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CHEMISTRY DIVISION - PROTECTIVE CHEMISTRY SECTION

9 November, 1945

SUMMARY REPORT ON DYESTUFFS USED FOR SIMULTANEOUS DYEING AND IMPREGNATING OF PROTECTIVE CLOTHING

By

H..R. Billica
and
G. M. Gantz

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Superintendent, Chemistry Division

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Director, Naval Research Laboratory

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NRL Problem P-60
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ABSTRACT

This report describes the history of the development of suitable dyestuffs for use in simultaneous dyeing and impregnation of protective clothing. Dyes had to be provided for both the solvent and aqueous systems of impregnation.

Anthraquinone vat dyes blended with an equal weight of dispersing agent have been used as pigments in the aqueous impregnation process. Vat colors have been used since they proved to be resistant to the impregnite CC-2. It has been found possible to use much cheaper pigments for aqueous impregnations such as phthalocyanine dyes, carbon black, and drude forms of vat colors.

Vat dyes have been used also in solvent process impregnation in the form of flushed pigment pastes. Much difficulty was encountered in obtaining identical pigment shades for colors made by different dyestuff manufacturers. It was found possible to eliminate the solvent in the flushed pigments and prepare solvent dispersible pigment powders from equal parts of dyestuff and ethyl cellulose.

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INTRODUCTION

A. 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-77-2(Dz), Serial 811 of 17 December 1940.

B. Statement of the Problem

2. The problem of developing dyes suitable for simultaneously dyeing and impregnating of protective clothing has involved the testing of many dyes for their compatibility with CC-2 both in storage and in outdoor exposure. Constant effort has been made to lower the cost and improve the color value of the dyes.

C. Known Facts Bearing on the Problem.

3. The Navy Department decided before the War to acquire only undyed fabric for protective suits in view of evidence that many dyestuffs were incompatible with CC-2. At that time the only impregnation process used was a tetrachloroethane solution of CC-2 and chlorinated paraffin. A dye preparation was developed by the Technical Laboratory of the du Pont Co., and the CWS for use in the tetrachloroethane impregnating solution. The preparation was known as Flushed Pigment No. 3031, and it consisted of a 5 percent dispersion of vat dyes (5 parts Indanthrene Khaki 2G to 1 part Indanthrene Yellow GK), in a solution of perchlorethylene containing 5 percent of ethyl cellulose.

4. In the development of Flushed Pigment No. 3031 it had been found that a number of vat dyes did not cause deterioration of CC-2 and that such dyes could be prepared for use as pigments in the impregnating solution.

5. The advent of the water dispersion impregnation process made it imperative that suitable dye preparations be developed for use in an aqueous medium.

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D. Theoretical Considerations.

6. Many factors are included in developing suitable dyes for the simultaneous dyeing and impregnating of protective clothing. It is essential that a suitable shade be obtained for camouflage and that whiteness of both the fabric and impregnating chemicals be masked. In addition to the qualities of light fastness and wash-fastness, it is most essential that the dyestuffs be chlorine-fast in view of the active chlorine contained in the impregnite.

7. As a class the vat dyestuffs meet most of the requirements outlined above. These dyes are usually applied or fixed into fabric by dissolving in an alkaline bath of hydrosulfite. Simultaneous dyeing and impregnating eliminates this possibility and necessitates use of the vat dyes as pigments. This in turn requires that the dyes be very finely divided and in such a form as to be readily incorporated into impregnating baths.

E. Previous Work Done at This Laboratory.

8. No previous formal report on this subject has been prepared by this Laboratory. This report is a summary of all the work done on the use of dyes for simultaneous dyeing and impregnating of protective clothing, and most of the data has been reported earlier in the form of letters to the Bureau of Ships or memoranda to the Director. Data in the following Memoranda to the Director are involved in this report:

"Studies of Water Dispersible Dyes for Simultaneous Dyeing and Impregnating Protective Clothing", dated 9 February, 1943; "Evaluation of Water Dispersible Dyes", dated 13 October 1943, and "Tests of Dyestuffs Submitted for Simultaneous Dyeing and Impregnating of Protective Clothing", dated 1 April 1944.

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EXPERIMENTAL

Part I - Dyes for Aqueous Impregnation Systems

A. Tests on First Samples from du Pont.

9. In the last half of 1942, laboratory results on aqueous impregnation systems showed great promise. Consequently, the Technical Laboratory of the E. I. du Pont de Nemours Co. developed water dispersion pigment forms of the vat dyes which had proved satisfactory in solvent impregnation with CC-2. Two shades were submitted to this Laboratory designated as TLX-12, a khaki corresponding to flushed pigment No. 3031, and TLX-15, a battleship grey corresponding to the color of Navy anti-flash clothing. Each shade was supplied in the form of a water dispersible dry powder, a concentrated aqueous paste, and a more dilute aqueous paste. In each case the solids were made up of approximately equal parts dyestuffs and dispersing agent. The dispersing agent used was a sodium sulfonate of a naphthalene-formaldehyde condensation product.

10. Laboratory work on these samples was primarily directed toward determining the best procedure for incorporating the dyes in the aqueous impregnating system. At that time, the steps followed in preparing an aqueous impregnating bath were as follows:

(a) A 10% solution of polyvinyl alcohol (PVA) is made.
(b) Chlorinated paraffin is added to the PVA solution with stirring to give a concentrated emulsion.
(c) The emulsion is diluted and micronized CC-2 and zinc oxide are stirred in.
(d) The concentrated suspension is passed through a colloid mill or other dispersing device and then diluted to proper strength.

11. It was found that the best method of incorporating the water dispersible dyes was to prepare a 10% dispersion in water to be added to the impregnating suspension during the final dilution. After the addition of dyes it was necessary to pass the entire mixture through a colloid mill to effect deffloccculation of the pigments.

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It was found that a concentration of 4-1/2% dyes based on the weight of CC-2 was ample for producing the required depth of shade on the impregnated clothing.

12. Following these results the Navy decided to procure three dye powders for aqueous impregnation. These were TLX-40, a mixture of 50 parts Lithosol Blue GL with 50 parts dispersing agent; TLX-41, a mixture of 50 parts Khaki 2G with 50 parts dispersing agent; and TLX-42, a mixture of 50 parts yellow ARP with 50 parts dispersing agent. The individual dyes were procured rather than blends to permit a greater latitude in formulation in the impregnating plants.

13. A policy was adopted by the Navy to expand production of impregnated clothing and to use the aqueous process of impregnation. It was further decided that protective suits issued to ships would be battleship gray while the suits issued to shore stations would be khaki. The battleship gray color was obtained by blending 3 parts of TLX-40 with 1 part of TLX-42 whereas the khaki color required 5 parts of TLX-41 with 1 part of TLX-42.

B. Evaluation of Alternate Blue Dyes.

14. In 1943, anthraquinone vat dyes became more critical principally because of a shortage of phthalic anhydride and greatly increased requirements by the Armed Services. The duPont Company submitted several water dispersible dye powders to be investigated as substitutes for Lithosol Blue GL (TLX-40). The dyes investigated in this series may be classified as follows:

I. The three anthraquinone dyes adopted for use by the Navy:

TLX-40 - Lithosol Blue GL
TLX-41 - Ponsol Khaki 2G
TLX-42 - Lithosol Fast Yellow ARP

II. Blue anthraquinone dyes related to TLX-40:

TLX-45
TLX-46

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III. Direct azo dyes as substitute for TLX-40:

TLX-33 TLX-35 TLX-37

TLX-34 TLX-36

IV. Miscellaneous dyes:

TLX-23 - Sulfanthrene Brown G
TLX-29 - Carbon Black

15. It was decided that the effect of these dyes on CC-2 in storage as well as their light fastness on outdoor exposure should be determined. Accordingly, cloth samples were impregnated by the standard aqueous process with the dyes under investigation. The series of impregnated cloth samples was subjected to an accelerated storage test at 75° - 75% R.H. for 96 hours, simulated tropical storage at 110°F - 75% R. H., for one month, outdoor storage for three weeks, and H vapor penetration tests.

16. The percentage of active chlorine retained by each sample after the storage and exposure tests is shown in Table I. Tensile strength measurements were also made but since all the samples retained adequate strength these data are not included.

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Table I

Per Cent of Active Chlorine Retained After Storage and Exposure Tests of Impregnated Cloth Samples Containing Water Dispersible Dyes

Dye (ratio)	Per Cent Cl+ Retained				
	96 hrs. at 75°C/75%RH	1 mo. at 110°F/75%RH	Outdoor 1 Week	Outdoor 2 Weeks	Outdoor 3 Weeks
No dye, control	34	81	58	30	37
TLX-33, TLX-42(2:1)	34	85	48	50	41
TLX-34, TLX-42(2:1)	30	78	50	31	31
TLX-35, TLX-42(2:1)	37	83	52	34	39
TLX-36, TLX-42(2:1)	36	77	53	35	31
TLX-37, TLX-42(2:1)	41	88	57	35	33
TLX-40, TLX-42(2:1)	44	83	50	34	33
TLX-41, TLX-40(5:1)	42	83	53	42	43
TLX-40	46	88	52	49	43
TLX-41	46	83	58	44	44
TLX-42	45	88	49	37	40
TLX-23	32	80	57	53	26
TLX-40, TLX-23(2:1)	32	74	71	44	36
TLX-40, TLX-23(1:1)	36	71	62	38	37
TLX-40, TLX-42(2:1)	60	40	51	38	31
TLX-45, TLX-42(2.7:1)	56	64	48	38	34
TLX-46, TLX-42(2:1)	68	46	53	41	38
TLX-40, TLX-29(5:1)	62	44	49	40	34
TLX-40, TLX-29(3:1)	63	55	60	46	34

17. It is apparent from the data in Table I that none of the dyes or combinations tested had a marked effect upon loss of active chlorine during the storage and exposure tests. However, a wide range of color-fastness was noted in these tests.

18. A description of the color fading of the impregnated samples during the storage and exposure tests is shown in Table II. In these tests it is expected that color changes on outdoor exposure are indicative of both fastness to light and chlorine, whereas the storage test would show only chlorine fastness.

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Table II

Degree of Color Fading During Storage and Exposure Tests of Impregnated Cloth Samples Containing Water Dispersible Dyes

Dye (ratio)	96 hrs. at 75°C/75%RH	1 mo. at 110°F/75%RH	Outdoor Weathering 1 week	2 weeks	3 weeks
TLX-33,TLX-42(2:1)	Moderate	Slight	Severe	Complete	Complete
TLX-34,TLX-42(2:1)	Severe	Moderate	Complete	Complete	Complete
TLX-35,TLX-42(2:1)	Moderate	Moderate	Severe	Complete	Complete
TLX-36,TLX-42(2:1)	Moderate	None	Complete	Complete	Complete
TLX-37,TLX-42(2:1)	Slight	Slight	Severe	Complete	Complete
TLX-40,TLX-42(2:1)	Slight	None	Slight	Moderate	Moderate
TLX-41,TLX-42(5:1)	None	None	None	None	Very Slight
TLX-42	None	None	None	None	Very Slight
TLX-23	None	None	Slight	Slight	Slight
TLX-40,TLX-23(2:1)	Slight	None	Slight	Slight	Slight
TLX-40,TLX-23(1:1)	Slight	None	Slight	Slight	Slight
TLX-40,TLX-42(2:1)	Slight	None	Slight	Moderate	Moderate
TLX-45,TLX-42(2:1)	Slight	None	Slight	Slight	Slight
TLX-46,TLX-42(2:1)	Moderate	None	Moderate	Moderate	Moderate
TLX-40,TLX-29(5:1)	None	None	None	Very Slight	Very Slt.
TLX-40,TLX-29(3:1)	None	None	None	Very Slight	Very Slt.

19. The severe fading of the azo dyes in outdoor tests shows that they have poor light-fastness as well as rather poor fastness to chlorine. The blue vat dyes TLX-40, TLX-45, and TLX-46 were less resistant to chlorine and light than yellow and khaki, TLX-42 and TLX-41, but they are considered suitable for protective clothing use. The slight changes noted for blends containing TLX-23 and TLX-29 are attributed to the blue component rather than the brown or black dyes themselves.

20. H vapor penetration tests run on the dyed cloth samples by the NRL Static Cup Method, (NRL Report No. P-1831) indicated that the dyes had no effect on H vapor capacity, since the protective capacities were all in the range of 70-100 minutes per 0.1 mg. of active chlorine per sq. cm.

C. Development of Green Dye Blends.

21. By the first part of 1944 it had become

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apparent that increased Navy requirements for impregnating dyes would necessitate an expanded procurement program instead of reliance upon the duPont Co. as the sole source. Several other companies were contacted and given the requirements for both water dispersible and solvent type dyes. Shortly after negotiations were started to expand procurement, the Navy eliminated the two shades for protective suits in favor of a single shade of green.

22. Laboratory tests showed that the desired shade of green could be obtained by blending five parts of khaki (TLX-41) with one part of phthalocyanine green (TLX-20). In addition it was found that available stocks of blue, khaki, and yellow could be used in a blend to give an acceptable green shade. This blend had the following composition: 5 parts TLX-40, blue, 8 parts TLX-41 khaki, and 7 parts TLX-42 yellow.

23. These findings limited expanded procurement to the two colors, khaki and phthalocyanine green. The phthalocyanine green dye presented no difficulties since its only use is as a pigment and material from the only two manufacturers (duPont and General Dyestuff) matched well when processed into the water dispersible form designated as TLX-20. It was found necessary to use 60 parts of dispersing agent to 40 parts of dye in order to obtain maximum color value.

24. Obtaining Khaki 2G was a more difficult problem. It was found that khaki dyes produced by different companies had different pigment shades although they might be identical from the standpoint of vat dye application. It was found that khaki from one manufacturer had an undesirable yellow shade, whereas the same dye from another manufacturer had a desirable green shade. The pigment shade of the dye was apparently greatly affected by processing the presscake. A workable solution to this problem was found in specifying that impregnation plants should use khaki dye from one processor in a ratio of 5 to 1 with phthalocyanine green and use khaki from other processors in a ratio of 4 to 1 with phthalocyanine green.

25. In cooperation with the Harman Color Works, it

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was found possible to use cheaper pigments to replace the expensive vat dye khaki 2G. A carbon black pigment designated as TLX-29, was found compatible with CC-2 and satisfactory in the aqueous impregnation system. The Harman Color Works was able to prepare a satisfactory water dispersible pigment from a crude form of the vat dye Indanthrene Yellow GK. Laboratory and plant trials showed that the following dye blend produced a satisfactory shade of green:

1 part water dispersible black, TLX-29.
1 part water dispersible crude yellow, TLX-42.
1 part water dispersible green, TLX-20.

In addition, this blend used at a concentration of 7% on the CC-2 produced the same depth of shade as did former blends at a concentration of 9% based on the CC-2..

26. Altogether the use of the blend given above effected a saving of 75% in the cost of dyes for simultaneous dyeing and impregnating of protective clothing.

Part II. Dyes for Solvent Impregnation System.

A. Use of Flushed Pigments 3031 and TLX-1A.

27. Flushed Pigment No. 3031 was adopted by the Navy prior to the War for simultaneously dyeing and impregnating of protective clothing by the solvent process. This material which was developed by the Technical Laboratory of the duPont Co., and the CWS, consisted of a dispersion of 5% dyestuffs in a 5% solution of ethyl cellulose in tetrachloroethylene. The dyestuff consisted of a blend of the anthraquinone vat dyes Indanthrene Khaki 2G and Indanthrene Yellow GK in the proportion of two to one. The flushed pigment No. 3031 was stirred into the solvent impregnating bath in the proportion of 90 parts to 100 parts of CC-2 by weight.

28. When the Navy adopted the policy of providing battleship grey protective suits for shipboard wear, it became necessary to use a different flushed pigment

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to provide the color. A flushed pigment designated as TLX-1A was made up by the Technical Laboratory of the duPont Co. and approved by the Naval Research Laboratory. This product was the same as flushed pigment No. 3031 except that the dyestuff used was Indanthrene Blue GCD.

29. During a plant trial of flushed pigment No. TLX-1A at the Impregnation Plant, Naval Clothing Depot, it was found the blue color of the dyestuff gradually changed in the impregnating bath to an undesirable greenish blue shade. Further work revealed that when the stabilizer zinc oxide was employed, no such color change occurred. A second plant trial was run in which zinc oxide was added to the impregnating solution before the addition of the TLX-1A. This procedure proved satisfactory and was adopted as a standard plant procedure.

30. Concurrent with expanded procurement of water dispersible dyes, greater demands were made for flushed pigment type dyes. Several processors were given the requirements for flushed pigment No. 3031, including the manufacturing process employed by the duPont Co. As in the case of the water dispersible khaki, great difficulty was encountered in attaining comparable shades of color from the different processors of 3031.

31. An additional difficulty with flushed pigment No. 3031 arose in finding a suitable laboratory test procedure. Security regulations did not permit release of CC-2 to the dyestuff processors so they had to employ simulated impregnating solutions without this ingredient. Moreover, it was found that great variations both in shade and depth of color were caused by different methods of drying. After considerable study in the Laboratory, a standard procedure was adopted in which test samples in the form of tubes were dried by flexing and rubbing over the flame of a Fisher burner.

32. A list of typical samples of flushed pigment No. 3031 submitted by different manufacturers is shown in Table III.

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Table III

Typical Samples of Flushed Pigment No. 3031

Code No.	Producer	Description
3031 Standard	E.I. duPont de Nemours Co.	5% Flushed Pigment (67% Khaki 2G, 33% Lithosol Yellow-ARP), 5% Ethocel-10C, 90% Perclene
TLX-54	"	Same as above except 5% Ethyl Cellulose N-22 (Viscosity rating)
TLX-55	"	Same as above except 5% Ethyl Cellulose N-50
TLX-56	"	Same as above except 5% Ethyl Cellulose N-100
TLX-77	"	Same as 3031 standard except 90% Triclene
TLX-78	"	Same as 3031 standafd except 57.6% Triclene and 32.4% Perclene.
Paste 3031 RBH	RBH Dispersions, Inc.	Same as 3031 standard except for a different method of making up the final paste.
Y-8721	Harmon Color Works, Inc.	5% Flushed Pigment (60% General Dyestuff's Khaki 2G, 40% General Dyestuffs' Yellow-GKA), 5% Ethyl Cellulose, 90% Perclene.
E-7147	"	Same as Y-8721.
RBH-3031 DPP	RBH Dispersions, Inc.	Milled paste made from dried presscake; 3031 standard proportions.
RBH-3031 Dry	"	Coarse dry powder-3031-50% pigment, 50% Ethyl Cellulose.
SW-3031	Sherwin-Williams Prod.	5% Flushed Pigment (General Dyestuffs), 5% Ethyl Cellulose, 90% Perclene

33. As development work was carried out by the different processors on the flushed pigment 3031, two new methods of preparation were found. The original procedure used by duPont was to add tetrachloroethylene to presscake of the dyes and distill until all water was removed. Since the presscakes contain 70 to 80% water, large distillation equipment is required to produce the flushed pigment. The Harmon Color Works found that if they coprecipitated an aqueous dispersion

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of the dye with an emulsion of ethyl cellulose and dried the precipitate carefully, it would disperse readily in tetrachloroethylene to give maximum color value. RBH Dispersions, Inc., worked out a method of milling the dry dyestuffs into a solution of ethyl cellulose to form a chip stock which dispersed readily in tetrachloroethylene to form a dye paste similar to flushed pigment.

B. The Development of Flushed Pigment 6062.

34. After the Navy adopted the aqueous system for impregnation, further work on solvent type dyes was carried out to meet the requirements of the Marine Corps. Considerable work had been done on the flushed pigment No. 3031 as indicated above when the Marine Corps decided to adopt a darker and greener shade of color for protective clothing. This shade was designated as No. 6062.

35. In view of the urgency for production, it was decided to use only the three dyes which had been used in flushed pigments and were known to be satisfactory, namely, Khaki 2G, Yellow GK and Blue GCD. Depending upon the pigment properties of the individual dyes and upon the method of manufacture, a satisfactory green color could be produced by blending them in the following proportions:

8 parts yellow, 7 parts khaki, and 5 parts blue.

36. After each processor had submitted a satisfactory sample to NRL a plant trial was made at the Impregnation Plant, Naval Clothing Depot, Brooklyn. Following a successful plant trial on each manufacturer's material, contracts could be let.

C. Investigations of 6062 Dye Powder.

37. In the Summer of 1944, the Marine Corps made arrangements with the Chemical Warfare Service to have a large number of protective suits impregnated at one of the Army's Z of I Impregnating Plants. The Marine Corps protective suits were undyed and, therefore, required simultaneous dyeing and impregnating. The Army's

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Z of I Plant operated on a solvent system, but had never used dyestuffs because at that time Army protective clothing was dyed prior to manufacture and impregnation.

38. The solvent process employed by the Army differs from the process used by the Navy inasmuch as a higher temperature is employed and also a higher concentration of CC-2. It was the opinion of CWS experts that the use of flushed pigment No. 6062 in the Z of I plant would result in a buildup of tetrachloroethylene in the system which would eventually prevent sufficient CC-2 from dissolving. This made it necessary to obtain a solvent type dye which would not introduce tetrachloroethylene into the impregnating system. The Harmon Color Works and RBH Dispersions were requested to prepare solvent dispersible dye powder of 6062 inasmuch as such powders were prepared at one step in the procedures they used to make the flushed pigment 6062.

39. After small samples had been tested at NRL and found to be satisfactory, plant trials were conducted at the Impregnation Plant, Naval Clothing Depot, Brooklyn. The 6062 powders prepared by both the Harman Color Works and RBH Dispersions were found satisfactory in operating the solvent plant on the Navy's low temperature-zinc oxide process. Harmon's 6062 was considerably finer than the RBH product and it was found more difficult to disperse because of lump formation.

40. A sample of 6062 powder was then sent to Edgewood Arsenal for test in the Army's solvent process. It was reported that the dye changed color during the impregnation.

41. A further investigation was carried out at the Navy plant in Brooklyn. It was found that the blue dye component of the 6062 was changing to green in the heated impregnating solution. However, it was found that the blue dye from one manufacturer was much less susceptible to color change in the hot impregnating solution than dye from another manufacturer. Moreover, the unchlorinated Indanthrene Blue (RS) did not exhibit the color change to green as did the chlorinated dye

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(GCD). Upon the basis of these findings, it was possible to stipulate the blue dye component of 6062 powder so that no fading would occur during the impregnation.

42. Impregnation of Marine Corps clothing was started at the CWS plant at the ASF Depot in New Cumberland, using 6062 powder in the solution as a dye. Word was received from the plant that some CC-2 was being used up in process since frequent additions of CC-2 were necessary to maintain the proper solution strength.

43. An investigation in the laboratory revealed that the ethyl cellulose in the 6062 powder was responsible for the slow decomposition. The rate of chlorine loss depends upon the temperature and the concentration of ethyl cellulose. The effect of ethyl cellulose on loss of CC-2 is illustrated in Table IV.

Table IV

Effect of 6062 Powder and Ethyl Cellulose on Loss of Chlorine in CC-2 Impregnating Solution at 60°C.

Time	*Solution without Dye	*Solution with 6062 Powder
Original	7.9	7.1
18 hours	7.9	6.1
25 hours	7.8	5.4
42 hours	7.8	5.1
48 hours	7.8**	4.9
66 hours	6.7	4.3

* The solution without dye was made up to contain 8% CC-2, 6% CP, and 0.8% $CaCO_3$ in Tetrachloroethane. The solution with 6062 powder contained in addition 1.4% 6062 Dye powder.

** After the 48 hour analysis 0.7% of ethyl cellulose was added to the solution.

44. No means of eliminating or minimizing the loss of chlorine in the impregnating solution due to the

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presence of ethyl cellulose was found in laboratory studies. Some improvement was effected when it was found in the impregnating plant that the amount of 6062 powder used could be reduced.

45. Repeated tests in the laboratory on impregnated cloth samples failed to show that the 6062 dye powder caused or promoted the loss of chlorine in tropical storage. Therefore, it was decided that the use of 6062 powder in the CWS Plant should continue until all the Marine Corps clothing had been impregnated.

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SUMMARY AND CONCLUSIONS

1. It was found that protective clothing could be simultaneously dyed and impregnated by using water dispersible powders made from equal parts of anthraquinone vat dyes and a dispersing agent. A blend of 5 parts dispersible Khaki 2G, TLX-41, with 1 part of dispersible Yellow GK, TLX-42, gave a khaki shade to suits issued to shore stations, while a blend of 3 parts dispersible Blue GCD, TLX-41, with 1 part dispersible yellow GK, TLX-42, gave a battleship grey shade for suits issued to ships. It was found desirable to incorporate the dyes in the aqueous system by preparing a water dispersion of the dyes and using this dispersion to dilute the CC-2 emulsion concentrate to impregnating strength.

2. An evaluation of anthraquinone and azo dyes as substitutes for Blue GCD, TLX-40, showed that whereas the dyes had no adverse effect on CC-2, the azo dyes in particular faded badly during outdoor exposure.

3. When the Navy decided to adopt a single shade of green for protective suits, a satisfactory blend was found in using 5 parts of Khaki TLX-41 with 1 part of phthalocyanine green, TLX-20. Also it was found that a suitable green shade could be produced by blending the three dyes then available for impregnation, namely, TLX-40, TLX-41, and TLX-42. Considerable difficulty was encountered in obtaining identical pigment shades for Khaki 2G from different manufacturers, and it was necessary to adjust the proportions of the blend of khaki and green in accordance with the source of the khaki.

4. Further studies of dyes suitable for producing the green shade adopted by the Navy revealed that expensive vat dyes could be eliminated. A satisfactory blend was found in using equal parts of dispersible green, TLX-20, a dispersible carbon black, TLX-29, and a dispersible crude vat yellow,GK, TLX-42. The use of these cheaper pigments effected a 75% saving in the cost of dyes for the aqueous impregnation process.

5. Anthraquinone dyes have been used in the

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solvent impregnation process in the form of flushed pigments made up of 5% dye and 5% ethyl cellulose in perchloroethylene. The first color used by the Navy was Flushed Pigment No. 3031 which produced an olive drab shade. This paste contained the dyes Khaki 2G and Yellow GK. Another flushed pigment designated as TLX-1A containing the vat dye blue GCD was procured for preparing suits of battleship grey.

6. In the course of expanding procurement of flushed pigments to different processors, it was found that dyes from different manufacturers varied considerably in pigment shade and color value. Another difficulty was found in that the shade and strength of flushed pigments in laboratory tests varied with the method of drying the impregnated cloth samples. This difficulty was minimized by adopting a standardized procedure.

7. After the Navy adopted the aqueous process of impregnation, very few protective suits were impregnated By the solvent process, and further procurement of flushed pigments was limited to the requirements of the Marine Corps. The Marine Corps decided to change the color of their protective suit from the olive drab of Flushed Pigment No. 3031 to a ddeper shade of green. A flushed pigment designated as No. 6062 was developed to give this shade and it contained the three vat dyes, Khaki 2G, Yellow GK, and Blue GCD.

8. Shortly after difficulties had been ironed out and procurement started on flushed pigment No. 6062, the Marine Corps arranged to have a quantity of clothing impregnated by the CWS in a Z of I plant. The CWS decided that flushed pigments could not be used in their solvent process in view of the danger of a buildup of perchloroethylene. Fortunately, two of the processors of Flushed Pigment No. 6062 had developed methods of preparation which involved a dry powder of equal parts dye and ethyl cellulose. This powder was found to be dispersible in the solvent impregnating plant and to give the desired shade in tests at the Navy's solvent plant in Brooklyn.

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9. A plant trial in the Army solvent plant at Edgewood Arsenal showed that the 6062 powder was unsatisfactory because of gradual change of color. This color change was traced to the blue dye component and later eliminated by selecting blue dyes which did not change color in the impregnating bath. It was discovered later that the ethyl cellulose in the 6062 powder caused a gradual loss of CC-2 when the solvent impregnating solution was kept at 55-60°C as employed by the Army. No method was found for eliminating this effect of ethyl cellulose.

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RECOMMENDATIONS

1. None. This report is merely a summary and recommendations have been made previously in letters and communications to the Bureau of Ships.

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ACKNOWLEDGMENT

Throughout the War much work on dyes for both aqueous and solvent impregnation systems was done by the CWS laboratory at Edgewood Arsenal and the NDRC workers at the duPont Experimental Station as well as this Laboratory.

Without doubt the cooperation and efforts of dyestuff manufacturers and processors was in a large measure responsible for the development of satisfactory impregnating dyes. From the Navy's standpoint, particular credit should be given to the technical personnel and research workers of the following concerns:

The Technical Laboratory of the duPont Co.
The Harmon Color Works, Inc.
RBH Dispersions, Inc.
The General Dyestuff Corp.
The Sherwin Williams Co.

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Distribution:

Bu Ships	17
Bu Med	2
OR&I	1
OQMG	1
CO, Naval Unit, EA	1
CWS, Tech. Div.	4
NDRC (Dr. Wilkins Reeve)	2
NRC, Wash. D. C.	2

[redacted]

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