was blended with ZnO, Daxad 11 and "Duponol" ME. Thorough dilution of the PVA with these dry materials insures that it will dissolve quickly. According to the revised procedure, these materials are blended at the time the set is to be used. This change avoids shipment of a PVA-ZnO blend, a mixture which has proved to be unstable at high temperature and humidity.

7. A new impregnating formula has been developed which uses Aresklene 400 as the dispersing agent. (Aresklene 400 is sold by the Monsanto Chemical Company and is said to be dibutyl phenyl phenol sodium disulfonate.) With the aid of Aresklene it is possible to prepare a disperse system by merely stirring all the materials together with a portion of the water. Working on a full scale about 15 minutes are required for mixing and diluting the suspension to impregnating strength. A typical formula contains S-461/ZnO/chloroparaffin/Aresklene/color in the ratio 100/20/75/15/5.

PROGRAM

No further work on the S-461 process is planned at the present time. Should active interest in the use of S-461 develop later, the field process would be reviewed to determine whether any simplification of the procedure might be made.

EXPERIMENTAL SECTION

I. Development of the S-461 Field Procedure for Impregnating Clothing

A. Adapting the S-461 T of O Formula to Field Use

The development of a procedure for impregnating clothing with CC-2 under field conditions suggested that a similar procedure could be used for S-461. The higher reactivity of S-461, however, required that the field procedure be based on a formula which would insure satisfactory aging characteristics in the impregnated fabrics. The formula selected for study was one which had given good results in T of O plant trials; it contained S-461/ZnO/chloroparaffin/PVA/Daxad 11 in the proportions 100/50/100/5/5. The following example (4975-147) illustrates the preparation of an S-461 suspension with only hand mixing:

Four hundred grams of 10% PVA (RH-403) solution, 100 g. water, and 40 g. Daxad 11 (a naphthalene sodium sulfonate condensed with formaldehyde) were put in a galvanized pail and stirred until the Daxad had dissolved. Then 800 g. of chloroparaffin was added and the mixture stirred by hand for 5 minutes. This produced a cream-colored viscous emulsion which was then diluted with 660 g. of water. Eight hundred grams of S-461 and 400 g. ZnO were added next. Thirty minutes of stirring gave a smooth suspension which was free of undispersed S-461. The suspension was diluted to 10% S-461 with 4800 g. of water. The system was well dispersed and the majority of S-461 and chloroparaffin particles were below 5 microns.

Swatches of herringbone twill fabric were soaked in the suspension, wrung out by hand, and dried on a line. The fabrics had satisfactory appearance. The average active chlorine content was 0.93 mg./sq.cm. The aging data below show the fabric impregnated by hand was fully equivalent in stability to fabric impregnated mechanically in the T of O plant:

Test	Chlorine Retained		Tensile Strength	
	Field	T of O	Field	T of O
SP-2 (70°C., 100% RH, 96 hours)	62%	50%	91 lb.	26 lb.
Tropical Storage; unwrapped single fabric; 28 days	76	69	75	57
Outdoors Florida; Method I; 14 days	82	57	83	72
Outdoors Wilmington; 28 days	39	46	99	97

B. Large-Scale Trial of Field Process

After demonstrating on a small scale that the field procedure could be used for S-461 impregnation, a large-scale test was made. In this case the complete field process was operated, i.e., a PVA solution was prepared from dry PVA. The formula was the same as that used for the small-scale test except that 1% "DuPont" ME was added to the PVA to increase its rate of solution. The field procedure consists of four steps:

1. Put 1 gallon of water in a 30 gallon drum and add Package #1. (Package #1 contained 1150 g. ZnO, 430 g. granular PVA (RH-403), and 4.3 g. "DuPont" ME.) Stir and add immediately 430 g. Dexad 11. Continue stirring for 15 minutes.
2. Add 18-3/4 lb. chloroparaffin and stir for 15 minutes.
3. Add 18-3/4 lb. S-461, 6 lb. ZnO, and 2-3/4 gallons of water. Stir for 15 minutes.
4. Dilute with 8-1/2 gallons of water and impregnate fabric.

In the actual test steps #3 and #4 were carried out on a reduced scale to conserve S-461. Five pounds of S-461, 1.82 lb. ZnO, and 3 quarts of water were stirred into 8.4 lb. of concentrated emulsion from step #2. The dilution (step #4) was made with 9 quarts of water.

This test showed that S-461 suspensions could be made readily in quantities large enough for field use. The only modification in procedure indicated by the test was an increased dilution in step #1. The interaction of PVA and Daxad made the

viscosity unexpectedly high. This effect had not been encountered before because the CC-2 field procedure did not use Daxad.

Herringbone twill fabric impregnated by hand had good appearance. The cloth contained about 1 mg. active chlorine/sq.cm. Complete aging data on the fabric (4975-166) will be found in Table I; the selected data below show the fabric had good aging characteristics:

<u>Test</u>	<u>Chlorine Retained</u>	<u>Tensile</u>
SP-2 (70°C., 100% RH); 96 hours	78%	74 lb.
Tropical Storage; unwrapped single fabric; 28 days	72	77
Outdoors Florida; Method I; 14 days	36	75
Outdoors Wilmington; 28 days	47	94

C. Tentative Field Procedure

On the basis of the large-scale test just described a tentative field procedure consisting of four steps was established:

FIELD PROCEDURE

(For 50 lb. of S-461)

Step 1. Put 4 gallons of water in a 50-gallon drum and add Package #1. Stir for 15 minutes. (Package #1 is a mixture of 2-1/2 lb. standard PVA (RH-403), 2-1/2 lb. Daxad 11, 25 lb. ZnO, and 0.075 lb. "Duponol" ME.)

Step 2. Add Package #2 (50 lb. chloroparaffin) and stir for 15 minutes.

Step 3. Add 6 gallons of water and Package #3 (50 lb. S-461). Stir for 15 minutes.

Step 4. Dilute with 22 gallons of water and impregnate.

It will be noted that in Step 1 granular PVA is no longer required. Work on the CC-2 field process had shown that standard, i.e., small particle size, PVA was satisfactory

~~SECRET~~

providing it was diluted with at least an equal weight of ZnO. If the ZnO is omitted, the PVA particles agglomerate and form lumps which are slow to dissolve. To wet out readily the larger volume of material added in Step 1, the concentration of "Duponol" ME has been increased to 3% on the PVA.

This recommended procedure was tested in the laboratory on a 1/50 scale (1 lb. S-461) with entirely satisfactory results (HHH, N.B. 5006-59). Aging characteristics of the impregnated fabric are included in Table I.

D. Demonstration of Process at Naval Research Laboratory

On December 21, 1942, a test of the tentative field procedure outlined above was made in the Naval Research Laboratory in Washington. The scale of the test was that equivalent to 25 lb. S-461. The viscosity of the system was still somewhat high during emulsification of chloroparaffin. Otherwise the test was entirely successful.

E. Field Trials at Edgewood Arsenal

1. First Trial

When plans were made for testing the CC-2 field process on a large scale at Edgewood, arrangements were made also for a limited test of the S-461 field method. Two five-pound field sets were prepared for the test. Each set contained 5 lb. S-461 and other chemicals in the proper proportions, as well as illustrated instructions, measures for water, canvas mixing bucket, and a paddle for stirring. The five-pound set was designed to impregnate five complete sets of clothing consisting of socks, gloves, underwear, hood, leggings, and coveralls (or trousers and shirt).

The field sets were given to troops without previous experience in the field procedure. The men were to prepare an impregnating suspension and impregnate clothing merely by following the instructions furnished with the set.

The procedure operated successfully in spite of unfavorable weather conditions. The test was made in January, and the temperature was at the freezing point throughout the trials. The low temperature increased the viscosity of the PVA solution and chloroparaffin beyond our expectation, with the result that mixing was more difficult than necessary. This trouble has been corrected by using more water in the first stages of the process.

~~CONFIDENTIAL~~

2. Second Trial

The S-461 field process was tested again at Edgewood in early April. Several changes had been made in the process since the January trials. These changes included standardization of the shipping container for the set, modified operating instructions, standardization of the set to a 24-lb. scale, use of color in the set, and modified dilution schedule. To conserve space, the chloroparaffin content of the formula was reduced to 75% (on S-461), giving S-461/chloroparaffin/ZnO/PVA/Daxad 11/"DuPontol" ME/color ratios of 100/75/50/5/5/0.15/3.

Again two field sets were given to troops without previous experience in the field process. They prepared the suspension and impregnated clothing according to the instructions supplied with the field set. The process operated very satisfactorily, even though the temperature was below freezing. Viscosity of the mixes was satisfactory at all stages. Both trials gave good suspensions.

F. Recommended Procedure for Future Use

The procedure which was followed in the second field trial described above was entirely satisfactory from the operational standpoint. On the basis of our experience with the CC-2 field process it is considered advisable to revise the S-461 field procedure so that it will not be necessary to ship a blend of PVA and ZnO. Investigation of the stability of the CC-2 field impregnating set has shown that the PVA-ZnO-"DuPontol" ME mixture has much lower stability than a mixture of PVA and "DuPontol" ME alone. The instability is greatest under conditions of high temperature and humidity. The original purpose in blending the ZnO and PVA was to dilute the latter sufficiently to obtain a solution rapidly. It has been shown, in the case of the CC-2 process, that adequate blending can be obtained if the dry materials are stirred together with a paddle in the field mixing bucket. This eliminates the need for shipping a pre-blended ZnO-PVA mixture. To obtain greater stability for the S-461 field set we propose therefore to package the ZnO and PVA separately. The revised process would consist of the following steps:

~~CONFIDENTIAL~~

S-461 Field Process

(For 24 lb. S-461)

Step 1: Pour Package #1 into mixing bucket. Sprinkle in Package #2 and stir the dry powder for 2 minutes. (Package #1 consists of 7.2 lb. ZnO. Package #2 consists of 1.2 lb. PVA, 1.2 lb. Daxad 11, and 0.036 lb. "Duponol" ME.)

Step 2: Add 3 gallons of water and stir rapidly for 20 minutes.

Step 3: Add Package #3 and stir for 20 minutes. (Package #3 is 18 lb. chloroparaffin.)

Step 4: Add Package #4 and 2-1/2 gallons of water. Stir for 20 minutes. (Package #4 consists of 24 lb. S-461 stabilized with 4.8 lb. blended ZnO.)

Step 5: Dilute with 10 gallons of water in which Package #5 has been dissolved. (Package #5 consists of 0.72 lb. dispersible camouflage color.)

Step 6: Impregnate clothing.

II. Properties of Herringbone Twill Cloth
Impregnated by the S-461 Field Process

Herringbone twill fabric impregnated by the field process with aqueous suspension containing 100 parts S-461, 50 parts ZnO, 100 parts chloroparaffin, 5 parts PVA, 5 parts Daxad 11, and 0.15 parts "Duponol" ME was compared with S-461 T of O fabric and CC-2 field fabrics for stability, laundry resistance, and H vapor resistance.

A. Stability

Aging tests show that herringbone twill impregnated by the S-461 field process was equivalent to T of O fabric in chlorine retention and fabric stability. The S-461 field fabrics were somewhat inferior to CC-2 field controls in tensile retention in the tropical storage-unwrapped single fabric test. In other respects there was very little difference in the aging characteristics of the three types of fabrics compared. This is shown by data given on the following page.

1. SP-2 (70°C., 100% RH)

<u>Designation</u>	<u>Method of Impreg.</u>	<u>Impregnite</u>	<u>Original</u>		<u>48 Hrs.</u>		<u>96 Hrs.</u>	
			<u>Mg.⁺ Cl/cm²</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>
A-F*	Field	S-461	1.08	83 lbs.	83	80 lbs.	67	59 lbs.
A-M-2**	T of O	"	1.88	105	76	57	50	26
5006-61-1	Field	CC-2	0.21	81	48	55	14	62

*Average of data for runs 4975-166, 5006-59, 5006-96 and 5007-7.

**Average of data for M-2 runs 41, 55, 56, 57.

2. Tropical Storage - Unwrapped Single Fabric (46°C., 80-85% RH)

<u>Designation</u>	<u>Method of Impreg.</u>	<u>Impregnite</u>	<u>Original</u>		<u>14 Days</u>		<u>28 Days</u>		<u>42 Days</u>	
			<u>Mg.⁺ Cl/cm²</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>
A-F	Field	S-461	1.08	83 lbs.	95	75 lbs.	70	60 lbs.	50	52 lbs.
A-M-2	T of O	S-461	1.88	105	-	-	69	57	-	-
5006-61-1	Field	CC-2	0.21	81	86	79	43	72	38	76

3. Outdoor Exposure - Florida Method 1.

<u>Designation</u>	<u>Method of Impreg.</u>	<u>Impregnite</u>	<u>Original</u>		<u>7 Days</u>		<u>14 Days</u>		<u>28 Days</u>	
			<u>Mg.⁺ Cl/cm²</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>	<u>% Cl</u>	<u>Tensile</u>
A-F	Field	S-461	1.08	83 lbs.	58	81 lbs.	30	73 lbs.	0	50 lbs.
A-M-2	T of O	"	1.88	105	69	77	57	72	22	43
5006-61-1	Field	CC-2	0.21	81	57	74	29	81	-	73

htt
12/3/43

B. Laundry Resistance

Washfastness of herringbone twill impregnated by the S-461 field process compared favorably with that of the S-461 T of O and CC-2 field controls. The following data, obtained by laundering impregnated fabric with Quartermaster Formula G soap solution at 32°C. in a small mechanical washer, provide the data for these conclusions:

<u>Code</u>	<u>Impregnation</u>	<u>Original Cl₂ Content mg./sq.cm.</u>	<u>% Chlorine Retained after Laundering</u>			
			<u>1st</u>	<u>2nd</u>	<u>3rd</u>	<u>4th</u>
A-F-L*	S-461; field	1.00	78	20	35	19
A-M-2-L**	S-461; T of O	1.73	75	59	43	32
A-F-C-L***	CC-2; field	0.46	69	51	41	39

* Average of 2 tests: 5006-69; 5006-97

** Average of 4 tests: M-2-41, -55, -56, -57

*** Average of 3 tests: Field trials 1-1, 5-1, 7-10 (N.B. 5003)

C. H Vapor Resistance

H vapor resistance of field and T of O fabrics was determined by the Charles Lennig Company, Philadelphia. Data given below show that the S-461 field fabrics neutralize mustard vapor as efficiently as S-461 T of O fabric. Comparison of the break times for S-461 field fabrics with a CC-2 control shows that S-461 gave much longer protection than the fabric treated with CC-2.

<u>Designation</u>	<u>Method of Impregnation</u>	<u>H Vapor Test</u>	<u>Chlorine mg./sq.cm.</u>	<u>Break time min.</u>	<u>Efficiency (minutes/ mg. of Cl)</u>
5007-7-2	Field; S-461	CWS	1.04	750	721
5007-7-2	" "	NRL	1.04	562	540
A-M-2*	T of O; S-461	CWS	1.64	1214	751
"	" "	NRL	1.64	1193	727
5401-158-2	Field; CC-2	CWS	0.51	506	994
"	" "	NRL	0.51	284	557

*Average of M-2 runs 41, 55, 56, and 57.

III. Factors Influencing the Field Performance of the S-461 Set

The practical utility of the S-461 field process will depend upon its performance under unfavorable operating or storage conditions. Some of the operating conditions most likely to be met have been simulated in laboratory tests. In addition, considerable information on the stability of the components of the field set has been obtained. There appears at present to be no particular storage problem for the S-461 field set, nor is there any specific operating condition which must be avoided for satisfactory performance of the set.

A. Storage Stability of the Field Set

1. Polyvinyl Alcohol

Extensive storage tests have been made of polyvinyl alcohol compositions in developing the CC-2 field procedure. These tests have been described in detail in another report.* Under certain conditions the PVA becomes insoluble and incapable of emulsifying chloroparaffin. The conclusions reached which are of interest here are: (1) The combination of PVA and ZnO is sufficiently unstable at high temperature and humidity that the mixture should not be used in field sets which are to have the longest possible period of utility. (2) Either PVA alone or a blend of PVA and "DuPont" ME is stable enough for use in the field set. It was on the basis of these conclusions that a revised field procedure, which does not require a premixed ZnO-PVA composition, was recommended for the S-461 field set. Although we have no reason to expect that the mixture of PVA-"DuPont" ME-Daxad 11 used in the revised process will be unstable, practical and accelerated storage tests of this composition are in progress.

2. Chloroparaffin

This material has satisfactory stability. The Chemical Warfare Service recommends the use of resin-coated steel containers.

3. Camouflage Color

The camouflage color is considered stable. It is not mixed with any other material, and to protect from moisture it is shipped inside the metal can which holds the PVA-Daxad-"DuPont" mixture.

* "Permeable Protective Fabrics VI - Development of Standard CC-2 Field Impregnation System," OSRD No. 3389.

4. S-461

The stability of S-461 under various conditions is the subject of a report to be issued by Dr. H. H. Hoehn. His work indicated that S-461 (unstabilized) is much more resistant to decomposition under tropical storage conditions (46°C., 85% RH) than unstabilized XXCC-2. A permeable container holding XXCC-2 tenders to the point of failure in about 16 weeks, but with S-461 the container is in good condition at the end of this time. Unstabilized S-461 produces container failure at some period between 16 and 32 weeks of tropical storage, indicating need for a stabilizer. Tests are now in progress in which CaCO₃ and ZnO are being evaluated for this purpose.

B. Operation of the Field Process
under Unfavorable Conditions

To determine as far as possible how the field process would perform under special conditions, the following tests were made.

1. Hard Water

The field method gave no trouble when a synthetic hard water was used exclusively. The water had a hardness of 1500 ppm. Ca (added as CaCl₂) and 1000 ppm. Mg (added as MgCl₂). A search of the literature on this subject indicated that this water probably exceeded in total hardness any water (except sea water or tide water) which would ever be found.

2. Sea Water

Synthetic sea water was prepared by adding the following salts:

485 grams	NaCl
57.6	MgCl ₂
40	MgSO ₄
25	CaSO ₄
11	KCl

to sufficient water to make a total volume of 18 liters. When the field suspension was made with sea water, slight flocculation on the fabric was observed at a concentration of 12.5% S-461. At a concentration of 10% S-461 flocculation was more severe, so it is considered advisable to operate the formula at 12.5% S-461. This is the dilution now obtained by following the field operating instructions.

3. Low Temperature

The field process operates satisfactorily under freezing conditions. This was first determined by laboratory tests and later confirmed in outdoor trials at Edgewood Arsenal in January and April, 1943.

4. High Temperature

The field process has given no operating difficulties at 45°C. at any time. In this respect the process is superior to the original CC-2 field method, since the latter had some tendency to give a flocculated suspension at high temperature until the process was revised to overcome this trouble.

C. Comparison of S-461 Supplied by Commercial Solvents Corporation and Merck

1. Dispersing Characteristics

S-461 obtained from CSC (Commercial Solvents Corporation) contained lumps of varying size which, although not very hard, were difficult to break up by hand stirring. The larger lumps could be broken down easily by sifting the impregnite through a 20-mesh screen. However, even if sifted CSC S-461 was used, it was often difficult to obtain a uniformly smooth, grit-free suspension. In contrast, S-461 obtained from Merck was free of lumps and could be used directly in the field process without further treatment. Merck S-461 closely resembled micronized CC-2 in general appearance and performance in the field process.

2. Particle Size

The similarity between Merck S-461 and micronized CC-2 in their dispersing characteristics may be related to a similarity in particle size distribution. Dr. C. K. Sloan has determined the particle size distribution of S-461 supplied by CSC and Merck and also of micronized CC-2 by means of a pipette sedimentation method. His results show that the particle size of Merck S-461 is more like micronized CC-2 than that of CSC S-461. Although lumpy, S-461 manufactured at CSC has a smaller primary particle size than either micronized CC-2 or Merck S-461. The particle size data are tabulated below:

Impregnite	Distribution of Particles		
	Below 4 Microns	Below 8 Microns	Below 12 Microns
Merck S-461	30%	58%	73%
Micronized CC-2	44	88	98
CSC S-461	83	91	97

3. Aging Properties of Fabrics Prepared Using CSC and Merck S-461

Aging tests showed that the source of impregnite had very little effect on the properties of impregnated HBT cloth. Merck S-461 may be somewhat superior with respect to chlorine retention over CSC, but only under tropical storage conditions. The following data are given as evidence.

SP-2 (70°C., 100% RH)

Designation	S-461	Original		48 Hours		96 Hours	
		Chlorine mg./sq.cm.	Tensile #	% Cl	Tens. #	% Cl	Tens. #
5006-59-1	CSC	1.20	81	93	78	73	64
5007-7-1	Merck	1.04	81	90	77	68	55

Tropical Storage - Unwrapped Single Fabric - (40°C., 80-85% RH)

Designation	S-461	Original		28 Days		42 Days	
		Chlorine mg./sq.cm.	Tensile #	% Cl	Tens. #	% Cl	Tens. #
5006-59-1	CSC	1.20	81	56	57	33	37
5007-7-1	Merck	1.04	81	82	50	73	37

IV. Simplified System Dispersed by Aresklene 400

In connection with another study of impregnating formulas*, it was found that Aresklene 400 (dibutyl phenyl phenol sodium disulfonate), sold by the Monsanto Chemical Company, had the unusual property of both dispersing chloroparaffin and emulsifying impregnite with the aid of hand stirring. With this dispersing agent it is possible to prepare suspensions of good quality by merely mixing all the constituents and part of

XIX

*"Permeable Protective Fabrics/ - CC-2 - Simplified Field Impregnation Systems - Exploration Studies", OSD 4610.

████████████████████

the water at one time. In the case of S-461 a typical formula would be S-461/ZnO/chloroparaffin/Aresklene/water in the ratio 100/50/75/15/100. Materials are mixed for ten minutes and then sufficient water is added to give a suspension with 12.5% S-461. About 15 minutes are required for preparation of the impregnating suspension, compared to 70 minutes required for the standard field process recommended above (Section I-F).

In developing this formula trouble was met in attempting to color the impregnating suspension to overcome the whitening of the fabric which would otherwise occur. The water-dispersible color used in the PVA suspension was unsatisfactory on account of migration. To avoid uneven color caused by migration the formula was changed to accommodate a color-in-oil paste. The mobility of color dispersed in mineral oil or chloroparaffin is so low that uneven coloring has never been observed in this system. The pigment found to be most satisfactory for this use was 5% (on weight of S-461) of a mixture of ferrite yellow (Mapico Yellow-Orange sold by the Magnetic Pigments Company) with Superba carbon black (Binney and Smith) in the ratio 8:1. If a mineral oil paste is used, equal parts of oil and pigment are blended on a roller mill to a smooth paste. In case chloroparaffin is to be the pigment vehicle, three parts of chloroparaffin are milled with two parts of pigment.

Submitted by:
Experimental Station
Chemical Department
E. I. du Pont de Nemours & Co.

11-23-43

TABLE I
AGING PROPERTIES OF HERRINGBONE TWILL CLOTH IMPREGATED WITH S-461 BY THE FIELD PROCESS

Designation	S-461	Mo	Chloroparaffin	FVA	D	"Durosol" NR	MozCl/cm ²	# Tensile	SF-2 (70°C., 100% R. H.)		Tropical Storage-Unwrapped Single Fabric						Outdoor Exposure**													
									% Chlorine Retained		# Tensile		% Chlorine Retained		# Tensile		Florida I				Wilmington									
									48 hrs.	96 hrs.	48 hrs.	96 hrs.	14 da.	28 da.	42 da.	14 da.	28 da.	42 da.	7 da.	14 da.	28 da.	7 da.	14 da.	28 da.	7 da.	14 da.	28 da.			
A-M-2*	100	50	100	5	5	--	1.88	105	76	50	57	26	--	69	--	--	57	--	69	57	22	77	72	43	--	--	46	--	--	71
4975-147-A	100	50	100	5	5	--	0.91	88	78	62	89	91	77	76	57	63	75	64	--	82	3	--	83	56	67	41	39	103	102	99
4975-166-k	100	50	100	5	5	0.05	0.99	88	85	78	105	74	88	72	45	81	77	77	56	36	0	87	75	64	67	60	47	101	99	94
5006-59	100	50	100	5	5	0.15	1.20	81	93	73	78	64	83	56	33	75	57	37	43	8	0	77	78	62	94	67	27	85	30	39

* T of O control - Average of data for M-2 runs 41, 59, 56 and 57.

** Results not exactly comparable because exposures were made at different times.