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Report on

Aqueous Impregnating Systems of Chlorinated Paraffin and Impregnite S-145

NAVAL RESEARCH LABORATORY
ANACOSTIA STATION
Washington, D. C.

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Date of Tests:	March 1942 - March 1943
Submitted by:	H. W. Fox, Contract Employee
	G. M. Gantz, Contract Employee
	W. H. Taylor, Associate Chemist
Reviewed by:	W. C. Lanning, Senior Chemist
	P. Borgstrom, Head Chemist, Superintendent, Chemistry Division
Approved by:	A. H. Van Keuren, Rear Admiral, USN, Director
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ABSTRACT

This report deals with the studies that have been made on aqueous systems for impregnating protective clothing with the impregnite S-145 and the binding agent, chlorinated paraffin. The experimental work has been divided into four parts. The first part deals with the problem of formulating stable aqueous systems which can be used for impregnating clothing. The second part deals with the study of the stability of aqueous impregnating systems and component parts of such systems. The third part is concerned with those problems encountered in the application of aqueous systems as impregnating baths. For the most part these problems are peculiar to the use of dispersions of the impregnating materials rather than true solutions. The fourth part is concerned with the evaluation of cloth impregnated by the aqueous process especially in comparison with cloth impregnated by the solvent process.

Upon the basis of the data given in this report it is recommended that the Navy adopt an aqueous impregnating process at central impregnating plants and consider the adoption of aqueous impregnations for use at shore stations and advanced bases. It is also recommended that wearing trials be conducted to establish the life of protective clothing and determine possible irritant effects when such clothing is worn under service conditions.

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AUTHORIZATION

1. This work was authorized under Project 547/41, "Maintenance, Bureau of Ships," dated 16 December 1940. The problems which were proposed for study were given in Bureau of Ships letter S-S77-2(Dz) Serial 811 of 17 December 1940.

STATEMENT OF PROBLEM

2. This study was undertaken to devise a suitable method for impregnating protective clothing without the use of organic solvents. The present Navy directive for impregnating protective clothing involves the use of a solution of the impregnite S-145 and chlorinated paraffin in tetrachloroethane. This method has an outstanding disadvantage in that tetrachloroethane is highly toxic. Other solvents less toxic are unsatisfactory for other reasons such as low solvent power, reactivity, inflammability and high cost.

KNOWN FACTS BEARING ON THE PROBLEM

3. The problem of protective clothing in general has been adequately discussed in NRL Report No.P-2000 and elsewhere. No further discussion is given here not relating directly to aqueous impregnating systems.

4. When this investigation was started the only known fact bearing on the problem was that the British had developed a process whereby a concentrated dispersion of their impregnite A. V. could be diluted with water as needed and used to impregnate clothing. The process was described as useful for emergencies since the concentrated mixture was not stable for more than two months under tropical conditions.

5. Work on the problem of aqueous impregnation processes received great impetus when it was made a project of an NDRC group at the DuPont Experimental Station. A large proportion of the fundamental development work has been done by the DuPont group. Large scale development and evaluation of aqueous impregnating processes have been carried out by the CWS at Edgewood Arsenal with the cooperation of the DuPont group.

6. Large scale tests at Edgewood Arsenal have shown that aqueous impregnating systems are adaptable to solvent impregnating plants with a minimum of added equipment. An aqueous impregnation process designed for use by troops in the field has also been developed and found satisfactory in large scale tests.

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THEORETICAL CONSIDERATIONS

7. The use of water as a vehicle for the impregnite and binding agent has many obvious advantages, namely, universal availability, non-toxicity, non-inflammability and total evaporation. Since neither the impregnite nor the binding agent is soluble in water, the use of water requires that the impregnite be made into a suspension and that the binder be made into an emulsion. The following factors must also be considered before any aqueous impregnation process can be considered satisfactory.

(1) The impregnation process must be adaptable to facilities now on hand at central impregnation plants and must also be capable of application in the field or on shipboard without special equipment.

(2) The materials used in the process must be stable to both heating and freezing and capable of withstanding storage for at least one year.

(3) Clothing impregnated by an aqueous process must be as satisfactory as clothing impregnated by the present solvent process in regard to protective power and stability on storage or wear. It should also be as satisfactory in regard to comfort and wearing qualities and resistance to laundering.

PREVIOUS WORK DONE AT THIS LABORATORY

8. NRL Report No. P-2000 describes preliminary results obtained in the studies of aqueous impregnating systems and describes in detail the many test methods employed in the evaluation of impregnated clothing.

EXPERIMENTAL PART

Part I. Formulation of Aqueous Impregnating Systems.

A. Preparation of Emulsions

(1) Comparison of Emulsifying Agents.

9. The necessity of having the binding agent, chlorinated paraffin, dispersible in water required a study of emulsifying agents. A wide range of emulsifying agents were examined. In general, it is believed that non-ionic types of agents are most satisfactory in preparing stable

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emulsions of chlorinated paraffin. This is shown in Table I where the results obtained with several different emulsifying agents are listed.

Table I

Comparison of Emulsifying Agents

Agents	Ratio of Agent to Chlorinated Paraffin	Vehicle for Agent	Method* for Mixing	Stablility of Emulsion	Particle Size in Microns
Cetyl Alcohol + Orvus	-	Oil + water	HB	Unstable	-
" " + Avitex SF	-	" + "	"	"	-
Cholesterol + Orvus	-	" "	"	"	-
Emulphor AL	-	Oil	"	"	-
Emulphor ELA	-	Water	HB	Unstable	-
Glue	1/4	"	CM	"	10
Sorbitol Laurate	1/2	Oil	HH	"	10
" " + Aerosol OT	1/4	"	"	"	25
Sorbitol Laurate + Aerosol	1/10	"	"	"	50
Turkey Red Oil	1/4	Water	HB	Unstable	20
Triethanolamine Stearate	1/4	Oil	HB	"	8
Igepal CA	1/4	Water	HB	"	10
" "	1/5	Oil	"	"	2
Triton NE	1/4	Water	"	Unstable	-
" "	1/5	Oil	"	Stable	2
RH-403	1/4	"	HH	Unstable	10
"	1/20	Water(conc.)	HB	Unstable diluted	8
" + Gardinol WA	1/4/20	"	HB	Stable diluted	2
Methocel (15 cps)	1/20	Water	HB	Unstable diluted	8
" + Gardinol WA	1/4/20	"	"	Stable	2

*HB = Hamilton Beach stirrer CM = Colloid Mill
HH = Hand homogenizer

10. These results indicated that the most promising agents were Igepal CA, Triton NE, methyl cellulose (Methocel), and polyvinyl alcohol (RH-403). All of these agents are of the non-ionic type. Since preliminary studies of impregnated

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cloth samples showed that the agent Igepal CA caused a pronounced loss of active chlorine, no further work was done with this agent.

11. Typical formulations for the three most promising agents are as follows:

> Five parts chlorinated paraffin + one part Triton NE (30% solution) mixed at high speed until homogeneous gives a viscous paste which may be gradually diluted to give an emulsion of good stability with a particle size around two microns. The paste may be stored and used when desired.
>
> Ten parts chlorinated paraffin + five parts of a 10% 15 CPS Methocel solution mixed at high speed until homogeneous gives a viscous paste which may be gradually diluted to give a fairly stable emulsion whose droplets will average 4-6 microns in diameter. The addition of 1/4% Gardinol WA to the Methocel solution gives an emulsion with particles averaging 2-3 microns in diameter.
>
> Ten parts chlorinated paraffin + five parts of a 10% PVA solution acts like the Methocel emulsion except that the particle size averages 8-10 microns. The addition of Gardinol WA again has the effect of reducing the average particle size to 2-3 microns.
>
> Ten parts chlorinated paraffin + five parts of a 10% PVA or Methocel solution - 0.02 parts Gardinol WA - 0.1 part of Daxad 11 were mixed together with a paddle until homogeneous. The viscous mixture was placed in the Eppenbach colloid mill and passed through once at a clearance of 0.030". The resulting viscous paste could be diluted to an emulsion whose particle size averaged 1-2 microns. The Premier colloid mill gave identical results at 17,000 RPM with the same clearance with the difference that the latter's rate of production was roughly double that of the Eppenbach mill.

(2) Combinations of Emulsifying Agents.

12. Further work with RH-403 and Methocel led to the discovery that a small amount of a wetting agent such as sodium lauryl sulfate caused a large reduction in particle size of chlorinated paraffin emulsions. This is illustrated in Table II.

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Table II

Combinations of Emulsifying Agents

Agents	Ratio of Agents to Chlorinated Paraffin	Particle Size in Microns
RH-403	1/20	8
Rh-403, Gardinol WA	1/.04/20	2
Rh-403, Gardinol WA, Daxad 11	1/.04/.2/20	2
Methocel	1/20	8
Methocel, Gardinol WA.	1/.04/20	2
Methocel, Gardinol WA, Daxad 11	1/.04/.2/20	2

13. The agent Daxad 11, the sodium salt of a sulfonated naphthalene-formaldehyde condensation product, was also found to be a desirable addition agent to the chlorinated paraffin emulsions. Whereas a slurry of S-145 and S-42 added to a Methocel emulsion containing no Daxad 11 caused the emulsion to break, the presence of Daxad 11 prevented this behavior. Though RH-403 emulsions were found to be more stable to the addition of S-145 and S-42, in this case the Daxad 11 had a beneficial effect in reducing the agglomeration of the S-145. It was also found that the optimum concentration of Daxad 11 was 1% by weight of the chlorinated paraffin.

(3) Methods of Preparing RH-403 and Methocel Emulsions.

14. Several methods of preparing chlorinated paraffin emulsions with Methocel or RH-403 have been investigated. In all cases it has been observed that best results are obtained with concentrated mixtures which have a very high viscosity. Since it is not feasible to prepare Methocel or RH-403 solutions of more than 10%, the maximum concentration of chlorinated paraffin is 66-2/3%.

15. Hand stirring with a paddle will not produce an emulsion of as small a particle size as mechanical mixing. High speed mixing with a Hamilton Beach stirrer operating at 10,000-12,000 rpm was very satisfactory for preparing emulsions in the laboratory. However, it is not evident whether a comparable high speed stirrer could be obtained for large scale operations.

16. Colloid mills have been found quite satisfactory for preparing chlorinated paraffin emulsions. It has been observed that dilute mixtures of low viscosity passed through

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a mill at a small gap clearance will not produce as good an emulsion as more viscous mixtures passes through a wider gap. This behavior was found to be true for both the Eppenbach mill (serrated rotor and stator) and the Premier mill (smooth rotor and stator).

17. Information from the NDRC group at the DuPont Experimental Station that centrifugal and gear pumps could also be used for preparing chlorinated paraffin emulsions was checked in this Laboratory. The particular type of centrifugal pump available was found to be unsatisfactory for preparing emulsions inasmuch as the pump could not handle mixtures of sufficient viscosity. Two gear pumps, of different mechanical design, were tried out and found to be satisfactory. In this case also it was necessary to employ viscous mixtures. When gear pumps are used with more dilute mixtures a greater output is obtained but the material must be recirculated through the pump to produce an emulsion without any large particles.

B. Preparation of S-145 Dispersions

(1) Grinding S-145

18. Both the DPU and TCA types of the impregnite S-145 are produced as well defined crystals. The size of the individual crystalline particles range from 50 to 125 microns in diameter. Particles of this size settle out of water suspensions rapidly. It has been found necessary to reduce the particle size to about 1-10 microns before reasonably stable suspensions can be prepared. No thorough study of methods of grinding S-145 has been attempted. It was found that a ball mill could be satisfactorily employed for grinding the crystalline S-145 down to a proper size. The discovery by the DuPont group that the micronizing process can be used to grind S-145 has now solved the problem of grinding.

(2) Dispersing Agents for S-145

19. The problem of obtaining stable aqueous suspensions of S-145 is not solved by the reduction in particle size alone. Small discrete particles of S-145 will not exist as such in a water suspension unless a dispersing agent is employed. Surface forces between the small particles cause them to cling together in a state of flocculation. Dispersing agents eliminate the forces between particles ostansibly by forming a protective coating over each individual particle. Conditions of flocculation or deflocculation are usually quite obvious and can be most easily determined by microscopic examination. Many different surface active agents were examined as dispersing agents for S-145 and the results are given in Table III.

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Table III

Effectiveness of Dispersing Agents for S-145

Agent	Ratio Agent to S-145	Conc. S-145	Character of Slurry	Character of S-145	Size in Microns	Sedimentation
Crystalline S-145 ground and dispersed in a ball mill						
Triton NE	.03	50	Thick foam	Flocculated	—	None
Glue + Alkanol SA	.05 .001	33	Gelled	"	1-6	"
Triton NE + Gelatin	.01 .02	33	"	Well dispersed	2-6	Easily redispersible
" " + Methocel	.01 .01	33	—	Flocculated	—	" "
" " + Tragacanth	.01 .005	40	Solid foam	"	8	—
Triton NE + RH-403	.02 .02	25	—	Well dispersed	1-4	Hard cake
" " + Pectin	.02 .02	25	—	Flocculated	1-10	Easily redispersible
Emulphor AG	.01	25	Thick	"	—	—
" ELA	.01	25	"	"	—	—
Bentonite	.01	25	"	"	—	—
Silica Gel	.01	25	Thick	Flocculated	—	—
No agent	—	25	Thick, foamy	"	—	—
RH-403	.01	25	"	"	—	—
RH-403	.025	25	Mobile	Small flocs	1-8	Hard cake
RH-403	.05	25	"	Few small flocs	1-8	" "
RH-403	.10	25	Viscous	Few small flocs	1-20	" "
Hornkem	.01	25	Mobile	Well dispersed	1-10	Hard to disperse
"	.05	25	"	" "	—	Impossible to disperse
Turkey Red Oil	.05	25	Thick, foamy	Flocculated	—	—
Methocel 25 CPS	.01	25	Mobile	Well dispersed	2-10	Easy to disperse

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Table III
(continued)

Agent	Ratio Agent to S-145	Conc. S-145	Character of Slurry	Character of S-145	Size in Microns	Sedimentation
Methocel 25 CPS	.025	25	Mobile	Well dispersed	1-10	Hard cake
" " "	.05	25	"	" "	1-10	" "
" 400 "	.025	25	"	" "	1-10	" "
" 1000 "	.025	25	Viscous foamy	" "	1-10	Easy to disperse
Daxad 11	.01	25	Mobile	" "	1-10	Very hard cake
Daxad 11	.05	25	"	" "	1-10	" " "
Darvan #1	.05	25	"	" "	1-10	Hard cake
RH-403 + Daxad 11 .05	.01	25	"	Few small flocs	1-10	Semi-hard cake
Methocel + Daxad 11.025	/01	25	"	Well dispersed	1-10	Hard cake
Micronized S-145 dispersed in a colloid mill						
Daxad 11	.01	25-60	Mobile	Well dispersed	1-5	Hard cake
RH-403	.01	50	Foamy	Flocculated	-	- -
Methocel 15 CPS	.01	25-60	Mobile	Well dispersed	1-5	Hard cake

The results given in the table as well as other considerations have led to the conclusion that Methocel and Daxad 11 are the most effective dispersing agents for S-145. RH-403 may also be used but greater concentrations of this agent are required.

(3) Micronized S-145

20. The use of micronized S-145 for preparing aqueous suspensions and impregnating baths presented certain difficulties. The micronized material is difficult to wet out in water. The powder floats on the surface of the water and can be wet out only with considerable agitation. The use of wetting agents to lower the surface tension of water greatly facilitates wetting out of the S-145 powder but often large quantities of foam are also produced. When the powder has become wet out, deflocculation is completed by passing the mixture through a colloid mill.

21. It was reported by the NDRC group at the DuPont Experimental Station that micronized S-145 could be added directly to chlorinated paraffin emulsions. This eliminated the necessity for making a separate aqueous dispersion of the S-145 and has the added advantage in that the S-145 wets out more rapidly in chlorinated paraffin emulsions than in water.

C. <u>Plant Formulation Procedure</u>

22. The present policy of impregnating protective clothing in central plants for later distribution to Naval vessels has necessitated the development of suitable procedures for using aqueous impregnating systems in the impregnation plants. It is important that such additional equipment required by the plants in changing from the solvent to an aqueous process be as simple as possible. Studies made in the laboratory on the most feasible plant procedures were aided by knowledge of the results obtained in plant trials by the CWS at Edgewood Arsenal.

23. The most important item in the preparation of aqueous impregnating mixtures for plant use is the mechanical device used to prepare chlorinated paraffin emulsions and S-145 dispersions. A colloid mill is well suited for this purpose. On the other hand, the sucessful use in CWS plant trials of a gear pump represents a considerable simplification. A gear pump is not only more economical than a colloid mill but it requires much less supervision. The work done in the laboratory on plant procedures may be best summarized by giving the details of an actual plant run carried out at the Impregnation Plant, Brooklyn Naval Clothing Depot on March 4, 5 and 6, 1943. The equipment employed consisted of three open-end

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drums of 55-gallon capacity, a 1/8 HP portable type "Lightnin" mixer, and a Brown and Sharpe gear pump rated at 9 GPM powered with a 1 HP motor. The following procedure was employed:

> 45 lbs. of 10% RH-403 solution were made by stirring the RH-403 slowly into water at 90° C with a "Lightnin" mixer and continuing to stir for about two hours. Nearly all the RH-403 was in solution by that time. To the cooled solution was added 4-1/2 lbs. of water containing 0.9 lbs. of Daxad 11 and 0.18 lbs. of Duponol ME. After mixing until homogeneous, 90 lbs. of chlorinated paraffin was added slowly with stirring, and the stirring continued until the viscous mass was homoganeous. The mixture was passed through the gear pump twice, diluted with 130 lbs. of water and passed through the gear pump again. The globule size of the chlorinated paraffin at this point was 1-4 microns averaging about 2 microns. 132 lbs. of CC-3 (micronized S-145 containing 10% S-42) and 18 lbs. of S-42 were added slowly with stirring to the emulsion. After all the powders were added, stirring was continued until the mixture was fairly smooth. The concentrated bath was passed through the gear pump, diluted with enough water to make the final concentration of S-145 equal to 10% and pumped into the storage tank of the impregnator. Suits impregnated from this bath were apparently satisfactory both in respect to active chlorine content and appearance.

24. This procedure was repeated in another run except that the last 200 lbs. of water used for dilution contained blue dye and again the impregnation was considered satisfactory.

D. Shipboard Formulation Procedure

25. The development of aqueous impregnating procedures suitable in the field or on shipboard with no more equipment than a container and paddle has been investigated extensively by the NDRC group at the DuPont Experimemtal Station. At the present time it has not been established whether premade emulsions of chlorinated paraffin and aqueous dispersions of S-145 can be adequately packaged and kept in storage for long periods of time. Such systems would require only dilution with water before use. A more promising procedure is to package the dry ingredients for the impregnating bath and to prepare the impregnating bath as needed. This procedure as demonstrated by the DuPont group at this laboratory

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before pepresentatives of the Bureau of Ships consists of the following steps:

> Two gallons of water and contents of package No. 1 are put into an open-end 50-gallon drum. Package No. 1 contains 5 lbs. of S-42, 1.88 lbs. of granular type RH-403, and 0.09 lbs. of Duponol ME. This mixture is stirred for 15 minutes with a wooden paddle to dissolve the RH-403. The contents of package No. 2 are added slowly to the mixture in the drum. Package No. 2 contains 37-1/2 lbs. of chlorinated paraffin. As the chlorinated paraffin is added the viscosity of the mixture increases and stirring becomes more difficult. After the chlorinated paraffin has all been added, stirring is continued for 15 minutes. Stirring the viscous mixture until it becomes a pasty white is a very important step in the procedure and the quality of the chlorinated paraffin emulsion depends on how well the mixture is stirred. The next step consists of adding six gallons of water and the contents of package No. 3 to the chlorinated paraffin emulsion. Package No. 3 contains 50 lbs. of micronized S-145. The mixture is stirred for 10 minutes to wet out the S-145 and break up lumps. When the mixture has been stirred until it becomes a smooth paste, thirty gallons of water are added with stirring. The bath now contains 10% S-145 and can be used to impregnate clothing.

It has been more recently reported that ordinary RH-403 (not of the granular type) can be used satisfactorily when mixed with the stabilizer S-42. The presence of the S-42 acts to prevent formation of lumps of the RH-403 and makes it possible to dissolve the RH-403 with about 15 minutes of stirring.

26. Laboratory experiments have shown that Methocel may be used in place of RH-403. A suitable solution can be made by stirring the Methocel in water for 15 minutes. In this case, however, it is important that Daxad 11 and Duponol ME are both used in conjunction with the Methocel. The Daxad 11 acts as a protective agent for the chlorinated paraffin emulsion and aids in deflocculating the S-145 and S-42.

Part II. Stability of Aqueous Impregnating Systems.

A. <u>Hydrolytic Stablity</u>

27. It is well established that high humidities promote

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the loss of active chlorine from cloth impregnated with S-145. Therefore, it was necessary to determine how stable S-145 would be when dispersed in water. The data shown in Table IV indicates that S-145 is quite stable in water at normal temperatures and is only slightly less stable at 110°F after more than four months storage.

Table IV

Stability of S-145 (TCA) in Water
(50% S-145 ball-milled in water for 48 hrs.)

75°F		110°F	
Days	% Cl^+	Days	% Cl^+
1	6.70	1	6.66
7	6.58	7	6.60
30	6.60	30	6.70
142	6.71	142	6.46

28. An accelerated test of the hydrolytic stability of S-145 in the presence of added materials has been used. The results are given in Table V.

Table V

Percentage Loss of Cl^+ on Boiling 0.2 grams of Unmicronized S-145 in 100 ml. of Water for 5 Hours

S-145 (TCA; 13.8% Cl^+)	19.2%
+25% S-42	23.0
+3" x 3" shredded cloth	18.6
+25% S-42 + 3" x 3" shredded cloth	24.0
+20% RH-403	23.5
+20% Methocel	5.7
+20% Triton NE	74.1
S-145 (DPU; 13.5% Cl^+)	20.8
+25% S-42	44.2
+3" x 3" shredded cloth	37.6
+25% S-42 + 3" x 3" shredded cloth	55.6

In the case of S-145 TCA only the Triton NE had an appreciable effect upon the loss of active chlorine. As will be seen later, this effect of Triton NE is substantiated in tests of impregnated cloth. The low loss of active chlorine for Methocel is not believed significant. The S-145 DPU is evidently more sensitive to the presence of added materials since both the stablilzer S-42 and cotton cloth caused an increase in the loss of active chlorine.

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29. It should be pointed out that the loss of active chlorine in the 5-hour boiling test is affected by the particle size of the S-145. Micronized S-145 shows a much greater loss than the crystalline S-145. A study is now in progress on the effect of S-145 particle size in this and other tests.

B. Thermal Stability

30. Table VI shows the stability of S-145 (TCA) stored at 100°C in the presence of 10% of various materials. The only material seriously affecting it is S-42. At the end of 48 hours nearly all the Cl^{+} is gone.

Table VI

Stability of S-145 (TCA) stored at 100°C in the presence of 10% of various agents

% Orig. Cl^{+} Content	Added Agent	% Cl^{+} retained: 24 hrs.	48 hrs.	72 hrs.	96 hrs.
13.75	None	91.4	80.9	84.7	79.3
13.05	Methocel	92.9	78.5	84.3	77.7
12.25	RH-403	89.9	80.0	78.0	63.8
12.80	S-42	35.2	3.8	3.8	0.7
11.30	MgO	96.5	96.0	87.2	84.5
12.90	Daxad 11	91.5	85.3	83.0	76.7
13.30	Darvan #1	89.0	83.0	81.7	78.8
12.65	Hornkem 101	94.5	88.5	89.0	74.2

The knowledge that S-42 had a very detrimental effect upon S-145 at 100° C coupled with the fact that S-42 and S-145 were being blended in the micronizing process as a means of preventing deterioration of fibre containers, led to a study of the effect of S-42 upon the loss of active chlorine of S-145 at different temperatures. The results are shown in Table VII.

Table VII

The Effect of S-42 on Micronized S-145 at Different Temperatures

Days	45°C % S-42: 0%	10%	25%	70°C % S-42: 0%	10%	25%	100°C % S-42: 0%	10%	25%
	% Active Chlorine Retained								
1	100	99.5	100	95.4	98.4	96.9	66.2	85.7	42.7
2	100	99.5	100	95.3	99.0	99.0	57.5	0.0	0.0
5				93.0	97.4	95.5	36.6	-	-
9				91.4	96.5	94.4	29.7		
28	97.6	98.0	96.4	84.4	92.0	85.0	16.8		
39	97.6	97.6	96.4	82.1	89.0	73.8	12.8		

[redacted]

It may be concluded that the presence of S-42 has no harmful effects on S-145 at temperatures likely to be encountered on storage.

C. Storage Stability

31. The foregoing data indicated that S-145 was sufficiently stable both as a dry powder and as a water dispersion. It had not been established that complete impregnating mixtures would likewise be stable. Several different impregnating mixtures were prepared and stored at room temperature for one month. The results of active chlorine determinations after two and four weeks are shown in Table VIII. Great difficulty was encountered in obtaining uniform samples for analyses since the S-145 had settled out and was difficult to redisperse. It is not believed that significant losses of active chlorine occurred.

Table VIII

Stability of Impregnating Mixtures Stored At Room Temperatures

(Every mixture contained 10 parts of S-145, 7.5 parts of chlorinated paraffin, and 88 parts of water plus the material listed)

Impregnite S-145	Parts, Emulsifying and Dispersing Agent		Parts S-42	Cl^+ Retained 2 wks.	4 wks.
TCA	1.75	RH-403	0	104	97
"	1.75	"	1	99	95
"	1.75	"	2	95	96
"	1.75	"	3	101	88
DPU	1.75	RH-403	0	97	99
"	1.75	"	1	94	93
"	1.75	"	2	96	100
"	1.75	"	3	85	88
DPU	1.66	Triton NE*	0	98	75
TCA	1.66	" *	0	100	104
DPU	1.66	Igepal CA*	0	98	106
TCA	1.66	" *	0	94	85

*0.05 parts of Methocel added as dispersing agent.

It has also been found that the pH of both chlorinated paraffin emulsions and S-145 dispersions decreases rapidly on storage unless S-42 is present. This is illustrated in Table IX.

[redacted]

Table IX

p H of Emulsions and Slurries on Aging
(Each emulsion contained 40% CP and 10% of the agent mentioned)

Agent	%S-42	Treatment	Orig.	1 wk	2 wk.	3 wk.	4 wk.
Triton	0	Rm. temp.	5.0	2.8	2.9	2.9	2.9
Igepal	0	" "	-	2.7	2.5	2.5	2.5
Triton	0	Frozen/heated alternately	-	2.5	2.3	2.4	2.4
Triton	2-1/2	Frozen/heated alternately	6.5	6.4	6.3	6.4	6.5
Igepal	0	" " "	3.3	3.0	2.5	2.6	2.5
Igepal	2-1/2	" " "	6.9	6.4	6.2	6.6	6.7

(Each slurry containing 10% S-145 (TCA) and 0.5% of the agent mentioned)

Agent	S-42	Orig.	1 wk.	2 wks.
None	0	4.0	2.5	
RH-403	0	4.3	2.9	2.9
Igepal	0	4.0	2.5	2.5
Triton NE	0	3.6	2.2	2.1
" K-60	2.5	6.6	6.6	6.5
RH-403	2.5	6.6	6.6	6.5

The low pH of the emulsions and dispersions not containing S-42 indicates that glass containers would be required for the storage and shipment of these systems.

D. Accelerated Storage Tests.

32. An accelerated storage test was devised for emulsions, dispersions, and complete impregnating mixtures. The test consisted of the following steps; freezing the sample for 16 hours, followed by eight hours standing at room temperature, heating for 16 hours at 70°C, followed by eight hours standing at room temperature. The samples were examined at the end of the eight-hour periods when they had come to equilibrium with room temperature.

33. Emulsions, both high and low concentrations, were made with Methocel, RH-403 and Triton NE. These were subjected to the test described above. All three agents made emulsions which were stable after six cycles. The dilute emulsions thawed out without apparent change in texture or particle size; the concentrated emulsions were dispersible as before the treatment.

[redacted]

34. Concentrated slurries (75% solids; micronized S-145 with and without 25% S-42 + Daxad 11) were treated in the same fashion. In no case was it possible to redisperse the slurries after four cycles. They had hardened to a solid mass. After two cycles they were still redispersible although difficultly so.

35. These slurries were blended with the concentrated emulsions and subjected to the test. In this case, the mixtures were again found not to be redispersible after four cycles, the Triton mixture failing after the first cycle.

36. These results are taken as evidence that such concentrated slurries and impregnating mixtures would not be suitable for service use.

37. On analyzing both the slurries and complete impregnating mixtures as long as they could be redispersed, no significant active chlorine loss was detectable. This result, however, must be qualified because of possible loss of water during the heating part of the cycles. Also it is doubtful whether perfect sampling was possible under conditions of difficult dispersibility.

Part III. Impregnation from Aqueous Systems.

A. <u>Impregnite Retention by the Cloth</u>

38. Five main factors have been found which affect the amount of impregnite retained by cloth upon impregnation. These are: (1) Concentration of impregnite, (2) Particle size of the impregnite, (3) Viscosity of the impregnating bath, (4) Finish of the cloth, and (5) Method of removing excess liquid. Two of these factors, the concentration of the impregnite and the viscosity of the impregnating bath are closely related. An increase in concentration results in an increase in viscosity. In Table X are shown the results obtained when the concentration of S-145 in the impregnating bath was varied from 10 to 25%. The active chlorine content was determined for cloth samples impregnated with a padding machine at a constant roller pressure.

Table X

Effect of S-145 Concentration Upon Active Chlorine Pick-up

% S-145 in bath	Active Chlorine on Cloth, mg./cm.^{2}
10	.20-.30
16	.40-.60
18	.55-.70
25	.95

[redacted]

It has been found that when Methocel (15 cps) is used in place of RH-403 for emulsifying the chlorinated paraffin, the resulting impregnating mixtures have different viscosities. A 10-20% greater active chlorine content is obtained with a mixture containing Methocel instead of RH-403. Table XI illustrates the effect of using different concentrations of RH-403 in an impregnating bath. The baths containing higher RH-403 concentrations have a higher viscosity and therefore result in a slightly greater retention of active chlorine.

Table XI

The Effect of RH-403 Concentration upon Active Chlorine Pick-up

% RH-403 in bath	Active Chlorine on Cloth. mg./cm^2
4	.52
8	.59
16	.63

39. The effect of the impregnite particle size upon the retention of impregnite can be illustrated by the following experiment: 10% suspensions of S-145 treated in different ways were filtered through a disc of Arnzen cloth held on a Buchner funnel. In every case where the S-145 was not finely ground and well deflocculated a layer of the S-145 was retained on the cloth. When micronized S-145, which had been well deflocculated, was used no filtering action was shown on the cloth.

40. A series of impregnations was carried out with impregnating baths which had been prepared with samples of S-145 carefully fractionated into different particle size ranges. Normal impregnite contents were obtained upon impregnation with all samples of a particle size range of 10 microns or less. The larger particle sizes did not give satisfactory impregnations since lower active chlorine contents were obtained and the impregnite seemed to be predominantly on the surface of the cloth. The relatively tight weave of Arnzen cloth apparently does not permit particles larger than 10 microns to penetrate between the fibres of the cotton threads.

41. It has been established that the finish of Arnzen cloth as received from the finishing plant is less suitable for aqueous impregnations than cloth that has been given further treatment. Laundering increases the active chlorine pick-up more than 40%. However, it has been demonstrated that protective suits of Arnzen cloth can be satisfactorily impregnated by both plant and shipboard methods and therefore,

[redacted]

the problem of cloth finish is not considered serious.

42. The three most commonly employed methods of removing excess liquid from cloth soaked in impregnating baths are: hand wringing, mechanical wringing with squeeze rolls, and centrifugal extraction. Of the three, hand wringing is the most subject to variation. Satisfactory active chlorine contents can be obtained on Arnzen suits by hand wringing when a bath containing 10% S-145 is used. The amount of liquid removed by mechanical wringing depends on the pressure between the rollers but in general a more concentrated bath is required. As for centrifugal extraction, it may be stated that satisfactory active chlorine contents were obtained on Arnzen suits impregnated in a plant run where the bath was 9% S-145 and the extraction was carried out for four one-minute periods at 200 rpm.

43. The data obtained at the plant trials of the water impregnation process at Brooklyn were insufficient to establish exact figures on impregnite retention. It may be stated, however, that with bath concentrations of 8-10% S-145 the active chlorine content of Arnzen suits was between 0.4 and 0.6 mg./cm.^{2}. Arnzen suits are impregnated by the solvent process in the Brooklyn plant to contain 0.3 to 0.4 mg. Cl^+/cm.^{2}.

B. <u>Uniformity pf Impregnation</u>

44. It is important from the standpoint of laboratory investigations that cloth be as evenly impregnated as possible. Results of subsequent tests of impregnated cloth are more difficult to interpret if the active chlorine content of the cloth is not uniform. The following procedure has been found most satisfactory for laboratory use. Arnzen cloth is laundered and given a final rinse in water containing 0.1% Aerosol OT. Swatches of dry cloth treated in this manner are then dipped into the aqueous impregnating bath and passed through the rolls of a padding machine. The soaking and wringing are repeated from three to five times to insure a thorough penetration of the cloth. Cloth swatches impregnated in this manner and analyzed at random generally show an average deviation from the mean of less than 5%.

45. It is important from the standpoint of adequate protection as well as economy that garments be uniformly impregnated. Garments impregnated by the field or shipboard procedures where hand wringing is employed show large variations in impregnite content. This is true for hand impregnations by both the solvent and aqueous process. In view of the fact that such impregnations will be used primarily for emergencies, the lack of uniformity of

impregnation is not considered a serious problem.

46. The magnitude of the variations of active chlorine content of suits impregnated by the aqueous process in a plant run is shown in Table XII. Suits were chosen at random from three different batches and analyzed as shown below. It is concluded that plant impregnations by the aqueous process are fully as satisfactory in regard to uniformity of impregnation as plant impregnations by the solvent process.

Table XII

Active Chlorine Analyses of Suits Impregnated by The Aqueous Process in the Impregnation Plant, Brooklyn Naval Clothing Depot

Active Chlorine in mg./cm.^{2}

Elbow	Shoulder	Waist	Crotch	Knee	Cuff	Average
		First Batch				
.53	.51	.50	.58	.48	.50	.51
.57	.67	.83	.51	.42	.50	.58
.57	.46	.49	.56	.50	.54	.52
.55	.63	.54	.54	.60	.58	.57
.60	.48	.63	.48	.62	.49	.55
.56	.54	.60	.53	.53	.52	.55
		Second Batch				
.48	.53	.40	.51	.39	.45	.45
.37	.44	.49	.46	.56	.53	.47
.54	.53	.78	.44	.43	.47	.53
.33	.61	.51	.51	.32	.34	.44
.47	.42	.54	.51	.45	.42	.47
.40		.62	.41	.47	.62	.51
.42	.52	.56	.47	.43	.47	.48
		Third Batch				
.59	.48	.51	.61	.55	.56	.55
.56	.61	.51	.40	.48	.60	.52
.50	.49	-	.62	.43	.47	.50
.54	.54	.56	.46	.35	.45	.48
.58	.64	.58	.48	.54	.52	.55
.55	.55	.54	.51	.46	.52	.52

C. Preferential Absorption of Components from the Impregnating Bath

47. This factor is most important from the standpoint of plant impregnations where large batches of impregnating mixtures are prepared and used over a period of time. This Laboratory has no data available on preferential absorption of components from impregnation baths in plant runs. However, the following evidence has been obtained in the course of laboratory studies. Two series of impregnations were carried out in which twenty cloth swatches were successively impregnated. One bath contained S-145 and chlorinated paraffin while the other also contained the stabilizer S-42. The amount of impregnating bath used was such that after the last swatch had been impregnated the bath was practically exhausted. The increase in active chlorine content and dry weight pick-up of the successive cloths is shown in Plate I. The evidence is clear that water was preferentially absorbed from the impregnating bath with the result that the bath became more concentrated after successive impregnations.

48. The question of preferential absorption of chlorinated paraffin emulsion from aqueous impregnating baths has been raised but as yet no satisfactory method of analysis has been devised.

49. Several series of impregnations were carried out in impregnating mixtures containing different concentrations of the stabilizer S-42. The results are shown in Table XIII. The fact that the ratio of S-42 to S-145 on the cloth is greater than the ratio in the impregnating bath is evidence that some S-42 was preferentially absorbed by the cloth. The behavior may be attributed to the fact that the S-42 is more finely divided than the S-145 and that the cloth exhibits a slight filtering action on the S-145.

Table XIII

Preferential Absorption of S-42

S-42 in bath % of S-145	S-42 on cloth % of S-145	% excess S-42 on cloth
5	7	40
10	13	30
17	23	35
25	29	16
50	56	12

[redacted]

It should be pointed out that the data presented above from laboratory experiments will have to be investigated in plant trials conducted over a period of time. However, if any preferential absorption is observed in plant impregnations it could be readily corrected by necessary adjustments of the impregnating bath during the course of successive impregnations.

D. Permeability and Handle of Impregnated Cloth

50. The permeability or air resistance of impregnated fabrics had been used as a measure of wearing comfort. Cloth samples impregnated in the laboratory as well as suits impregnated in plant trials have been tested. The results obtained showed that cloth impregnated by the aqueous process is fully as permeable as cloth impregnated by the solvent process. In both cases the air resistance is approximately double the value for unimpregnated cloth.

51. The appearance and handle of cloth impregnated by aqueous systems have been found to depend on the concentration and type of emulsifying and dispersing agents employed. The formula first recommended by the NDRC group at the DuPont Experimental Station contained RH-403 to the extent of 17-1/2% by weight of the S-145. Polyvinyl alcohol is a film-forming material and cloths impregnated in this system became quite stiff and boardy upon drying. The use of tumbler driers in plant impregnations considerably reduced the stiffness of impregnated garments but they still had a harsh feel. The formula recommended by this Laboratory contains RH-403 and Daxad 11 to the extent of 3.75 and 0.75, respectively, based on the weight of S-145. At this concentration of RH-403, impregnated cloth does not become stiff upon drying. A series of impregnations carried out with different concentrations of RH-403 have shown that amounts up to 10% of the S-145 can be tolerated. Methyl cellulose is similar to polyvinyl alcohol in that it causes undue stiffness and harshness at high concentrations. Other emulsifying agents such as Triton NE are without any film forming tendencies and do not affect the handle of the cloth.

52. Arnzen suits impregnated by the aqueous process compare favorably with solvent impregnated suits except that they are wrinkled and have an un-ironed appearance. When predyed garments are impregnated by the aqueous process they become considerably lighter in shade and contain many light streaks and spots. The Navy practice of simultaneously dyeing and impregnating obviates these difficulties.

Part IV. Performance Tests of Cloth Impregnated by Aqueous Systems.

[redacted]

A. Accelerated Storage and Weathering

53. The behavior of cloth impregnated by the aqueous process has been studied in respect to the following storage and weathering tests: (1) Accelerated storage test 75°C-75% relative humidity, (2) The sealed tube test at 70°C-100% relative humidity, (3) Tropical storage at 110°F-75% relative humidity, and (4) Outdoor exposure in Florida. The first two tests are accelerated tests and are used primarily as laboratory methods for quick evaluations. The tropical storage and outdoor exposure tests are considered more indicative of actual service conditions of storage and wear.

(1) Accelerated Storage Test at 75°C-75% R. H.

54. The accelerated storage test at 75°C-75% R. H. has been employed as the most useful method of obtaining comparative values of impregnite stability in a relatively short time. A 96-hour exposure in this test usually results in an active chlorine loss of about 50% and a tensile strength loss of 20% or more (when no stabilizer is used) for the impregnite S-145 (TCA).

55. It has been found that the percentage of active chlorine lost in this test is dependent upon the initial active chlorine content of the cloth samples. Several small discs of cloth were impregnated in a standard aqueous system in such a manner as to obtain a variation in impregnite concentration. After 24, 48, 72 and 96-hour periods of exposure in the accelerated test entire discs were removed and analyzed for active chlorine. The results are given in Table XIV.

Table XIV

Effect of Initial S-145 Concentration upon the Loss of Active Chlorine in the Accelerated Storage Test 75°C-75% R. H.

Orig. Cl^+ mg./$cm.^2$	Percentage Cl^+ retained			
	24 hr.	48 hr.	72 hr.	96 hr.
0.41	69	50	34	37
0.47	74	54	42	47
0.56	80	64	49	48
0.62	81	72	54	50

It is apparent that the percentage retention of Cl^+ increases with the loading of the cloth. It has been observed that the amount of impregnite decomposed in a given period of time is approximately constant and independent of initial concentration. More evidence must be obtained to definitely

[redacted]

establish this as a fact. Upon the basis of the above results an endeavor has been made to use only those cloth samples which contain approximately 0.5 mg. $Cl^+/cm.^2$ for accelerated storage tests.

56. A comparison of three emulsifying agents, RH-403 Methocel, and Triton NE in regard to their effect upon impregnite stability has been made. The results are shown in Table XV.

Table XV

Comparison of Emulsifying Agents in Accelerated Storage 75°C-75% R. H.

Emulsifying Agent % S-145	% Cl^+ retained		% Tensile retained	
	48 hr.	96 hr.	48 hr.	96 hr.
RH-403, 17-1/2	66	60	88	81
RH-403, 17-1/2 (+10% S-42)	79	60	99	88
Triton NE, 17-1/2	42	29	86	70
Triton NE, 17-1/2 (+10% S-42)	40	25	87	74
Methocel, 8-3/4	69	63	93	90
Methocel, 8-3/4 (+10% S-42)	65	62	97	93

It is evident that Triton NE causes undue loss of active chlorine. This behavior is confirmed by the results for another series of impregnations wherein different concentrations of the stabilizer S-42 were used. These results are shown in Table XVI. For comparison, average values that have been previously obtained for cloth samples impregnated by the solvent process are included at the bottom of the table. It can be seen from these results that the stability of S-145 on cloth samples impregnated by aqueous systems containing either RH-403 and Methocel is essentially the same and furthermore is equivalent to the stability of S-145 impregnated by the solvent process. The effect of the stabilizer S-42 upon the loss of active chlorine in the accelerated storage test is negligible.

Table XVI

Effect of Emulsifying Agents and Varied S-42 Concentrations upon S-145 in Accelerated Storage at 75°C-75% R.H.
(Concentration of emulsifying agents 6% by weight of S-145)

Emulsifying Agent; Amt. of S-42*	$\%Cl^+$ retained		% Tensile Retained	
	48 hr.	96 hr.	48 hr	96 hr.
RH-403		52		77
RH-403, 5% S-42		73		87
RH-403, 10% S-42		61		88
RH-403, 25% S-42		55		87
Triton NE		31		78
Triton NE, 5% S-42		38		83
Triton NE, 10% S-42		26		87
Triton NE, 25% S-42		21		90
Methocel		68		78
Methocel, 5% S-42		66		90
Methocel, 10% S-42		54		91
Methocel, 25% S-42		55		90
Values for Samples Impregnated by Solvent Process				
None	77	60	92	85
None, 10% S-42	70	52	94	88
None, 25% S-42	86	67	91	95

*Amount of S-42 based on weight of S-145

57. This is further demonstrated by the results obtained for a series of samples impregnated from mixtures containing RH-403 as the emulsifying agent with various concentrations of S-42. The results are shown in Table XVII.

Table XVII

Effect of S-42 upon S-145 Stability in Accelerated Storage 75°C-75% R.H.
(3.75% RH-403 as emsulfying agent)

S-42 as % of S-145	Orig. mg. Cl^+/cm^2	$\% Cl^+$ retained		% Tensile Retained	
		48 hr.	96 hr	48 hr.	96 hr.
0	0.60	97	89	82	75
5	0.62	92	72	83	84
10	0.60	100	76	85	85
17	0.55	93	73	90	86
25	0.60	96	72	85	91
50	0.65	92	74	88	88

[redacted]

The effect of S-42 on the tensile strength retention of samples exposed in the 75°C-75% R.H. test is evident when samples containing no S-42 are compared to samples containing S-42. However, no important differences in tensile strengths can be detected between different concentrations of S-42. The retention of active chlorine for the 0% S-42 in this particular series is abnormally high and not significant (cf Table XV and XVI)

(2) The Sealed Tube Test at 70°C-100% R.H.

58. This test is also used as a laboratory method for rapid evaluation of impregnated cloth samples. Since the cloth samples are kept in a sealed system, volatile decomposition products of the impregnite do not escape and can cause further decomposition of the impregnites. For this reason the test is believed to be somewhat indicative of baled storage conditions where very little ventilation occurs. A comparison of the three emulsifying agents, RH-403, Methocel, and Triton NE, as shown in Table XVIII again indicate that Triton NE is unsatisfactory.

Table XVIII

Effect of Emulsifying Agents upon S-145 Stability
Sealed Tube Test at 70°C-100% R.H.

Agent, S-42 as % of S-145	% Cl^+ retained		% Tensile retained	
	48 hr.	96 hr.	48 hr.	96 hr.
RH-403	0	0	2	2
RH-403 10% S-42	22	-	84	81
Triton NE	0	0	2	7
Triton NE, 10% S-42	4	0	84	74
Methocel	0	0	6	3
Methocel, 10% S-42	31	12	78	39

59. The presence of a stablizer such as S-42 is much more marked in the sealed tube test than in the accelerated storage test at 75°C-75% R. H. This is illustrated further in Table XIX. The data in this table clearly show the stabilizing action of S-42 both in regard to loss of active chlorine and loss of tensile strength. Furthermore, it is apparent that 5% S-42 is not sufficient to neutralize all the S-145 decomposition products. Concentrations of 10% or more of S-42 are substantially equivalent in this test both in regard to stabilizing action on the impregnite and prevention of cloth deterioration.

[redacted]

Table XIX

Stabilizing Action of S-42 in Sealed Tube
Test 70°C-100% R. H.

Agent, S-42 as % of S-145	% Cl⁺ retained 48 hr.	96 hr.	% Tensile retained 48 hrs.	96 hr.
RH-403	39	3	24	5
RH-403, 5% S-42	67	11	76	10
RH-403, 10% S-42	40	30	81	67
RH-403, 17% S-42	62	25	49	71
RH-403, 25% S-42	46	23	87	75
RH-403, 50% S-42	43	18	82	79
Methocel	47	0	22	3
Methocel, 5% S-42	58	11	83	11
Methocel, 10% S-42	45	36	85	72
Methocel, 17% S-42	56	26	89	80
Methocel, 25% S-42	40	26	81	53
Methocel, 50% S-42	41	22	87	76

(3) The Tropical Storage Test at 110°F-75% R. H.

60. The tropical storage test at 110°F-75% R. H. requires exposure periods of several months for completion. For that reason it is not well suited for use in laboratory development work where more rapid results are necessary. The data presented in Table XX for several different aqueous impregnations substantiate the data obtained in the accelerated tests on the suitability of the three emulsifying agents considered. It is evident that Triton NE is detrimental to S-145. On the other hand, the effect of S-42 in the tropical storage test as conducted on these samples (with ventilation) is not marked. Presumably this is because the slow rate of impregnite decomposition coupled with the rapid ventilation rate does not permit impregnite degradation products to act on the cloth.

Table XX

Stability of S-145 on Cloth Samples Impregnated by
Aqueous Process Tropical Storage Test at 110°F-75% R. H.
Ventilation, 5 cu.ft./min. through 120 cu.ft. chamber

System	% Cl⁺ retained 30	60	90	120	% Tensile retained (days) 30	60	90	120
RH-403	73	60	43	-	96	99	88	-
RH-403, 10% S-42	93	75	67	-	97	95	95	-
RH-403, 25% S-42	94	78	70	-	100	96	100	-
Triton NE	80	45	38		86	87	78	-
Methocel	88	75	-	54	95	95	-	89
RH-403, Khaki dye	45	29	20	18	85	87	85	84

(4) Outdoor Exposure.

61. Outdoor exposure tests of cloth swatches were carried out during February 1943 at Miami, Florida. In these tests the cloth samples are given a southern exposure at 45° for 24 hours a day. Samples were examined after one, two, three and four weeks exposure for loss of active chlorine and loss of tensile strength. The results are given in Table XXI.

62. Several conclusions can be drawn from these results. First, the loss of active chlorine is much more rapid during the first week than during successive weeks. This behavior has also been observed for solvent-impregnated samples exposed outdoors. It is attributed to a shielding effect of the cloth or impregnite decomposition product which serves to prevent ultra-violet rays from reaching undecomposed impregnite. The somewhat higher chlorine retention for the samples containing the khaki dye are believed significant and also attributable to shielding action. Second, the effect of the different S-42 concentrations upon the loss of tensile strength is important. The 5% sample was good after one week exposure but failed in succeeding weeks. 10% S-42 gave good protection for three weeks but broke in the fourth week.

Table XXI

Outdoor Exposure of Cloth Samples Impregnated with S-145 from Aqueous Systems, Miami, Florida, February 1943.

System	Orig. mg. $Cl^+/cm.^2$	% Cl^+ retained				% Tensile retained			
		1	2	3	4	1	2	3	4wks.
RH-403	0.63	55	38	39	32	52	29	22	17
RH-403, 5% S-42	0.59	52	36	34	30	88	49	38	29
RH-403, 10% S-42	0.61	49	45	37	29	92	94	85	57
RH-403, 17% S-42	0.61	54	48	38	28	90	89	95	84
RH-403, 25% S-42	0.62	55	47	40	29	94	97	98	98
RH-403, 50% S-42	0.65	63	44	39	38	92	94	95	94
Methocel	0.67	55	33	31	-	50	31	25	-
Methocel, 25% S-42	0.66	68	48	44	37	91	94	97	92
RH-403, blue dye	0.53	57	36	48	35	54	30	24	17
RH-403, blue dye, 25% S-42	0.56	60	33	36	30	94	94	95	93
RH-403, khaki dye	0.53	60	48	48	51	67	35	29	24
RH-403, khaki dye 25% S-42	0.56	68	49	48	49	94	97	100	97

[redacted]

63. In connection with the above tests, an independent method of establishing the optimum S-42 concentration has been devised. The method consists of analyzing cloth samples for basic S-42, i.e., S-42 which has not been converted to the chloride. The analysis is based on an acid extraction of the cloth sample followed by back titration with base. The details of the method are described in another report (now in preparation) on the stabilizer S-42. The results of the basic S-42 analyses on those samples exposed outdoors are given in Table XXII.

Table XXII

Effect of Outdoor Exposure on the Basic S-42 Content of Cloth Impregnated with S-145

System	Basic S-42 in $mg./cm.^2$		
	Orig.	2 weeks.	4 weeks.
RH-403, 5% S-42	0.31	0.00	0.00
RH-403, 10% S-42	0.57	0.07	0.0
RH-403, 17% S-42	0.92	0.38	0.15
RH-403, 25% S-42	1.27	0.69	0.49
RH-403, 50% S-42	2.63	1.74	1.29

64. The sample containing 5% S-42 contained no basic S-42 after two weeks' exposure. The 10% S-42 sample still had an appreciable amount of basic S-42 after two weeks but had none after four weeks. The 17% sample still contained some basic S-42 after the four week exposure. Upon the basis of these results it is believed that 25% S-42 should be recommended rather than any lower percentage.

B. Resistance to Vesicant Vapors

(1) Comparison of Aqueous and Solvent Methods of Impregnation.

65. To determine the degree of protection afforded by clothing impregnated from water dispersions of S-145, a number of HS and M-1 vapor penetration tests have been conducted on samples of cloth impregnated by this method. The samples were tested by the NRL static penetration method previously described in NRL Report No. P-1931 and the results obtained are given in Table XXIII.

[redacted]

Table XXIII

HS and M-1 Vapor Penetration Tests

Impregnation Method	Vesicant Agent	Protection time (min.) mg.Cl^+/cm.^{2}	Protection time (min.) mg.S-145/cm^2	% Cl^+ Remaining after test
Solvent	HS	407	59	2.3
Aqueous	HS	766	111	0.6
Solvent	M-1	45	7	1.8
Aqueous	M-1	100	15	9.2

66. It may be seen from the data in the above table that the protective capacity (per unit weight of Cl^+ or S-145) of clothing impregnated by the aqueous process is about twice that of clothing impregnated by the solvent process. This is true as regards protection against both HS and M-1 vapor penetration. As shown later, indications are that this may be due to the smaller particle size of the S-145 when impregnated by aqueous processes.

(2) Variations in HS Penetration Results for Aqueous Impregnations.

67. The HS protective value of 111 min./mg. impregnite/cm.^{2} for cloth impregnated by an aqueous process is the average of a large number (34) of individual determinations. Values for individual determinations ranged from 70 to 149 minutes with 83% of the values being from 90 to 120 minutes. This is a greater variation than is observed in the case of samples impregnated from tetrachloroethane solutions and cannot be explained entirely by variations in the uniformity of impregnation. Variations in particle size may be responsible to some extent. Table XXIV shows HS vapor penetration data obtained for two samples for which the uniformity of impregnation was known.

Table XXIV

HS Vapor Penetration Tests on Samples of Known Uniformity of Impregnation

mg.Cl^+/cm.^{2}	Uniformity of Cl^+%	Protective Time (min.) mg.Cl^+/cm.^{2}	Protective Time (min.) mg. S-145/cm.^{2}	% Cl^+ Remaining after test
0.341	± 5.0	880	129	0.0
0.341	± 5.0	627	91	0.0
0.341	± 5.0	627	91	0.0
0.342	± 7.6	717	104	0.0
0.342	± 7.6	724	105	0.0
0.342	± 7.6	841	122	0.0
0.342	± 6.3	736	107	0.0

[redacted]

68. The above data show protective values of 91 to 129 min./mg. S-145/cm.^{2} with an average value of 107. This average value is in good agreement with that given in Table XXIII. Whereas the uniformity of impregnation is ± 6.3% the average deviation in protective value is ± 11.6%.

(3) HS Resistance of Samples after Storage and Weathering.

69. To determine the effect of storage and weathering on the protective efficiency of aqueous impregnated clothing, HS vapor penetration tests were made on samples exposed for 96 hours accelerated storage at 75°C-75% relative humidity and also on samples exposed two weeks outdoors in Florida (cf IV A.)

70. The data obtained in these tests are given in the following table along with that for unexposed samples. Comparable data for solvent impregnated samples are also presented.

Table XXV

HS Vapor Penetration Tests after Storage and Weathering

Impregnation Process	Storage or Weathering	Protective Time (Min.) mg. Cl+/cm.^{2}	mg. S-145/cm.^{2}	% Cl+ remaining after test
Solvent	none	407	59	2.3
Aqueous	"	766	111	0.6
Solvent	75°C-75% R. H.	386	56	2.2
Aqueous	" " "	505	73	3.9
Solvent	Outdoor (Fla.)	415	60	0.0
Aqueous	" "	806	117	1.1

71. The protective efficiency of S-145 is the same before and after exposure to the weathering and accelerated storage tests on cloths impregnated by the solvent process. The protective efficiency of the S-145 on cloths impregnated by the aqueous process is the same after two weeks outdoor exposure in Florida, but is considerably lower after the 96 hour accelerated storage test. The drop in efficiency is from 111 to 73 min./mg. impregnite/cm.^{2}, the latter value being only slightly higher than the value for solvent impregnations (56 minutes). This lowering may be due to an increase in the particle size of the S-145 during the storage test.

(4) The Effect of Polyvinyl Alcohol (RH-403) on HS Vapor Penetration Tests.

72. Whereas the RH-403 used as an emulsifying and dispersing agent in the Aqueous impregnation systems has no inhibiting effect on the measured protective capacity of impregnated cloth, it was observed that the presence of this agent caused a marked effect in the manner and rate of HS penetration. Plate II shows three HS penetration curves. No. 1 is the curve obtained for penetration of HS through a representative cloth impregnated by the solvent process, No. 2 is the curve obtained for a representative aqueous impregnation cloth with RH-403 used as the emulsifying agent, and No. 3 is that obtained for an aqueous impregnation cloth with Triton NE used as the emulsifying agent. Complete HS penetration test data for these three samples are given in Table XXVI.

Table XXVI

HS Vapor Penetration Tests - Effect on RH-403

Impregnation Process	mg. Cl /cm.^{2}	Bubbler Times (Minutes)								
		1	2	3	4	5	6	7	8	9
Aqueous-RH-403	0.424	34.0	38.5	35.5	37.6	38.4	41.1	40.8	29.0	20.0
Solvent	0.358	67.4	39.7	32.0	18.2	10.0	-	-	-	-
Aqueous-Triton NE	0.343	75.5	62,0	54.5	15.2	11.0	-	-	-	-

73. It may be observed from the data in Table XXVI and from Plate II that the presence of RH-403 causes a higher initial leakage of HS which is maintained for a considerable number of bubblers before the break-point concentration of 0.05 mg./l. HS in the effluent air stream is reached. In the absence of RH-403 this break-point concentration is normally attained at the end of five or less bubblers. This corresponds to a penetration of 1.0 mg. HS before the break-point (0.05 mg./l.) concentration is attained. With RH-403 present nine or more bubblers are required, which means that 1.8 or more mg. HS penetrate the cloth before the break-point is reached.

74. To study the effect of concentration on the "polyvinyl alcohol effect," HS vapor penetration tests were made on a series of samples in which the concentration of RH-403 was varied from 0 to 23-1/4%(by weight of S-145). The data obtained in these tests are given in Table XXVII.

[redacted]

Table XXVII

Effect of RH-403 Concentration on "Polyvinyl Alcohol Effect"

RH-403 Conc. (% of S-145)	No. of Bubblers	Total HS Penetration before Break-point (mg.)	Protective Time (Min.) mg. Cl+ /cm.2	Protective Time (Min.) mg. S-145/cm.2	%Cl+ remaining after test
3-1/4	10	2.0	842	122	0.0
17-1/2	12	2.4	750	109	0.5
13-3/4	10	2.0	795	115	1.0
8-3/4	11	2.2	660	96	0.8
6-1/4	7	1.4	718	104	1.9
3-3/4	6	1.2	779	113	2.2
0	5	1.0	819	119	0.0

In Table XXVII it is shown that the protective efficiency of the impregnated samples is unaffected by the concentration of RH-403 since all values are within the range of 90-120 min./mg. S-145/cm.2. However, the "polyvinyl alcohol effect" as signified by the number of bubblers before the "break-point," decreases markedly below concentrations of 8-3/4%. The effect is relatively constant above 8-3/4%.

75. A further study of the "polyvinyl alcohol effect" was made by subjecting a number of impregnated samples containing 17-1/2% RH-403 to various methods of drying after impregnation. HS vapor penetration tests were made on these treated samples with the following results:

Table XXVIII
Effect of Drying Treatments on "Polyvinyl Alcohol Effect"

Treatment	No. of Bubblers	Total HS Penetration before Break-point (mg.)	Protective Time (Min.) mg. Cl+ /cm.2	Protective Time (Min.) mg. S-145/cm.2	% Cl+ Remaining after test
Air Dried	12	2.4	750	109	0.5
Dried 1 hr. 40°C	9	1.8	793	115	0.5
Dried 1/2 hr. 70°C	8	1.6	878	127	3.3
Dried 1/4 hr. 105°C	7	1.4	647	94	0.0
Air dried, softened by flexing	9	1.8	750	109	2.7

[redacted]

The above data show that an increase in drying temperature of impregnated samples containing 17-1/2% RH-403 resulted in a significant decrease in the "polyvinyl alcohol effect". However, the initial leakage of HS vapor was not reduced to the level of solvent impregnations as shown by the failure of the number of bubblers required to be reduced to five or less.

76. An effect similar to the "polyvinyl alcohol effect" has been observed when methyl cellulose is used as the emulsifying and dispersing agent in aqueous impregnation systems. This effect has not been extensively studied since polyvinyl alcohol (RH-403) appears to be somewhat more satisfactory from certain standpoints than methyl cellulose (Methocel).

(5) The Effect of S-42 on HS and M-1 Resistance.

77. To determine the effect of the stabilizer S-42 on the protective capacity of clothing impregnated from aqueous systems, numerous HS and M-1 vapor penetration tests have been made on samples containing various concentrations of this agent. Since either 10 or 25% S-42 (% of S-145) appears to be the most likely concentration of this agent, the data reported in Table XXIX are confined to these two concentrations. Values are also given for HS vapor resistance on sample exposed to weathering and storage tests, and data for solvent impregnations are presented for comparison.

Table XXIX

Impregnation Process	S-42 (% of S-145)	Weathering or Storage	Agent	Protective Time (min.) mg.Cl+/cm.^{2}	mg. S-145/cm.^{2}	% Cl+ remaining after test
Solvent	0	None	HS	407	59	2.3
"	10	"	"	382	55	27.6
"	25	"	"	363	50	30.3
Aqueous	0	None	HS	766	111	0.6
"	10	"	"	694	101	9.8
"	25	"	"	694	101	11.9
Solvent	0	75°C-75%R.H.	HS	386	56	2.2
"	10	" " "	"	297	43	43.7
"	25	" " "	"	273	40	35.0

[redacted]

Table XXIX
(continued)

Impreg-nation Process	S-42 (% of S-145)	Weathering or Storage	Agent	Protective Time (Min.) mg.Cl⁺/cm.²	Protective Time (Min.) Mg. S-145/cm²	% Cl⁺ remaining after test
Aqueous	0	75°C-75% R.H.	HS	505	73	3.9
"	10	" " "	"	568	82	5.8
"	25	" " "	"	450	65	44.2
Solvent	0	Outdoor (Fla.)	HS	399	58	2.0
"	25	" " "	"	414	60	30.2
Aqueous	0	Outdoor(Fla.)	HS	399	58	2.0
"	25	" " "	"	711	103	13.2
Solvent	0	None	M-1	45	7	1.8
"	10	"	"	78	11	14.6
"	25	"	"	111	17	21.1
Aquеous	0	None	M-1	100	15	9.2
"	10	"	"	139	21	14.4
"	25	"	"	168	24	5.6

78. The results shown in Table XXIX indicate that in the case of HS vapor penetration the protective efficiency of the impregnite is lowered slightly in the presence of S-42. This decrease is not as pronounced for water dispersion impregnations as for solvent impregnations. No difference is observed between 10 and 25% S-42 for either solvent or water impregnations. Samples containing either 10 or 25% S-42 show the same protective efficiency as unstabilized samples after outdoor exposure (Florida, 2 weeks) and after the accelerated storage test (96 hours, 75°C-75% R.H.). In the case of M-1 vapor penetration the protective capacity of the impregnite increases in the presence of S-42. The magnitude of this increase is a function of the S-42 concentration.. This substantiates peevious work which shows that S-42 alone offers good protection aginst M-1 vapor penetration.

(6) The Effect of S-145 Particle Size on HS Vapor Resistance.

79. As shown previously, the protective capacity of clothing impregnated from aqueous systems is about twice that of clothing impregnated from tetrachloroethane solutions, and it was mentioned that this might be due to the lower particle size of the S-145. This was studied by subjecting a series of samples impregnated from a water system with S-145 which had been carefully fractionated into particle size

[redacted]

ranges to HS vapor penetration tests. The results of these tests are shown in Table XXX.

Table XXX

Effect of S-145 Particle Size on HS Resistance

Particle Size of S-145 in microns	Protective Time (Min.) $mg.Cl^{+}/cm.^{2}$	Protective Time (Min.) $mg.S\text{-}145/cm.^{2}$	% Cl^{+} remaining after test
Micronized, 1-8	754	109	0.3
0.5-1	888	129	1.3
1-3	735	106	0.0
5-10	794	115	0.0
10-15	653	95	0.0
20-30	453	66	5.3
44-53	332	48	13.0
Mixed, 20-100	323	47	11.4

80. The data in the above table show that below 15 microns the protective values are all near the normal range of 90-120 min./mg. S-145/cm.^{2}. A drop in protective efficiency occurs if the particle size exceeds 15 microns. Samples impregnated with S-145 of 44-53 microns diameter and with the unground mixture have an even lower protective value than solvent impregnated samples. The data in the table show that the size range of 20-30 microns gives a protective value equivalent to that of solvent impregnated samples (cf. Table XXIII). No method has yet been found to establish definitely the actual particle size range of S-145 on solvent impregnated clothing. However, microscopic examinations have revealed many S-145 particles on solvent impregnated samples in the range of 20-30 microns.

C. Laundering Resistance.

81. Perhaps the main objection to clothing impregnated by aqueous systems is inferior laundering resistance when compared to clothing impregnated by the solvent method. A comparison of the laundering resistance of clothing impregnated by aqueous and solvent methods is shown in Table XXXI. These laundering tests were carried out at two different temperatures, 140°F (60°C) and 90°F (32.2°C) in a household type washing machine. Laundering at 140°F was done with 0.5% mild soap (Ivory) solution for 15 minutes. The clothing was then rinsed twice with water at 140°F and once with unheated water. The 90°F laundering was done with 0.5% mild soap solution, with three five minute sudsings, followed by three rinses with water at 90°F. Clothing was air dried for 16 hours or more between each laundering.

Table XXXI

Laundering Resistance of Clothing Impregnated by Solvent and Aqueous Processes

Impregnation Process	Orig. mg. Cl⁺/cm.²	% Cl⁺ retained after 1 wash	2 wash	3 wash	4 wash
		90°F			
Solvent	0.39	84	82	75	76
Solvent, 25% S-42	0.36	83	78	72	63
Aqueous, 25% S-42	0.54	68	50	36	31
		140°F			
Solvent, 25% S-42	0.45	67	50	35	18
Aqueous, 25% S-42	0.55	50	29	18	-

82. It is evident from the data in Table XXXI that clothing impregnated by the aqueous process is much less resistant to laundering than solvent impregnated clothing both at 140°F and 90°F. It is further evident that the 140° laundering is more vigorous than the laundering done at 90°F. The superior laundering resistance of solvent impregnated clothing is not entirely unexpected. During the evaporation of solvent from clothing impregnated by this method, the crystals of impregnite formed must be coated with chlorinated paraffin. The very great laundering resistance of chlorinated paraffin itself has been demonstrated (cf. NRL Report No. P-2000). Hence the impregnite particles coated with chlorinated paraffin on solvent impregnated clothing are somewhat protected during laundering operations. When clothing is impregnated by the present aqueous process, however, both the impregnite and chlorinated paraffin are applied as discrete and separate particles and no continuous film of chlorinated paraffin can form over the impregnite.

SUMMARY AND CONCLUSIONS

Section I. Formulation of Aqueous Impregnating Systems.

(1) The best emulsifying agents for chlorinated paraffin were found to be Triton NE, Methocel and RH-403. The latter two are considerably improved when used in combination with Daxad 11 and Gardinol WA. Chlorinated paraffin can best be emulsified when the emulsion mixture is concentrated and has a high viscosity. High speed stirrers, colloid mills, and gear pumps are satisfactory for preparing emulsions of chlorinated paraffin.

[redacted]

(2) A ball mill is suitable for grinding and dispersing S-145. The micronizing process for grinding S-145 is superior to a ball milling process and gives a satisfactory product. Daxad 11 and Methocel are considered to be the best dispersing agents for S-145. Micronized S-145 may be added directly to chlorinated paraffin emulsions.

(3) Aqueous impregnating processes are adaptable to existing solvent impregnation plants and require very little added equipment. A satisfactory plant trial of an aqueous impregnating system has been carried out using only a portable stirrer and a gear pump in addition to the plant equipment.

(4) The NDRC group at the DuPont Experimental Station has developed a satisfactory aqueous impregnation system for use in the field or on shipboard which requires no mechanical equipment.

Section II. Stability of Aqueous Impregnating Systems.

(1) Dispersions of S-145 in water are stable on storage at normal temperatures. A very slight loss of active chlorine occurs after four months storage at 110°F. The presence of S-42, cotton cloth, RH-403 and Methocel do not appreciably affect the stability of S-145 TCA in boiling water.

(2) Complete impregnating mixtures show no significant loss of active chlorine when stored for four weeks at room temperature. The S-145 settles out on storage and is difficult to redisperse. The pH of chlorinated paraffin emulsions and S-145 dispersions falls rapidly on storage unless S-42 is present.

(3) Emulsions of chlorinated paraffin prepared with different emulsifying agents can withstand alternate heating and freezing with no apparent damage. Concentrated aqueous dispersions of S-145 and concentrated complete impregnating mixtures become hardened after alternate heating and freezing and cannot be dispersed in water. For this reason such concentrates are not considered suitable for service use.

Section III. Impregnation from Aqueous Systems.

(1) The five main factors which affect the quantity of impregnite retained by cloth upon impregnation are: (a) Concentration of impregnite, (b) Particle size of the impregnite, (c) Viscosity of the impregnating bath, (d) Finish of the cloth, and (e) Method of removing excess liquid.

(2) A method has been found for uniformly impregnating

[redacted]

small cloth swatches for laboratory testing. Arnzen suits impregnated in plant trials by an aqueous process are as uniformly impregnated as suits impregnated by the solvent process. The field or shipboard impregnation procedure which involves hand wringing results in less uniform distribution of impregnite.

(3) Laboratory experiments have indicated that both water and S-42 are preferentially absorbed on cloth during successive impregnations.

(4) The air resistance of Arnzen cloth impregnated from aqueous systems is the same as Arnzen cloth impregnated by the solvent process. The emulsifying and dispersing agents, RH-403 and Methocel, affect the handle of impregnated cloth. An RH-403 concentration of 17-1/2% by weight of S-145 results in stiff, boardy cloths whereas concentrations of 5.0% or less have very little effect upon cloth handle.

(5) The Navy practice of simultaneously dyeing and impregnating protective clothing is satisfactory for aqueous impregnating systems.

Section IV. Performance Tests of Cloth Impregnated from Aqueous Systems.

A. Accelerated Storage and Weathering.

(1) The loss of active chlorine and loss of tensile strength for cloth impregnated from aqueous systems of chlorinated paraffin and S-145 have been determined in accelerated storage and weathering tests. The dispersing agent Triton NE accelerated the loss of active chlorine in accelerated storage tests when compared to the agents RH-403 and Methocel.

(2) The stabilizer S-42 improves the retention of tensile strength in the accelerated storage tests but has no appreciable effect on the active chlorine retention. Different concentrations of S-42 cannot be distinguished by their effects in the test.

(3) The effect of S-42 is much more marked in the sealed tube test since it acts to stabilize against both loss of active chlorine and loss of tensile strength.

(4) The data obtained in the tropical storage tests appear to be consistent with the results obtained in the more accelerated storage tests except that the stabilizing action of S-42 is less pronounced.

[redacted]

(5) Cloth samples exposed outdoors in Florida show a very high loss of active chlorine (40-50%) during the first week of exposure. The loss during subsequent weeks is appreciable but proportionately much less. This behavior is attributed to a shielding action of the fabric or impregnite decomposition products on the S-145 which prevents the passage of ultra-violet rays. The loss of tensile strength of samples containing varying amounts of S-42 have been correlated with loss of basic S-42 upon exposure. From these results the use of 25% is recommended.

B. Resistance to Vesicant Vapors.

(1) It has been found that the clothing impregnated by an aqueous process with S-145 has approximately twice the protective capacity against both HS and M-1 per unit of impregnite of clothing impregnated by the solvent process.

(2) Cloth samples impregnated from an aqueous system (in contrast to solvent impregnated samples) show a considerable loss in protective capacity after exposure in the accelerated storage test. Outdoor weathering, however, does not influence the protective efficiency of samples impregnated by either the aqueous or solvent methods.

(3) The emulsifying and dispersing agent RH-403 causes a high initial "leakage" concentration of HS through impregnated cloth. This effect is appreciable only for concentration of 8% (by weight of S-145) or higher. Methocel behaves similarly when it is used as an emulsifying and dispersing agent.

(4) The stabilizer S-42 causes a slight lowering of the HS protective capacity per unit of impregnite but the effect is less pronounced for aqueous impregnations than for solvent impregnations. On the other hand, S-42 results in an increase in protective capacity against M-1 for both solvent and aqueous impregnated cloths.

(5) A study of the effect of S-145 particle size on the HS vapor penetration of cloth samples impregnated by the water dispersion method has shown that the protective capacity decreases with increasing particle size. The particle size range of 20-30 microns gave a value equivalent to solvent impregnated samples whereas larger size ranges exhibited much less protective capacity.

C. Laundering Resistance.

(1) The laundering resistance of impregnated clothing has been determined at 140°F and 90°F. The results showed

[redacted]

that clothing impregnated by the aqueous process is inferior in regard to laundering resistance than clothing impregnated by the solvent process. The 90°F laundering is much milder than the laundering at 140°F.

RECOMMENDATIONS

(1) It is recommended that the Navy replace the present solvent process of impregnation in central impregnating plants with an aqueous method of impregnation. Pending further experimental results, the aqueous system would employ a combination of polyvinyl alcohol (RH-403), a naphthalene-formaldehyde sulfonate (Daxad 11), and sodium lauryl sulfate (Gardinol WA, Duponol ME) for the emulsification of chlorinated paraffin and the dispersion of micronized S-145. The stabilizer S-42 should be incorporated in the aqueous impregnating system and at a concentration of 25% by weight of the S-145.

(2) It is recommended that the Navy consider the adoption of the field or shipboard process of aqueous impregnation for emergency use at shore stations and advance bases. In the event that a change of policy regarding impregnation on shipboard occurs, it is recommended that an aqueous system be adopted.

(3) It is recommeded that work on aqueous impregnating systems be expanded to include other impregnites and other binding agents which hold promise for application to aqueous systems.

(4) It is recommended that a study of laundering resistance of aqueous impregnations be considered as urgent and be investigated from the standpoint both of improved formulation and improved laundering technique.

(5) It is recommended that wearing trials be conducted for establishing the useful life of protective clothing impregnated by an aqueous system and thereby establish the relative importance of laundering resistance. These trials would also be useful for determining possible irritant effects of protective clothing worn under service conditions.

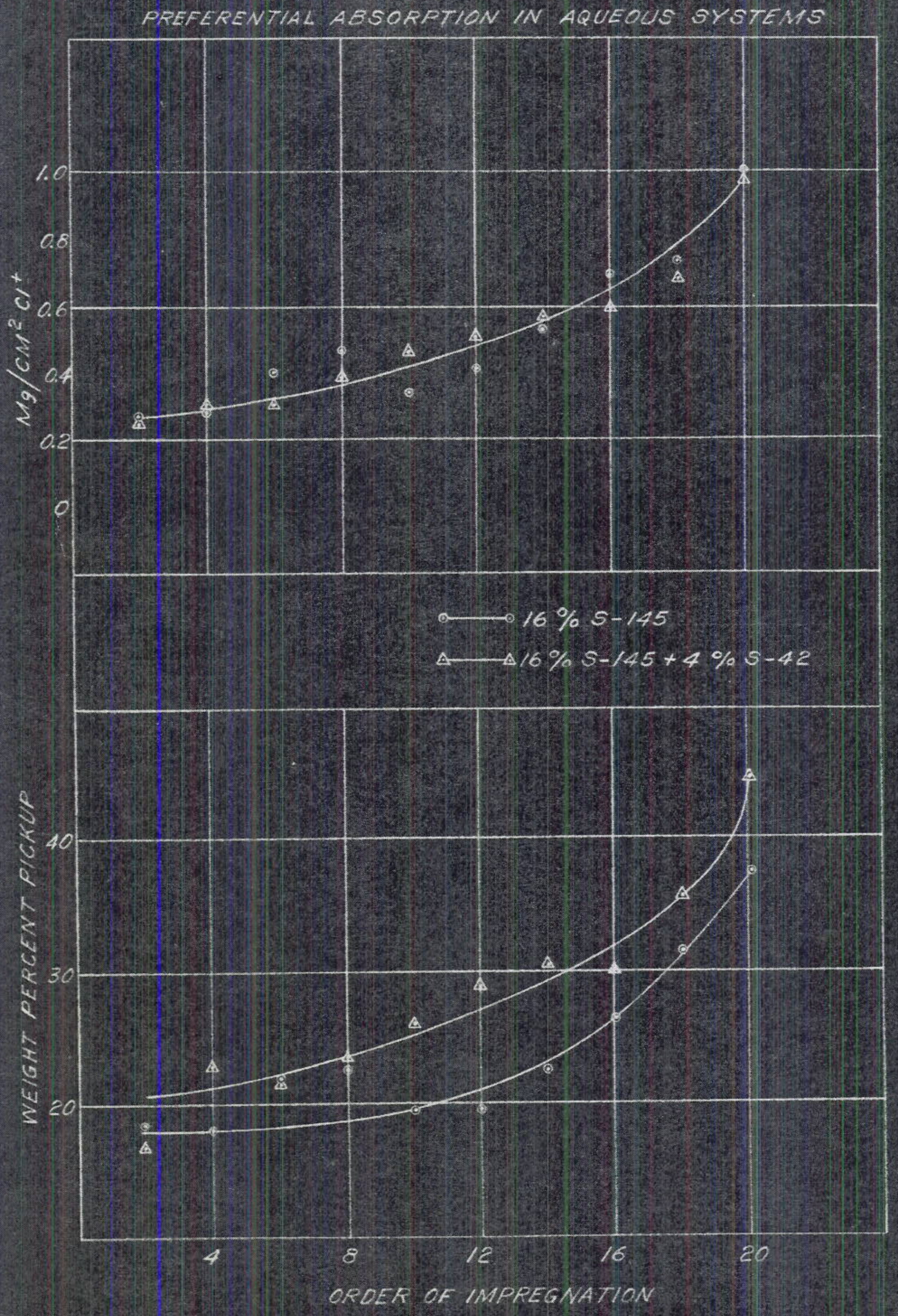

PLATE I

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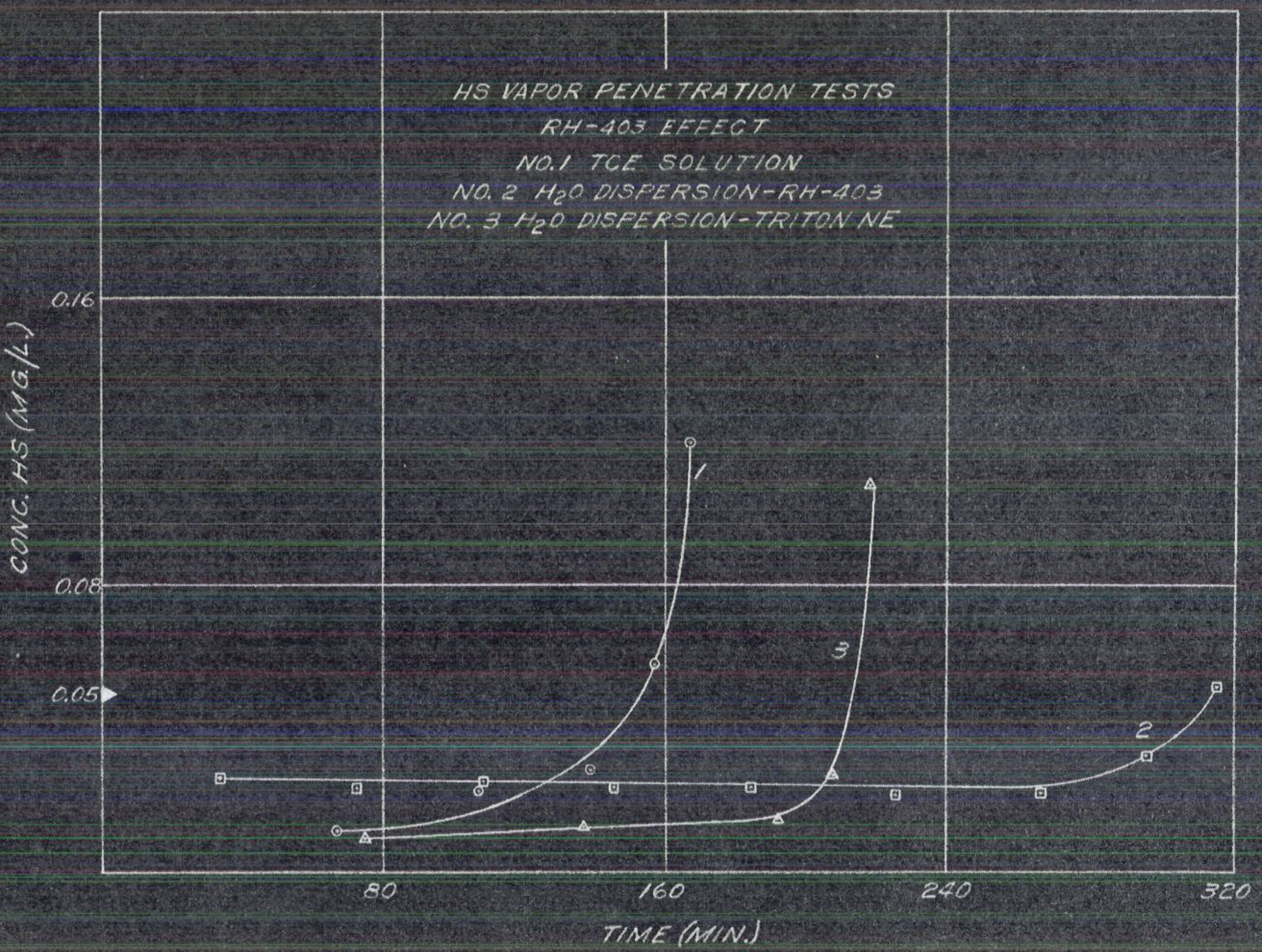
HS VAPOR PENETRATION TESTS
RH-403 EFFECT
NO.1 TCE SOLUTION
NO. 2 H_2O DISPERSION-RH-403
NO. 3 H_2O DISPERSION-TRITON NE
CONC. HS (MG./L.)
0.16
0.08
0.05
1
2
3
80
160
240
320
TIME (MIN.)

PLATE 2