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of Vesicants

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Report on

Chloroamide Paste Systems for Decontamination of Vesicants

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ABSTRACT

This is a progress report on the study of the decontamination of vesicants. Several non-aqueous paste systems were examined. Of these, the best formulation contains Perclene, potassium oleate and S-461. In addition, investigation of aqueous decontamination systems is reported. The most promising development in this direction is an emulsion paste containing Perclene, an emulsifying agent, a chloroamide and a large proportion of water.

AUTHORIZATION

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

STATEMENT OF THE PROBLEM

2. This study was undertaken to investigate the relative merits of various paste systems for the decontamination of vesicant agents.

KNOWN FACTS BEARING ON THE PROBLEM

3. In earlier NRL reports on decontamination of vesicant agents (P-1944, P-2125), it was shown that solutions of chloroamides in certain solvents will decontaminate vesicant agents in deck paint readily. The present directive for decontamination on shipboard calls for the use of a solution of RH-195 in tetrachloroethane (TCE). However, TCE and o-dichlorobenzene, which has been suggested as a substitute solvent for shipboard decontamination, are good paint removers. It was also found that chloroamides when used with a number of other solvents did not readily decontaminate deck paint.

4. In NRL Report No. P-2125, "A Study of Perclene as a Solvent for Use in the Decontamination of Airplanes," it was shown that a spray of RH-195 in tetrachloroethylene (Perclene) will decontaminate vesicants in airplane lacquer, but will not readily decontaminate vesicants in deck paint. Certain additives were shown to increase the decontamination efficiency of a spray of Perclene/RH-195 on deck paint. It was found also that simple pastes made by mixing Perclene with RH-195 or S-461 decontaminated deck paint readily. It is stated in Informal Report No. 8 of Section 9-1-1 of NDRC, entitled "A New System for the Decontamination of Painted Surfaces and Metallic Surfaces which have been Exposed to C. W. Agents," that a paste mixture of Perclene, potassium oleate and S-461 is a satisfactory decontaminant for deck paint.

5. It was shown in NRL Report No. P-2125 that Perclene is a good decontamination solvent for naval use for the following reasons:

(a) Perclene is a satisfactory paint penetrant which does not appreciably harm airplane or deck paint.

(b) Perclene is not harmful to the clear plastics: cellulose acetate, Plexiglas and Lucite, under ordinary conditions.

(c) Perclene is not harmful to doped airplane fabrics, doped with either cellulose acetate or cellulose nitrate.

(d) Perclene is relatively non-toxic.

(e) It is not inflammable.

(f) It is commercially available at a low cost.

(g) Solutions or slurries of RH-195 or S-461 in Perclene are stable enough for decontamination use. However these mixtures cannot be stored for future use.

6. A disadvantage of Perclene is its relatively high rate of evaporation at ordinary or higher temperatures. Additives have been found, however, which retard this evaporation greatly, lengthening the time of contact of Perclene with the painted surface.

7. TCE is inferior to Perclene as a decontamination solvent for naval use in the following respects: toxicity, and injurious effect on aircraft and deck paint, Plexiglas, Lucite, cellulose acetate and cellulose acetate butyrate doped fabric.

8. It was shown in NRL Report No. P-1944 that solutions or slurries of various materials using water as the solvent did not readily decontaminate painted metal surfaces.

THEORETICAL CONSIDERATIONS

9. No solvent/chloroamide spray system which will decontaminate deck paint has been found to be sufficiently superior to TCE/RH-195 in other respects to warrant replacing the present system. However, there are several features of the TCE/RH-195 decontamination system which make it undesirable for shipboard use: a large amount of solvent must be stored on shipboard, the toxicity of TCE is high, TCE/RH-195 loosens and tends to remove deck paint, and two or three applications of TCE/RH-195 spray are necessary to decontaminate moderately or heavily contaminated surfaces painted with deck paint.

10. There are certain advantages over the spray system, TCE/RH-195, which might be obtained by using a non-aqueous paste system. The application of a paste can be limited to the area desired to avoid contact with equipment and instruments which might be harmed. The areas so treated will be instantly apparent, whereas an area sprayed with solution is not obvious when the solvent has evaporated. The paste requires only one application which can be left on as long as desired.

11. In choosing a solvent for a non-aqueous paste, Perclene was expected to be superior to most solvents because of findings discussed in paragraphs 4 and 5 of this report. As was reported earlier, a paste of S-461 or RH-195 in Perclene will decontaminate deck paint in a reasonable length of time (1-2 hours). It was hoped that a suitable additive could be found which would increase the decontamination efficiency, especially on a wet surface, and give better adhesion of the paste to the surface. Furthermore, an additive with a thickening action would cut down the amount of chloroamide necessary to make a suitable paste.

12. Most of the disadvantages of the TCE/RH-195 system would be absent if water could be used as the solvent; however, previous work has shown that water solutions or slurries decontaminate deck paint only very slowly. Some preliminary experiments showed that a paste made by mixing a chloroamide and water sometimes decontaminated deck paint if allowed to stay on the surface as long as 24 hours. Since H does not dissolve readily in water it was felt that the addition of a suitable surface-active agent might increase the speed of decontamination of such aqueous pastes. Furthermore, there is the added possibility that the addition of a water-soluble paint penetrant would further increase the speed of decontamination. Some method of thickening a chloroamide/water paste which would reduce the amount of materials which must be carried would be desirable.

13. Another possible way of increasing the decontaminating efficiency of a water/chloroamide system would be to incorporate into it a water-soluble paint penetrant, such as Perclene. The obvious way to accomplish this would be to make an emulsion of the water and paint penetrant. Some water-in-oil emulsions are thicker than either the water or oil alone, so that a paste could be made using less chloroamide. For decontaminating efficiency it would seem best to have the paint penetrant as the continuous phase so that better contact of the penetrant with the paint is obtained.

PREVIOUS WORK DONE AT THIS LABORATORY

14. A number of possible decontamination systems were studied and the results reported in NRL Report No. P-1944, "The Use of RH-195 for the Decontamination of HS and M-1." A more extensive study of the use of solvents with chloroamides was reported in NRL Report No. P-2125, "A Study of Perclene (Tetrachloroethylene) as a Solvent for Use in the Decontamination of Airplanes."

EXPERIMENTAL PART I. NON-AQUEOUS PASTES FOR DECONTAMINATION

A. Preliminary Work

(1) Experimental Procedure.

15. Blue deck paint panels were prepared by brushing one coat of BuShips Spec. 84-D-1 primer on steel strips, followed by two brush coats of BuShips Spec. 20B blue deck paint. Two-inch square panels were used for the tests. These panels were contaminated by spreading two drops of vesicant and allowing it to remain for one hour. The decontamination paste was applied with a spatula or brush and left for a given length of time. The paste was then removed by washing with water and brushing. The panels were blotted and allowed to dry for a few minutes, placed in a constant temperature box at 35°C for ten minutes and tested with the appropriate test paper. For H the Congo Red S-328 paper (freshly prepared using pure S-328) was used in conjunction with sodium carbonate impregnated paper. For HN1 and HN3 the DB3 paper with a carbonate filter paper was used. For L the DT paper was used without a carbonate filter paper. The first appearance of a positive test was recorded as the paper test time.

(2) Experimental Results.

16. It was found that pastes made with Perclene and S-461 or RH-195 would decontaminate H in deck paint panels if the paste applications were left on an hour or two. The fact that these pastes will decontaminate deck paint whereas a spray of the same materials will not, indicates that the rate of evaporation of Perclene is a big factor. Consequently, a search was begun for a suitable additive which would retard the evaporation of Perclene or otherwise improve the characteristics of a Perclene/chloroamide paste.

17. Ethyl cellulose when added to Perclene/RH-195 for use as a spray increased its effectiveness. Therefore, ethyl

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cellulose was tried in a paste. This paste was an effective decontaminant, but it was difficult to remove from the paint. This was still true, although to a lesser extent, when sorbitan monolaurate was added in an attempt to facilitate the removal of the ethyl cellulose paste. The difficulty of obtaining ethyl cellulose was another factor which would discourage its use.

18. 5% paraffin or 10% chlorinated paraffin in Perclene when used in Perclene/chloroamide pastes did not show any added advantages. Aluminum stearate had to be heated in Perclene to obtain a uniform thickened mixture. Pastes made using aluminum stearate decontaminated deck paint fairly well but the pastes did not adhere well to painted surfaces. 2% G.I. brown laundry soap in Perclene formed a thick gel when heated, but the addition of a chloroamide broke the gel. The decontamination efficiency was poor. Lauric acid and oleic acid did not appreciably thicken Perclene. There was no indication that these additives improved the decontamination efficiency of the pastes.

19. In order to establish the necessity of having a chloroamide in these pastes, some were prepared using $CaCO_3$, Fuller's earth or sodium perborate instead of a chloroamide. These pastes did not show any indication of decontamination of H in deck paint.

B. <u>Decontamination Pastes Using Potassium Oleate and Similar Materials as Additives.</u>

20. The NDRC has recommended a paste of Perclene, S-461 and potassium oleate for decontamination of painted surfaces. For this reason this mixture was investigated in detail at this Laboratory. The principal objection to the use of potassium oleate at the time its use was proposed was that it reacts with RH-195. Consequently, RH-195 cannot be substituted for S-461 in this paste. Because it was felt that other soaps might be stable with RH-195 and yet be just as effective, potassium stearate, diglycol stearate and diglycol laurate were studied at the same time for comparison.

(1) Preparation of the Pastes.

21. The decontamination pastes used were prepared as follows:

(a) Paste A. Perclene/potassium oleate/S-461. A mixture of two parts of Perclene and one part potassium oleate was made by stirring together the ingredients at room temperature. Five parts of this mixture and one part of S-461 were stirred together to make the paste. The potassium oleate was supplied by the NDRC decontamination group at DuPont.

(b) Paste B. Perclene/potassium stearate/S-461. A mixture of three parts Perclene and one part potassium stearate was used in some earlier experiments, but was found to be too thick. This ratio was changed 5:1. Five or six parts of this mixture was mixed with one part S-461. The potassium stearate was obtained from the Beacon Company.

(c) Paste C. Perclene/potassium stearate/RH-195. This paste is the same as Paste B except that RH-195 was substituted for S-461.

(d) Paste D. Perclene/diglycol stearate/S-461. At first the ratio of Perclene to diglycol stearate (Glyco Products Co.) used was 2:1. This was changed to a ratio of 4:1. Two parts of this mixture was mixed with enough S-461 (about one part) to make a smooth paste.

(e) Paste E. Perclene/diglycol stearate/RH-195. This paste was prepared in the same way as Paste D except that about equal parts of RH-195 and the Perclene/diglycol stearate mixture was used.

(f) Paste F. Perclene/diglycol laurate/S-461. Two parts of Perclene and one part of diglycol laurate (Glyco Products Co.) were mixed together and three parts of this mixture were stirred with enough S-461 (about two parts) to make a smooth paste.

(g) Paste G. Perclene/diglycol laurate/RH-195. The same proportions of Perclene and diglycol laurate were used as in Paste F. One part of this mixture was mixed with one part of RH-195.

22. It was necessary to heat the Perclene/potassium stearate and Perclene diglycol stearate to obtain good mixtures. Except for a few earlier experiments, the RH-195 used was micronized.

(2) Decontamination of Deck Paint.

23. The pastes described above were tested for the decontamination of H (under various conditions), L, HN1 and HV

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(H containing 8% chlorinated rubber). In each case the pastes were left on for one hour. The procedure outlined in paragraph 15 was used with the variations and vesicants as noted below:

I. H. No variations.

II. H. The contaminated panels were rinsed with water immediately preceding the application of the pastes.

III. H. The panels were cooled at 0°C and H was applied. The ingredients of the pastes were kept at 0°C for one hour before mixing. The panels were kept at 0°C for one hour after application of the paste. The panels were then washed off with water at room temperature and tested at 35°C according to the usual procedure.

IV. HV. (H containing 8% chlorinated rubber). No variations.

V. HN1. No variations.

VI. L. No variations.

24. The paper test times in minutes at 35°C obtained in these tests are recorded in Table I. The decontamination tests using these pastes were run with duplicate panels. All the test times recorded as > 60 indicate that the panels did not give a positive test within 60 minutes. Experiments run at this Laboratory showed that deck paint panels which give test times greater than 45 minutes at 35°C may be considered to be safely decontaminated. Preliminary tests also indicate that panels contaminated with HN1 which give paper test times of greater than 60 minutes may be considered to be safely decontaminated.

25. It is apparent from the data in Table I that the Perclene/potassium oleate/S-461 paste safely decontaminated all these vesicants. In the case of the thickened mustard (HV), paper test times of about 30 minutes were obtained. Except under extreme conditions, these panels can be considered to be safely decontaminated. All the pastes used decontaminated both HN1 and L satisfactorily. HV-contaminated panels were most difficult to decontaminate. However, in some cases these panels were found to be completely decontaminated by the second day tests. The H-contaminated panels which gave positive tests the first day frequently gave no test the second day.

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Table I

Decontamination of Vesicants

Perclene Paste	Paper Test Times (Minutes)											
	H Dry Panel R.T.		H Wet Panel R.T.		H Dry Panel 0°C		HV Dry Panel R.T.		HN1 Dry Panel R.T.		L Dry Panel R.T.	
	1st day	2nd day	1st day	2nd day	1st day	2nd day	1st day	2nd day	1st day	2nd day	1st day	2nd day
A. Potassium oleate/S-461	>60		>60		>60		30	>60	>60		>60	
	>60		>60		>60		28	>60	>60		>60	
B. Potassium stearate/S-461	20	>60	9	>60	45	>60	5	>60	>60		>60	
	35	>60	15	>60	45	>60	4	>60	>60		>60	
C. Potassium stearate/RH-195	13	>60	2	12	1	>60	1	6	>60		>60	
	20	>60	3	18	1	>60	1	16	>60		>60	
D. Diglycol stearate/S-461	>60		5	30	>60		4	40	>60		>60	
	>60		12	>60	>60		35	>60	>60		>60	
E. Diglycol stearate/RH-195	>60		4	14	9	>60	1	6	>60		>60	
	17	>60	9	32	>60		1	7	>60		>60	
F. Diglycol laurate/S-461	12	>60	10	55	>60		3	21	>60		>60	
	55	>60	10	>60	35	>60	5	>60	>60		>60	
G. Diglycol laurate/RH-195	2	>60	2	4	1	>60	1	12	>60		>60	
	>60		2	4	3	>60	1	12	>60		>60	

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26. In general, the S-461 pastes gave more nearly complete decontamination than corresponding RH-195 pastes. In most cases the Perclene/potassium oleate/S-461 paste gave better decontamination than any of the other pastes. It should be noted that the application to deck paint of the pastes described above caused no appreciable damage to the paint after one hour exposure.

(3) Effect of Addition of Water to Perclene Pastes.

27. It was learned from the DuPont (NDRC) group that the potassium oleate supplied by them contained about 20% water. To determine the effect of the addition of water to the other pastes another series of tests were run. The tests were run according to the usual procedure. The pastes were prepared in essentially the same way as described previously. The additives used were potassium stearate, potassium stearate containing 25% added water, diglycol stearate, diglycol stearate with twice its weight of water added, diglycol laurate and diglycol laurate containing 25% added water. It was observed that when RH-195 was added to the Perclene/diglycol stearate/water mixture, part of the water separated out. These pastes were tested for the decontamination of H on both wet and dry deck paint panels. The results are given in Table II.

Table II

Decontamination of H Using Pastes
With and Without Added Water

	Paper Test Times (Minutes)			
	No Water Added		Water Added	
Perclene Pastes	Dry Panels	Wet Panels	Dry Panels	Wet Panels
Pot. stearate/S-461	>60	>60	>60	>60
	49	>60	>60	>60
Pot. stearate/RH-195	20	11	>60	10
	31	>60	10	10
Diglycol stearate/S-461	>60	27	>60	>60
	>60	15	>60	>60
Diglycol stearate/RH-195	13	26	>60	>60
	11	23	>60	>60
Diglycol laurate/S-461	1	2	>60	>60
	1	>60	>60	>60
Diglycol laurate/RH-195	1	19	2	3
	1	4	5	5

Note: The second day tests showed that all the panels were safely decontaminated. Examination of the data indicates that addition of water aided in the decontamination by the pastes containing diglycol laurate and diglycol stearate. This was not found true for potassium stearate which, however, already contained a significant amount of water (12.5%).

(4) Decontamination of Airplane Lacquer.

28. Airplane lacquer panels were prepared by spraying steel strips with two coats of primer and two coats of M-485 blue gray or DuPont 71 Line Aircraft finishes. Two inch squares were used for these tests. Pastes made with Perclene/RH-195 or Perclene/S-461 with no additive decontaminated H within a half hour. Pastes made using potassium oleate, potassium stearate, diglycol stearate or diglycol laurate also decontaminated H in less than a half hour. In every case there was evidence of softening and removal of the paint. This was more pronounced on the M485 than on the 71 Line paint. This effect on the paint was particularly true of the paint where H had been applied. The paste made using Perclene/potassium oleate/S-461 was the least harmful of these pastes, being no worse than Perclene/S-461 alone.

(5) Decontamination of Clear Plastics.

29. Two-inch squares of cellulose acetate, Plexiglas and Lucite were cut from sheets of these materials 3/32-inch thick. The decontamination test procedure was the same as that used for painted panels except that the vesicant drops were applied and left one hour without spreading. The pastes used were Perclene/potassium oleate/S-461 and Perclene with RH-195 or S-461 and potassium stearate, diglycol stearate or diglycol laurate added. The three latter additives contained 25% added water. All of these showed at least a slight beneficial action against H on all three plastics. In virtually all cases the panels were completely decontaminated within 24 hours after the tests were run. No harmful effects on the plastics were noted except from the action of the vesicants.

30. The Perclene/potassium oleate/S-461 paste was again tried against H on plastics, for one-hour and two-hour applications of the paste. Panels contaminated by H for one hour were completely decontaminated the first day except for cellulose acetate which gave a positive test

for H even after 24 hours. However, the two-hour cellulose acetate panels after 24 hours gave a positive test of about 40 minutes.

31. One hour application of the potassium oleate paste decontaminated Lucite and cellulose acetate which had been contaminated with drops of HN1 for one hour. Plexiglas panels required a two-hour application of the paste. Plastic panels contaminated with drops of L for one hour were not decontaminated by a two-hour application. However, 24 hours later there was definite indication that some decontamination had taken place for L on Lucite and Plexiglas but not in cellulose acetate.

(6) Comparison of S-461 Protective Ointment and Potassium Oleate Paste for Decontamination of Clear Plastics.

32. S-461 Protective Ointment and Perclene/potassium oleate/S-461 paste were left on uncontaminated panels for 1, 6 and 24 hours, respectively. The potassium oleate paste showed no harmful effect even after 24 hours. Within six hours the S-461 ointment had clouded the plastics slightly, cellulose acetate being affected most. After 24 hours Plexiglas and Lucite were pitted slightly and clouded. The cellulose acetate was clouded and softened so much that removal of the ointment by brushing left marks on the plastic surface.

33. S-461 ointment was not as effective as the potassium oleate paste for the decontamination of vesicants in these plastics. Both systems will decontaminate HN1 in these plastics within two hours. Neither will decontaminate L completely although the Perclene/potassium oleate/S-461 paste shows definite beneficial action. The potassium oleate paste decontaminates H in these plastics much more quickly than does S-461 ointment. A one or two-hour application of S-461 ointment did not decontaminate H in these plastics as shown by first day tests.

34. Although S-461 protective ointment is less effective than the potassium oleate paste for the decontamination of the clear plastics, it has the advantage that the S-461 ointment can be stored indefinitely and, consequently, is ready for immediate use while the potassium oleate paste must be mixed before using.

(7) Decontamination of Doped Airplane Fabric.

35. The doped airplane fabric panels were prepared by stretching airplane cloth over 7x9-inch wooden frames and stapling along the edges. The cellulose acetate butyrate and cellulose nitrate dopes were applied in the same way with the exception of the thinner used. Two coats of reduced clear dope were brushed on, followed by two coats of unreduced clear dope. After drying overnight the panels were sanded with 4/0 sandpaper. Three spray coats of pigmented dope were then applied, the panels sanded again and given a final spray coat of pigmented dope.

36. The decontamination tests were run as follows: 20 drops of H was spread over each 7x9-inch panel and left one hour. The pastes were applied and left for one hour, then washed off with brushing. The panels were tested for completeness of decontamination by the usual procedure. The pastes used were the Perclene/potassium oleate/S-461 paste previously described; Perclene/S-461 and Perclene/RH-195 (micronized). The latter two pastes were prepared by adding enough chloroamide to the Perclene to make a smooth paste.

37. The Perclene/potassium oleate/S-461 paste decontaminated H in cellulose acetate butyrate doped fabric satisfactorily within one hour. The cellulose nitrate fabric was partially decontaminated by this paste within one hour and by the second day the tests for H were negative. The Perclene/S-461 and Perclene/RH-195 pastes did not completely decontaminate either type of fabric the first day but the tests were all negative by the second day. The Perclene/RH-195 paste bleached the cellulose nitrate fabric contaminated with H but no other visible effect of these pastes on the fabrics was evident.

(8) Effect on the Tensile Strength of Doped Fabrics.

38. The effect of H and decontaminating pastes and the pastes alone on the tensile strength of doped fabrics was determined. For the tensile strength measurements two 2x6-inch strips were cut from the fabric panel and each strip was broken by the one-inch grab method on the Scott Tester. The results are given in Table III.

Table III

Tensile Strength of Doped Airplane Fabrics

	Tensile Strength (Lbs.)			
	Cellulose Acetate Butyrate		Cellulose Nitrate	
Treatment Used	1 Treatment	3 Treatments	1 Treatment	3 Treatments
1. *None	122	117	126	116
2. Perclene/pot. oleate/S-461	127	116	123	105
3. Perclene/S-461	121	109	118	119
4. Perclene/RH-195	110	116	121	116
5. H + Perclene/pot. oleate/S-461	119	122	122	117
6. H + Perclene/S-461	94	49	123	110
7. H + Perclene/RH-195	56	47	112	99

*These fabric panels were controls subjected to outdoor weathering only.

Panels 2, 3 and 4 were not contaminated with H. They were, however, treated with the paste for one hour each time the paste was applied. Treatments were made at three-day intervals. After the last treatment in each case, the panels were left outdoors for two weeks, followed by 24 hours at 75°F and 50% R.H. before the tensile strength measurements were made.

39. From Table III it is evident that application of the pastes alone caused no significant decrease in tensile strength. From previous experiments (NRL Report No. P-2125) it is known that as many as three applications of H alone do not affect the tensile strength of either type of doped fabric. In Table III it is evident that decontamination of H in the doped fabrics using Perclene/potassium oleate/S-461 paste caused no loss in tensile strength. Cellulose acetate butyrate fabrics contaminated with H and decontaminated with Perclene/S-461 paste or Perclene/RH-195 paste showed a great loss in tensile strength. H followed by Perclene/S-461 paste caused no apparent damage to cellulose nitrate fabric panels but H followed by Perclene/RH-195 caused a distinct loss in tensile strength.

40. It may be concluded from these experiments that Perclene/potassium oleate/S-461 will decontaminate H in doped **airplane** fabrics without harming the fabric. The potassium oleate

has a beneficial action because Perclene/S-461 alone when used to decontaminate H in cellulose acetate butyrate fabric caused the tensile strength to fall greatly.

(9) Corrosion of Metals.

41. The details of the test procedure used for determining corrosion of metals by decontamination pastes are given in NRL Report No. P-2125. The metal samples were cleaned and weighed, two-thirds immersed in the test mixture for a given length of time, cleaned again, and weighed. Test samples were kept at 45°C for two weeks and at room temperature (about 30°C) for four weeks. The pastes used were Perclene/potassium oleate/S-461, Perclene/potassium stearate/S-461, and these pastes containing 10% ZnO based on the weight of the chloroamide. Perclene/potassium oleate/S-210 is included because of the possible substitution of S-210 for S-461. The data is given in Table IV and is expressed in terms of the percentage of the original weight of the metal panels lost during the test. Similar data for TCE/5% RH-195 and Perclene/5% RH-195 are included for comparison.

42. It is evident that the potassium oleate paste is in most cases much less corrosive than the TCE/5% RH-195 or Perclene/5% RH-195 mixtures. This is especially striking in the case of stainless steel. Under these conditions in TCE/RH-195 stainless steel lost more than 1% of its weight. In the potassium oleate paste there was no loss. The potassium stearate paste was more corrosive than the potassium oleate paste in the case of a few metals. It is noted that, in general, the addition of ZnO did not decrease the corrosive action of the pastes. However, if it should be desirable to add ZnO to the S-461 for any other reason these tests show that the mixture would not be significantly more corrosive.

(10) Stability on Storage.

(a) Perclene Mixtures with Soaps.

43. Mixtures of Perclene with potassium oleate, potassium stearate, diglycol stearate and diglycol laurate were prepared as described previously. Part of each mixture was stored at 0°C, 45°C, and room temperature (R.T.). These mixtures were examined at intervals and the effect of storage is given in Table V. The observed changes

Table IV

Corrosion of Metals

Specimens Stored at 45°C for Two Weeks

	% Loss in Weight						
	A12S	A152S	Copper	Brass	Monel	Cold R. Steel	St. Steel
*TCE/5% RH-195	0.28	0.24	5.09	1.15	0.35	1.91	1.33
*Perclene/5% RH-195	0.20	0.91	0.89	0.79	0.07	0.24	0.21
Perclene/pot. oleate/S-461	0.09	0.08	0.73	1.31	0.03	0.45	0.00
Perclene/pot. oleate/S-461/ZnO	0.10	0.09	0.41	0.49	0.02	0.49	0.00
Perclene/pot. oleate/S-210	0.07	0.04	0.14	0.14	0.04	0.21	0.00
Perclene/pot. stearate /S-461	0.05	0.07	0.52	1.70	0.33	0.45	0.00
Perclene/pot. stearate/S-461/ZnO	0.10	0.14	0.67	1.38	0.03	0.52	0.00

*Test made at 50°C

Specimens Stored at R.T. for Four Weeks

	% Loss in Weight						
	A12S	A152S	Copper	Brass	Monel	Cold R. Steel	St. Steel
TCE/5% RH-195	0.08	0.38	0.94	3.36	0.08	1.01	1.06
Perclene/5% RH-195	0.07	0.12	0.59	2.66	0.17	0.62	0.44
Perclene/pot. oleate/S-461	0.07	0.07	0.24	0.35	0.03	0.44	0.00
Perclene/pot. oleate/S-461/ZnO	0.15	0.29	0.22	0.23	0.02	0.62	0.00
Perclene/pot. oleate/S-210	0.10	0.00	0.10	0.10	0.00	0.06	0.00
Perclene/pot. stearate/S-461	0.31	0.25	1.57	0.82	0.03	1.31	0.00
Perclene/pot. stearate/S-461/ZnO	0.34	0.27	1.19	0.66	0.04	1.55	0.00

[redacted]

occurred in the first few days in storage. Thereafter, no changes were observed during three months of the test.

Table V

Storage of Perclene Mixtures

	Description			
	When	After storage at		
Additive	Mixed	R.T.	45°C	0°C
Potassium oleate	Paste	Separated slightly	Separated partially	No change
Potassium stearate	Paste	No change	Separated partially	No change
Diglycol stearate	Partially separated	No change	Liquefied	Solidified
Diglycol laurate	Liquid	No change	No change	No change

44. The Perclene/potassium oleate mixture separated partially at R.T. and 45°C, but was easily mixed again by stirring. The mixture darkened gradually on storage, especially at the higher temperatures. The mixture could be used at any of the three storage temperatures. Potassium stearate separated from the Perclene somewhat at 45°C. No appreciable change was observed at other storage temperatures. The Perclene/diglycol stearate mixture was much affected by temperature changes. It was liquid at 45°C and quite solid at 0°C. It would be difficult to handle this mixture at 0°C and mix it with the chloroamide. Perclene/diglycol laurate is a fluid solution at 0°C to 45°C. Very little thickening by the diglycol laurate or diglycol stearate was observed which means that a larger proportion of chloroamide would be necessary to make a suitable paste.

(b) Perclene Mixtures with Chloroamides Added.

45. Pastes were prepared as described in paragraph 20. After analyzing for active chlorine, the pastes were stored at 45°C in glass bottles. The ingredients of the pastes and the percent loss of active chlorine at 45°C are given in Table VI.

[redacted]

Table VI

Stability of Pastes at 45°C

	% Loss of Active Chlorine			
Composition of Paste	24 hrs.	48 hrs.	1 wk.	2 wks.
Perclene/pot. oleate/ S-461		46.5		62.3
Perclene/pot. stearate/ S-461	9.1	7.5		6.5
Perclene/diglycol stearate/S-461	7.2	20.8	47.0	
Perclene/diglycol laurate/S-461	carbonization with 24 hours			
Perclene/pot. stearate/ RH-195	36.0	67.3		
Perclene/diglycol laurate/RH-195	0.0	7.0	34.1	
Perclene/diglycol stearate/RH-195	0.0	5.0	18.5	

46. The Perclene/potassium oleate/S-461 paste lost about 50% of its active chlorine within 48 hours at 45°C. This paste mixture cannot be stored for more than a few days. Of the four S-461 pastes, the one made using potassium stearate was quite stable, losing only about 6-7% of its active chlorine within two weeks at 45°C. None of the pastes made using RH-195 were stable for more than a few days at 45°C.

47. Because of the superiority of the potassium oleate paste in many other respects, an attempt was made to stabilize this paste by the addition of ZnO. The amounts of ZnO used were 10% and 25% on the weight of S-461. The loss of active chlorine as compared to the paste containing no zinc oxide is shown in Table VII, It is clearly evident from these data that zinc oxide is not a stabilizer for the prevention of loss of active chlorine in the Perclene/potassium oleate/S-461 paste.

Table VII

Stability of Pastes with ZnO

	% Loss of Active Chlorine			
	24 hrs.	48 hrs.	72 hrs.	2 wks.
Perclene/pot. oleate/S-461	-	46.5	-	62.3
Perclene/pot.oleate/ S-461 + 10% ZnO	50.7	-	59.0	74.3
Perclene/pot.oleate/ S-461 + 25% ZnO	45.0	63.5	-	91.0

C. Discussion of Perclene Paste Systems for Decontamination

(1) General Considerations.

48. In the experimental work given thus far major emphasis has been placed on the evaluation of the Perclene/potassium oleate/S-461 paste and its comparison with pastes using potassium stearate, diglycol stearate and diglycol laurate instead of potassium oleate. The potassium oleate paste appeared on the whole to be the most satisfactory of the pastes studied. Some of the unsatisfactory characteristics of the other pastes might well be summarized here.

49. Potassium stearate/Perclene/S-461 paste did not decontaminate as well as that with potassium oleate and did not adhere as well to painted surfaces, although it was more stable to loss of active chlorine. The potassium stearate paste made with RH-195 was decidedly inferior in decontaminating efficiency. Potassium stearate required heating to mix well with Perclene. Diglycol stearate/Perclene mixtures were too much affected by temperature changes. On cooling to 0°C, the mixture solidified and at 45°C it was liquid. It was necessary to heat the Perclene/diglycol stearate to obtain a good mixture. Diglycol stearate did not thicken Perclene to any extent at ordinary temperatures. Diglycol laurate/Perclene was a liquid solution and required excessive amounts of chloroamide to thicken it into a paste.

(2) Other Considerations Concerning Perclene/Potassium Oleate/S-461 Paste.

50. Storage of Ingredients: It was found that the Perclene/potassium oleate mixture can be stored indefinitely at

temperatures ranging from 0°C to 45°C. It is claimed by the NDRC that storage is satisfactory at temperatures as low as -15°C. There is slight separation at 45°C but a homogeneous mixture is easily made by stirring. S-461 is inflammable and not suitable for shipboard storage. Addition of equal weights of bentonite or Fuller's earth will cut down the inflammability to a safe degree. To date, the storage characteristics of these mixtures are not well known. S-210 is not inflammable and is more stable to loss of active chlorine than RH-195. S-210 was found to decontaminate satisfactorily when substituted for S-461 in the Perclene/potassium oleate/S-461 paste, although S-210 has less than half as much active chlorine as S-461.

51. Availability and cost of materials: Perclene costs about the same as TCE and its use is also under allocation by WPB. Potassium oleate is readily available at a fairly reasonable cost. S-461 is in commercial production at a cost less than that for RH-195. S-210 has been prepared in laboratory amounts and probably can be produced commercially without much difficulty at a cost somewhat higher than that for RH-195.

52. Methods of application: The potassium oleate paste can be applied by brush or spray. It may be necessary to use a somewhat thinner paste for spray, and straining through a sieve before filling the spray pump may be advisable. Several trials at decontaminating a 3-inch Navy gun, partially contaminated with H and nitrogen mustard, showed that the difficulty of application varied with the proportions used. The best proportions for optimum results have not yet been determined.

53. Ease of Removal of the Paste: The Perclene/potassium oleate/S-461 paste is somewhat difficult to remove by hosing with water, but hosing with water combined with scrubbing is satisfactory.

54. Incorporation of a Dye in the Paste: It is likely that any paste used on shipboard would have to be colored to camouflage it because the chloroamides used are white. Preliminary work indicates that suitable pigments can be incorporated into the Perclene/potassium oleate mixture which will mask the paste satisfactorily.

55. Toxicity of Paste and Ingredients: The toxicity of Perclene vapor is relatively low when compared to TCE.

Its volatility will be much lower when applied as a paste. The other ingredients of the paste are not particularly toxic or irritating. However, the paste itself is somewhat irritating to the skin and should be washed from the skin as soon as possible.

EXPERIMENTAL PART II. AQUEOUS PASTE SYSTEMS FOR DECONTAMINATION

56. Because of the desirability of eliminating all or part of the organic solvent used with chloroamides, a more intensive investigation of aqueous systems for decontamination of vesicants was begun. It had been found earlier that a paste mixture of water and a chloroamide such as S-461 or RH-195 will decontaminate H in deck paint in about 24 hours. Such a paste does not adhere well to vertical surfaces and a large amount of chloroamide is necessary to thicken the paste in comparison with the Perclene/potassium oleate/S-461 paste described in Experimental Part I. The following types of paste systems were investigated to determine their general characteristics in an attempt to overcome these objections.

A. Water + Surface-Active Agent + Chloroamide

57. In general, these paste systems were prepared by mixing 10 parts of water, 1-2 parts of surface-active agent and 5-10 parts of chloroamide. Of the surface-active agents tried the one that gave best results was sorbitan monopalmitate. Both S-461 and micronized RH-195 were used, but invariably the use of RH-195 resulted in better decontaminating efficiency. The shortest time required for completely decontaminating H in deck paint was 4-6 hours with RH-195 pastes.

58. This type of system reduced the time necessary to obtain complete decontamination from 24 hours to 4-6 hours, but other objections still remained, including the need for excessive amounts of chloroamide and the injury to the deck paint. A suitable water thickener to reduce the amount of chloroamide used would make these systems more promising.

B. Water + Water-Soluble Paint Penetrant + Chloroamide

59. These paste systems were prepared by mixing 10 parts water, 2 parts paint penetrant and 5 to 10 parts of chloroamide. The paint penetrants which gave the best results were ethyl phosphate, carbitol acetate, and ethylene glycol diacetate. The chloroamides used were

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SUMMARY

Part I.

1. A very efficient decontaminating system consisting of a paste of Perclene, potassium oleate and S-461 has been studied. The system decontaminates H, L, and HN1 on deck paint within an hour or less. It decontaminates HV (H thickened with chlorinated rubber) in a somewhat longer time. It is harmless to deck paint if left on for one hour and is less corrosive than TCE/RH-195.

2. This system decontaminates airplane lacquer within a few minutes, and a one-hour application decontaminates doped fabrics. With the exception of L, it decontaminates vesicants in clear plastics in one to two hours. It softens airplane lacquer somewhat but is harmless to the clear plastics, Plexiglas, Lucite and cellulose acetate, and to fabrics doped with cellulose nitrate or cellulose acetate butyrate.

3. The S-461 in this paste can be replaced by S-210 to some advantage since S-461 is inflammable.

Part II.

1. In the examination of water systems for decontamination, it has been found that certain water-and-oil type emulsions containing Perclene, and emulsifying agent, S-461 and more than 50% water are excellent decontaminating systems. The results would indicate that these systems are as effective for decontamination of deck paint as the Perclene/potassium oleate/S-461 paste described in Part I.

2. The use of these emulsion pastes would constitute a large saving in storage and shipping space because of the use of a large proportion of water.

3. More thorough investigation of these emulsions for decontamination is actively in progress.

RECOMMENDATIONS

63. None. The investigation continues with particular emphasis on systems containing as large a proportion of water as is possible for effective decontamination.

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