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REPORT ON

AN EMULSION-PASTE SYSTEM FOR DECONTAMINATION OF VESICANTS (C)

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NRL Report No. P-2457
NRL Problem No. P-60

NAVAL RESEARCH LABORATORY
Anacostia Station
Washington 20, D. C.

CHEMISTRY DIVISION
Protective Chemistry

Report on

AN EMULSION PASTE SYSTEM FOR DECONTAMINATION OF VESICANTS

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ABSTRACT

A new decontamination system for vesicants has been developed, which consists of a chloroamide dispersed in an emulsion of water in Perclene (tetrachloroethylene). This paste system was investigated in detail to determine the effect of varying its composition and the conditions of use on its effectiveness as a decontaminant. The improved NDRC potassium oleate paste was examined and both paste systems compared to TCE/RH-195 solution. TCE/RH-195 was found to be the most efficient and the easiest to use of the three systems for the decontamination of H in deck paint.

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INTRODUCTION

Authorization

1. This work was authorized under Bureau of Ships Project No. 397/44. 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 is a continuation of the study of the decontamination of C. W. Agents, with special emphasis on the development of a paste system containing a large proportion of water.

Known Facts Bearing on the Problem

3. The present Navy standard decontamination system for shipboard use is a 10% solution of RH-195 in tetrachloroethane (TCE/RH-195). The objectionable characteristics of this solution have been discussed in previous reports from this Laboratory. They include its toxicity, corrosiveness and injurious effect on various materials such as paint, clear plastics, rubber and cellulose acetate butyrate doped fabrics.

4. A paste system developed by the NDRC, which consists essentially of Perclene (tetrachloroethylene) potassium oleate and S-461 or S-210, has some advantages over the TCE/RH-195 system. This Perclene/potassium oleate/chloroamide paste was discussed in detail in NRL Report No. P-2211. This report also mentioned the preliminary work at this Laboratory on a promising emulsion paste containing Perclene, an emulsifying agent, a chloroamide and a large proportion of water.

Theoretical Considerations

5. For shipboard use it would be desirable to have a decontamination system requiring the storage of as small an amount of materials as possible. One means of accomplishing this might be the substitution of water for all or part of the solvent, retaining a chloroamide as the active ingredient. Preliminary examination of water systems indicated that an emulsion applied as a paste (too fluid an emulsion would run off vertical surfaces quickly) offered promise. The emulsion should contain a paint penetrant, preferably one which will dissolve certain chloroamides to some extent.

6. To decontaminate most efficiently, it was felt that the emulsion should be of the water-in-oil type so that the chloroamide-bearing paint penetrant could come in contact with the contaminated surfaces. Furthermore, previous work indicated that some water-in-oil emulsions would stick to vertical surfaces, whereas oil-in-water emulsions tended to run off quickly. The emulsion should be fairly stable so that it will not break readily when applied to a vertical surface and allow the paste to run off.

7. In the preliminary work toward development of an emulsion paste a suitable water-insoluble solvent was needed. Perclene (tetrachloroethylene) was selected as the most likely solvent to use on the basis of certain desirable characteristics found in earlier work. One disadvantage of Perclene as compared to TCE is that Perclene is much more volatile than TCE at ordinary temperatures. It was felt that incorporation in an emulsion would increase the contact time of the Perclene with the painted surfaces, thereby increasing the efficiency of the solvent.

Previous Work Done at this Laboratory

8. Previous work done at this Laboratory related to this problem has been presented in NRL Report No. P-1914, "The Use of RH-195 for the Decontamination of HS and M-1", dated 8 October 1942; NRL Report No. P-2125, "A Study of Perclene (Tetrachloroethylene) as a Solvent for use in the Decontamination of Airplanes", dated 29 July 1943; and NRL Report No. P-2211, "Chloroamide Paste Systems for Decontamination of Vesicants" dated 31 December 1943.

EXPERIMENTAL

PART I. DEVELOPMENT OF AN EMULSION PASTE FOR DECONTAMINATION

A. Screening Tests of Emulsifying Agents

9. It was desired to make an emulsion of water-in-Perclene into which a chloroamide could be incorporated. Therefore, an arbitrary series of requirements for candidate emulsifying agents was set up as follows:

(1) The emulsifying agent must make a water-in-oil type of emulsion in which Perclene is the continuous phase.

(2) The emulsifying agent must make an emulsion which will adhere well to vertical surfaces.

(3) The emulsion must be stable when chloroamides are dispersed in it.

(4) The chloroamide-containing emulsion must be prepared easily by stirring the ingredients by hand with a paddle.

(5) The emulsifying agent should preferably be soluble in Perclene, and this solution should be stable under various conditions of storage.

(6) A somewhat viscous emulsion would be desirable because it would require less chloroamide powder to make a suitably thick paste.

Much of the preliminary work consisted of screening the emulsifying agents on the basis of these requirements. Additional factors were expected to influence the selection of the best emulsifying agent from those which satisfied these six requirements.

(1) Preliminary Tests

10. Over 100 surface-active agents were tested as follows: One gram of the surface-active agent was mixed with 20 grams of Perclene and the mixture examined for solubility of the agent in Perclene. Water was then added in 10 ml. increments, usually up to a total of 50 ml., and the mixture stirred after each addition with a Hamilton Beach high-speed stirrer. The mixture was then examined for (a) emulsion formation, (b) type of emulsion, (c) stability of the emulsion, and (d) thickening action. The results are tabulated in Appendix A, Table I.

11. Those agents which gave no emulsion were dropped, as were a number which gave O/W emulsions. None of the O/W type showed evidence of appreciably thickening the emulsions. Of those agents which gave water-in-oil emulsions, a number were eliminated because the emulsions broke quickly.

(2) Preparation of Hand-Stirred Emulsions Containing Chloroamides

12. The 25 agents which survived the screening tests were examined further by a procedure similar to that described above, except that hand stirring only was used. At the same time emulsions were prepared with the addition of S-461 powder. In no case did the addition of S-461 decrease the ease of emulsification or stability of the emulsion. In some cases the addition of S-461 improved the speed of emulsification. From observations based on these experiments, the emulsifying agents were divided arbitrarily into three classes: most promising, intermediate and least promising. This was the basis for the classification shown in Appendix A, Table II. In the most promising class were eight emulsifying agents, all of which were long chain fatty acid esters of sorbitan, mannitan or mannide, and all were products made by the Atlas Powder Co.

(3) Decontamination Tests on Deck Paint

13. Emulsion pastes were made up using the eight most promising emulsifying agents with S-461, RH-195 and S-210, according to the following formula:

20 g. Perclene
1 g. Emulsifying Agent
30 g. Tap water
5 g. S-461, RH-195 or S-210

14. Decontamination tests were made in the following manner: Two-inch square steel panels previously painted with zinc chromate primer and two coats of deck paint were contaminated with 2 drops (0.05 g.) of H spread on and allowed to stand for one hour. The decontamination paste was then liberally applied and left for one hour, then washed with water under the tap, the panels blotted and dried and tested at 35°C with Congo Red-S-328 test paper, in conjunction with sodium carbonate filter paper. The paper test times were recorded in minutes and all tests which were still negative after 45 minutes were recorded as negative.

15. The results obtained for the decontamination of deck paint with the emulsion pastes are given in Appendix A, Table III. Satisfactory decontamination was obtained with all pastes except S-461 and RH-195 with mannitan triricinoleate, RH-195 with mannitan diricinoleate and S-210 with Span 40 and Span 60.

In general, pastes made with S-461 were most viscous, less viscous with S-210, and the RH-195 pastes were thin. Mannitan diricinoleate and mannitan triricinoleate emulsions were the least satisfactory with all three chloroamides. They gave poor emulsions which broke and dried out on the panels, especially with RH-195. Arlacel A was somewhat better, but not as good as the remaining five emulsifying agents. Span 40, Span 60 and mannitan monolaurate made the thickest pastes when used in the above formulation. It was later learned that the manufacture of derivatives of mannitol had been suspended. The mannitan derivatives were in general no better than those of sorbitan. Therefore, only a few additional experiments were made with mannitan esters.

B. Factors Affecting the Efficiency of Emulsion Pastes for the Decontamination of H:

16. Because of the promise shown by the emulsion pastes in preliminary tests, a study was made of the effect of changing the proportions and ingredients. At the same time the decontamination efficiency for H under various conditions was studied.

(1) Effect of Varying the Perclene/Water Ratio

17. The first tests were made with a Perclene/water ratio of 20/30 by weight. In another experiment emulsion pastes of S-461 were prepared in which the amount of water was increased to give a ratio of 20/50. This data is included in Appendix A, Table III. These pastes decontaminated about as well as did similar 20/30 pastes. The increased water content made the pastes more viscous.

18. In another experiment four S-461 emulsion pastes were prepared using a 20/80 ratio of Perclene/water. The emulsifying agents used were Arlacel C, Span 80, Span 60 and Span 40. These pastes decontaminated H satisfactorily as shown in Appendix A, Table III. However, increasing the Perclene/water ratio to 20/80 made it more difficult to prepare the emulsions by hand. The emulsions were less stable and allowed water to separate.

19. The Perclene/water ratio was varied also in other experiments. It was found that a smaller proportion of water gave less viscous emulsions which tended to run off vertical surfaces. A larger proportion of water gave unstable emulsions which were more difficult to prepare by hand stirring. As a result of these experiments a 20/50 ratio of Perclene/water by weight was chosen as most likely to give good results.

(2) Effect of Varying the Amount of Emulsifying Agent

20. Pastes with a Perclene/water ratio of 20/80 were prepared using 5% S-461, RH-195 or S-210. The amount of emulsifying agent, Span 40 or Span 80, was varied from 1%, 5% to 10% of the weight of Perclene. These pastes were tested for decontamination of H in deck paint. All the S-461 and RH-195 pastes decontaminated H completely. The data for the S-210 pastes is given in Appendix A, Table IV.

21. There was no significant difference in decontamination efficiency due to variation in percentage of emulsifying agent. The emulsions made using 1% Span 40 or Span 80 were difficult to prepare and broke quickly. The 5% and 10% emulsions were satisfactory except for the RH-195 pastes which were less stable. The 10% emulsifying agent emulsions were the most viscous, the 5% somewhat less viscous and the 1% were quite thin. This effect was noted in other experiments in which the amount of emulsifying agent was varied from 5% to 10% and the Perclene/water ratio was 20/30, 20/50, 20/65 and 20/80. On the basis of these and other experiments, a 10% solution of emulsifying agent in Perclene was considered to be the most satisfactory.

(3) Choice of Chloroamide

22. As a result of a number of preliminary experiments some of which have already been described, the characteristics of RH-195, S-461 and S-210 in the emulsion pastes had been noted. The RH-195 and S-210 used had been micronized. The emulsions containing S-461 were stable and the decontamination efficiency was good. S-210 emulsion pastes were not as viscous as those made with S-461 and did not decontaminate quite as well, although they were stable. RH-195 emulsion pastes were thin, unstable with "leaking" of water, and were not suited for application to vertical surfaces. When applied to horizontal panels decontamination was usually obtained.

23. Of the chloroamides listed above, S-461 gave the best emulsion pastes However, S-461 is easily ignited and decomposes spontaneously after ignition. A number of other chloroamides and mixtures of S-461 with materials designed to inhibit the decomposition of S-461 on ignition were tested for use in emulsion pastes. Attempts were made to prepare 20/50 type emulsions containing 5% by weight of Span 20 (sorbitan monolaurate) or Span 80 (sorbitan monooleate) in Perclene and containing the chloroamides or mixtures listed below in the amount of 10% of the final paste:

(a) S-330 (micronized) made good stiff emulsions with both Spans, with no sign of breaking after 3 hours.

(b) S-222 made good, fairly stiff emulsions which were still quite good after 3 hours.

(c) S-300 emulsions made with Span 80 broke within a few minutes. They were readily re-emulsified, but were thin and broke quickly. S-300 emulsions made with Span 20 were much better than with Span 80, although they "leaked" some after an hour and were definitely thinner after three hours.

(d) S-145 emulsions began to break after about one hour. Span 20 was better than Span 80.

(e) RH-851 emulsions with Span 20 had broken slightly within an hour; with Span 80 they broke quickly and became quite thin.

(f) S-436 reacted vigorously with Span 80 with the evolution of heat. However, stable viscous emulsions containing S-436 were prepared using Span 20.

(g) S-461/Infusorial earth mixtures in Perclene prevented emulsification.

(h) Infusorial earth prevented emulsification.

(i) S-461/Bentonite mixtures prevented emulsification.

(j) Bentonite prevented emulsification.

24. Of those listed above, S-330, S-222 and S-436 made the best emulsions and were tested for the decontamination of H in deck paint. The results are given in Appendix A, Table V. Except for S-330 pastes made with Span 80, none of these pasts decontaminated satisfactorily. The pastes made with these chloroamides "skated" over the panels where liquid mustard was still standing and did not mix well with the H. This is in contrast to S-461, S-210 and RH-195 pastes, which seemed to absorb liquid H readily, adhering as well to the places where liquid H was still standing as where liquid H was not present.

(4) <u>Substitution of Sea Water for Tap Water</u>

25. The emulsion pastes studied thus far were made with tap water. Because it would be desirable to be able to make these pastes with sea water as well as fresh water, some emulsions were prepared using a "synthetic" sea water. Emulsion pastes prepared with sea water, Perclene, S-461 and mannitan monolaurate, Span 40, Span 60 or Span 80 were thin and broke quickly. They were unsatisfactory for application to vertical surfaces, but when applied to horizontal deck paint panels decontaminated H satisfactorily. Sea water emulsion pastes made with S-436 had fairly good physical properties, but decontaminated H poorly as did similar S-436 emulsions made with tap water.

26. It was found possible to make fairly satisfactory emulsion pastes with sea water by adding polybutene or other stabilizers in addition to the emulsifying agent. In the event that emulsion pastes should be considered practical for use, suitable modification could doubtless be made to allow the use of sea water in the formulations.

(5) <u>Length of Time of Application of Emulsion Paste</u>

27. All previous trials for decontamination of H in deck paint were made by leaving the paste on for one hour. S-210 pastes using both Span 20 and Span 80 were used to decontaminate H in deck paint, the paste being left on for 20, 40 or 60 minutes. The results of these tests are given in Appendix A, Table VI. It is apparent that 20 minutes is not sufficient time and 60 minutes gives somewhat better results than 40 minutes. On the basis of these results it was judged that a 60-minute application should be used.

(6) Effect of Temperature on Preparation and Decontamination

28. Pastes were made using Span 20 or Span 80, Perclene, S-461 and water as follows: (1) ice water, (2) tap water at room temperature, and (3) tap water at 70°C. Water at all three temperatures was easily emulsified. There was a slight difference in the viscosity of the pastes, the hot water pastes being less viscous than the cold. The emulsion pastes made with ice water were kept at 4°C, those made with room temperature water were kept at room temperature, and those made with 70°C water were kept at 45°C for one-half hour. There was no observable change in these pastes during that time.

29. The decontamination of H in deck paint at low temperatures was tested as follows: Deck paint panels were contaminated at room temperature and left for 45 minutes, then placed outdoors (11°C) for 15 minutes and finally kept at 3°C for 15 minutes before the paste was applied. The pastes were 20/50 type with S-210 and were prepared from (1) 5% Span 20 solution in Perclene kept at 3°C for 6 days, (2) 10% Span 20 solution in Perclene kept at 3°C for 10 days, and (3) 5% Span 80 solution in Perclene kept at 3°C for 18 days. The water used was at 3°C. There were no significant differences in the ease of preparation or stability of these pastes compared to pastes prepared from similar materials previously brought to room temperature. The pastes prepared at 3°C were applied to the H-contaminated panels and left at 3°C for one hour, then the pastes were removed and the panels tested at 35°C. All results showed complete decontamination under these conditions.

(7) Sequence of Addition of Ingredients

30. It was found that S-461 emulsion pastes could not be prepared by first mixing the S-461 with water, then adding the Perclene solution of the emulsifying agent. Emulsifying agents tried were Span 40, Span 60 and Span 80. Emulsion pastes containing S-210 and RH-195 could be prepared by mixing the chloroamide and water first. Emulsion pastes of all three chloroamides were successfully prepared by adding the ingredients in the following order: Perclene solution of Span, water, chloroamide; or Perclene solution of Span, chloroamide, water. The latter procedure was the easiest to use. It was concluded that the best method of preparing the emulsion pastes was to wet out the chloroamide in the Perclene solution of the emulsifying agent first, and then add the water with vigorous stirring.

(8) The Use of Other Solvents

31. Although Perclene was a desirable decontamination solvent in many ways, certain tests indicated that Perclene was more volatile than desired, even when used in the emulsion pastes. For this reason several other solvents were examined.

32. The characteristics of the solvents were as follows:

Hexachlorobutadiene (Hooker Electrochemical Company) is non-inflammable, b.p. 210-220°C., m.p. -19 to -22°C., and is not easily hydrolyzed by water or mild alkalies. Emulsion pastes made with this solvent were physically satisfactory and did not harm deck paint appreciably.

Hexachloropropene is non-inflammable and has a high boiling point and a low melting point. It likewise was readily emulsified and did not harm deck paint.

Chloropropane Liquid 170 (Hooker Electrochemical Company) is non-inflammable and has a boiling range of 160° to 260°C and does not freeze but becomes quite viscous below -50°C. Good emulsion pastes were made with this solvent which did not harm deck paint.

Butyl laurate is a relatively non-inflammable, high-boiling solvent with a melting point of less than -10°C. Emulsions made with this solvent were not very stable, although there was no indication of a reaction between chloroamide and solvent.

33. The results of decontamination tests with pastes made using these solvents are given in Appendix A, Table VII. None of the emulsion pastes containing these solvents decontaminated deck painted panels very well. Slurries of chloroamides mixed with these solvents did not decontaminate well either. Determinations of the solubility of chloroamides in hexachlorobutadiene and hexachloropropene (Table VIII) indicated that the failure to decontaminate was probably not due to lack of solubility of the chloroamides in these solvents.

34. Several other solvents were tried, but they did not perform well in the pastes. Tetrachloroethane made good emulsion pastes, which, however, completely stripped the paint from the test panels, so that no tests were made for completeness of decontamination. It was concluded that Perclene was the best solvent of those tried for use in these emulsion pastes in spite of its volatility.

(9) Conclusion

35. As a result of the studies outlined above, the following ingredients, proportions and procedure were selected for the preparation of the emulsion type decontamination pastes:

Ingredient	Parts by Weight
Perclene	18
Span 20 (sorbitan monolaurate) or Span 80 (sorbitan monooleate)	2
S-461 or S-210	7
Water	50

The paste is prepared by mixing the chloroamide with the Perclene solution of the emulsifying agent and then adding the water with stirring. This paste is then applied to the contaminated surface and left for one hour, after which it is flushed off with a stream of water, using brushes if necessary.

C. Decontamination of H in Various Materials

(1) Wet Deck Paint

36. In the tests of the emulsion pastes for the decontamination of H described thus far, steel panels painted with deck paint were used exclusively.

The panels used were dry. Further tests were made to determine whether the emulsion pastes would decontaminate deck paint wet with fresh water or sea water. After the H had been left on the panels for one hour, they were wet down with fresh water and sea water respectively, and then the emulsion paste was applied. The pastes adhered very well to the wet panels and did not harm the paint. The paper test times are given in Appendix A, Table IX. It is evident that decontamination of H in deck paint was successful under these conditions also. It may be noted here that the emulsion pastes did not have any harmful effect on deck paint, wet or dry.

(2) Airplane Paints

37. Decontamination tests were made for H on both M485 and DuPont 71 Line lacquers over zinc chromate primer on steel. These panels were contaminated with H and decontaminated with the pastes in the same manner as were the deck paint panels. The results of the tests on M485 lacquer are given in Appendix A, Table X. The M485 paint was considerably softened so that some of it was removed during the removal of the paste. The DuPont 71 Line panels were decontaminated completely by the emulsion paste treatment even after 20 minute application, and were apparently unharmed by the treatment.

(3) Doped Fabrics

38. S-461 and S-210 emulsion pastes using Span 20 or Span 80 were used to decontaminate H in doped fabrics, both cellulose nitrate and cellulose acetate butyrate types. The results are given in Appendix A, Table XI. The cellulose nitrate doped fabrics were decontaminated but the cellulose acetate butyrate fabrics were not. No harmful effect was noted on either type of coating. The experiment was repeated on cellulose acetate butyrate panels, except that the pastes were left on for two hours. Slightly better results were obtained. To determine the effect on the tensile strength of fabrics contaminated with H and decontaminated with the pastes, or fabrics treated with the pastes only, measurements were made on the series listed in Appendix A, Table XII. Each contamination and decontamination treatment was made three times at three day intervals. Where H was used, it was left at least one hour before applying the pastes. The tensile strength measurements (one-inch grab test) were made after two weeks outdoor exposure following the last treatment. Each figure given in the table is the average of four measurements. The data show that tensile strength of these fabrics was practically unaffected by the emulsion pastes applied with or without previous contamination with H.

(4) Clear Plastics

39. Decontamination of H in Lucite, Plexiglas and cellulose acetate sheet was attempted with pastes of the 20/50 Perclene/water type made with S-210 and S-461. Span 20 and Span 80 were the emulsifying agents. Two drops (0.05 g.) of H was left on 2" x 2" plastic panels for one hour and the paste applied and left for one hour. All the panels were completely decontaminated except one Plexiglas panel, which gave a negative test after 24 hours. No damage to the plastics was apparent except that caused by the exposure to liquid H.

D. Decontamination of Other C. W. Agents

(1) Decontamination of HV

40. Emulsion pastes of the 20/30 Perclene/water type made with S-461 using 5 different emulsifying agents were used to decontaminate HV (H containing 8% chlorinated rubber) on deck paint panels. The HV was left on for one hour before application of the paste. The results of the paper tests are given in Appendix A, Table XIII. The decontamination was good and the paint was not harmed.

(2) Decontamination of L

41. S-461 emulsion pastes of the 20/30 Perclene/water type, using 5 different emulsifying agents were used to decontaminate L on deck paint panels. The L was allowed to remain for one hour before decontamination was attempted. DT paper was used to detect any residual L after decontamination. If no positive test was obtained in 120 minutes at 35°C the test was recorded as negative. The results are given in Appendix A, Table XIII. In all cases decontamination was complete. The panels were bleached and pin-point blisters were numerous on each panel wherever L had been spread. This is the usual effect of L on deck paint.

(3) Decontamination of Nitrogen Mustards

(a) HN-3

42. Deck paint panels (2" x 2") were each contaminated with 0.05 g. of HN-3. After one hour, decontamination was attempted by several different methods. Tests for residual HN-3 were made with sodium carbonate papers spotted with DB-3 reagent used in conjunction with a filter of sodium carbonate paper. If no positive test was obtained within 30 minutes, the tests were reported as negative. The results are given in Appendix A, Table XIV. In addition to emulsion pastes of S-210 and S-461, decontamination was attempted by spraying the panels with a solution of RH-195 or S-436 in TCE. Comparison with the blank shows that all the methods used were helpful in the removal of HN-3 but that the spray of TCE/S-436 was outstanding in its beneficial effect.

(b) HN-1

43. Tests similar to those for HN-3 were made using HN-1 as the contaminant. The results are given in Appendix A, Table XV. The S-436 emulsion paste was the best decontaminating system and the spray of TCE/S-436 was good. None of the other methods were very effective, although the surface contamination was removed.

(4) Decontamination of Lachrymators

44. Some attempts were made to decontaminate CN and BBC in deck paint. The emulsion pastes were not very effective in the removal of either of these lachrymators. The Perclene/potassium oleate/S-461 paste discussed in a previous report was very effective in removing CN but did not decontaminate BBC completely.

E. Other Considerations Regarding Emulsion Pastes

45. The emulsion pastes were examined in regard to several other properties of importance for a decontaminating system.

(1) Storage of Perclene/Emulsifying Agent Solutions

46. Solutions of Span 20, Span 40, Span 60 and Span 80 in Perclene were stored at 5°C., room temperature and 45°C. for 4 weeks. The results are given in Appendix A, Table XVI. At 5°C there was considerable separation of Span 40 and Span 60 from a 5% solution. The solutions of Span 20 and Span 80 showed no separation except for a slight amount of insoluble material which did not increase in amount on standing. Solutions filtered before storage did not show this slight separation.

47. Perclene solutions of Span 20 (5%) and two different samples of Span 80 (each 10%) were placed at -20°F for 24 hours. All the samples were frozen. When warmed to room temperature each melted with the separation of the Spans which redissolved easily when the mixtures were agitated. The samples were again stored at -20°F. After 48 hours the temperature was raised to 0°F. The solutions were homogeneous when examined 24 hours after raising the temperature. The samples were then stored at room temperature for 5 months. There was no separation or evidence of reaction or decomposition of the components.

48. Solutions of Span 20 and Span 80 in Perclene made satisfactory emulsion pastes after eight months storage at room temperature, which included summer temperatures exceeding 30°C at times. There was no evidence that any deterioration of the solutions had occurred.

(2) Storage of Prepared Emulsion Pastes

49. The stabilities of emulsion pastes made with Span 20 or Span 80 and S-461, S-210 or RH-195, stored at room temperature and 45°C are tabulated in Appendix A, Table XVII. Examination of the data shows that S-210 pastes lost no active chlorine when kept at room temperature for 8 weeks. S-461 pastes made with Span 20 were likewise stable for 8 weeks but those made with Span 80 began to deteriorate after 4 weeks at room temperature. Pastes made with RH-195 were somewhat less stable than those made with S-461.

50. At 45°C, the S-210 pastes did not show loss of active chlorine even after 8 weeks. S-461 pastes were fairly stable for two weeks and RH-195 pastes showed considerable loss of active chlorine after one week.

(3) Addition of Thickening Agents

51. A number of formulations for emulsion pastes were made using polybutene and aluminum soaps as thickeners. The thickeners were dissolved in the Perclene after solution of the Span. In every case the resulting emulsion was easy to prepare, smooth and gave more effective adhesion to painted panels. Decontamination of H was effective, but the dried paste films containing the thickeners were more difficult to remove completely from the panels, except where aluminum oleate was the thickener. Because of the better adhesion, it is to be recommended that, if emulsion pastes are considered for use, a thickener such as aluminum oleate be added to the extent of 1 or 2% of the Perclene solution. Such solutions including Span 20 or Span 80 show no separation or deterioration after storage for 8 months at 5°C.

(4) Corrosive Action of Emulsion Pastes on Metals

52. It was observed that the emulsion pastes were less corrosive to metals than was TCE/RH-195 solution. The NDRC group studying decontamination at the DuPont Experimental Station also observed this and stated that the emulsion pastes were about as corrosive as the Perclene/potassium oleate/S-461 paste. This latter system was found to be much less corrosive than TCE/RH-195 solution.

(5) Storage of S-461 in Perclene/Span 80 Solutions

53. S-461 powder is believed to be unsuitable for storage aboard ship because of its inflammability. Numerous attempts have been made at this and other laboratories to reduce the hazard by the addition of water, hydrated salts and inert materials such as bentonite. None of these attempts proved successful from a practical standpoint. Likewise mixtures of S-461 with as much as 60% of a non-inflammable chloroamide, such as S-210 and S-436 did not inhibit the decomposition of S-461 sufficiently.

54. Because of the stability of the emulsion pastes to loss of active chlorine, it seemed possible that S-461 might be stored in the Perclene/Span solution. Accordingly, mixtures of S-461 in Perclene/Span 80 solution were put in storage at room temperature, 45°C and 60°C. The storage tests were dropped after 3 weeks because of lack of stability of the mixtures due to loss of active chlorine. The results are given in Appendix A, Table XVIII.

(6) Attempts to Camouflage Emulsion Pastes

(a) Addition of Dispersible Pigments

55. A number of S-461 emulsions were prepared using Span 80 as the emulsifying agent and with a Perclene/water ratio of 20/30, 20/50 or 20/80. DuPont numbers TLX-1A and TLX-68 were blue pigment pastes and TLX-69 was a carbon black paste. Suitable blue-gray pastes could be prepared by mixing one of the blue pigments with TLX-69. The addition of TLX-1A made it difficult to prepare the emulsions and the resulting emulsions were less stable. TLX-68 and TLX-69 did not affect the emulsions. It has been reported by the NDRC that carbon black cannot be stored with chloroamides without loss of active chlorine. Consequently, attempts were made to store TLX-68 and TLX-69 as a dispersion in the Perclene/Span solution. TLX-68 alone showed very little separation after three weeks, but TLX-69 and a mixture of TLX-68 and TLX-69 showed considerable separation within a few days. Because of this separation, other means of camouflaging the pastes were sought.

(b) Addition of Perclene-soluble dyes

56. A number of oil soluble black and blue dyes were obtained and tested as follows. One gram of the dye was dissolved in 100 g. of Perclene. Solubility and color of the solution was noted. Emulsion pastes were then prepared using Span 80, S-461 and a 20/50 ratio of Perclene/water. The color of the paste, both wet and dry, was recorded. The results are shown in Appendix A, Table XIX. Mixtures of Oil Black 5115 PDR, Oil Black 24087 or Calco Oil Black 8603 with AZP Oil Blue Black B, Calco Oil Blue Goo or Sudan Blue GA 247459 incorporated in the pastes gave satisfactory camouflage colors. Storage of these dyes in Perclene/Span 80 solutions was satisfactory. It was concluded that the use of Perclene soluble dyes was the most satisfactory way of coloring the pastes.

PART II. COMPARISON OF THE EMULSION PASTES WITH OTHER DECONTAMINATING SYSTEMS

A. Decontamination Efficiency

57. A number of experiments were carried out in an attempt to determine, for each of four decontamination systems, the relative coverage in sq.yd./gal. which would effectively decontaminate H in deck paint. At the same time observations were made regarding the ease of handling the various systems. The systems used were TCE/RH-195 solution, S-461 or S-210 emulsion pastes, Perclene/potassium oleate/S-461 paste and a modified potassium oleate paste. This improved potassium oleate paste is described in NDRC Report OSRD No. 3927, "Improved Decontaminating Systems", August 1, 1944. The NDRC (DuPont Group) found that the addition of a special wax (Aristowax 160/165) to the potassium oleate/S-210 paste considerably improved its decontaminating efficiency and made it somewhat easier to remove than earlier pastes of this type.

58. Each of these systems would require the storage of two components. Representative formulations used were as follows:

	Parts by Weight	
System	Component A	Component B
TCE/RH-195	1 part RH-195	10 parts TCE
Emulsion Paste *	1 part S-461	2.6 parts Perclene 0.3 parts Span
K Oleate Paste	1 part S-461	3.3 parts Perclene 1.7 parts K Oleate
Improved K Oleate Paste	1 part S-210	0.53 parts Perclene 1.75 parts K Oleate (28% H_2O) 0.27 parts $Ba(OH_2).8\ H_2O$ 0.15 parts Aristowax 160/165

* Exclusive of water used for make-up.

59. Navy 20B blue deck paint on steel was the surface used in all the decontamination tests. Small panels (2" x 2" and 6" x 12") were used indoors at room temperature and outdoors at temperatures as low as 45°F. A 3-inch Navy gun and mount was decontaminated at temperatures from 45°F to 80°F. Steel panels, 36" x 72", were used in experiments, most of which were carried out in a large gas chamber at 95°F. In all cases where used, TCE/RH-195 was sprayed on the contaminated panels. In the larger scale tests both the Navy 3-gallon and the Army 3-gallon sprayer were used. The Army sprayer was the more convenient to use in applying the solution efficiently to a small area. In the larger scale tests, the pastes were sprayed with the 3-gallon sprayers or applied with a paint brush. Details of some of these experiments are given in Appendix B.

60. On the basis of the data from Appendix B, it was estimated that, with most efficient usage of each system, H contamination of 0.75 oz/sq.yd. could be decontaminated safely by the application of 0.7 lb. of TCE/RH-195 solution, 1.5 lb. of S-461 emulsion paste, 1.5 lb. of K Oleate/S-461 paste, or 0.9 lb. of Improved K Oleate paste per square yard of contaminated surface. The pastes were applied by brushing in most cases. The TCE/RH-195 was sprayed twice with a 10-minute interval between sprayings. The density of each system was found to be: TCE/RH-195, 13.2 lb/gal.; emulsion paste, 9.5 lb./gal; K Oleate/S-461 paste, 11.3 lb/gal; and Improved K Oleate paste, 12.1 lb./gal. From these figures was calculated the coverage in sq.yd./gal. for each system. These figures are tabulated below:

System	Lb/yd^2 Needed	Density Lb./Gal.	Coverage Sq.Yd/gal.	Coverage Sq.yd./3 gal. sprayer
TCE/RH-195	0.7	13.2	19.0	57
Emulsion Paste	1.5	9.5	6.3	19
K Oleate Paste	1.5	11.3	7.5	22
Improved K Oleate	0.9	12.1	13.4	40

From these figures, it is evident that TCE/RH-195 solution would be the most convenient to use because it will cover a larger area than that covered by a similar volume of paste.

B. Storage Requirements

61. Using the values for the coverage by each system obtained in the preceding paragraph, the relative weight and volume requirements necessary for decontaminating a square yard of surface would be as follows:

	Weight to be carried (Pounds)		
	Solvent or Solvent Mixture	Chloroamide	Total
Emulsion Paste, S-461	0.39 *	0.14	0.53
TCE/RH-195	0.63	0.07	0.70
K Oleate Paste, S-461	1.25	0.25	1.50
Improved K Oleate Paste	0.81	0.09	0.90

* This does not include 0.97 lb. of water needed.

	Relative Volume to be carried		
	Solvent or Solvent Mixture	Chloroamide**	Total
Emulsion Paste, S-461	0.24	0.34	0.58
TCE/RH-195	0.39	0.14	0.53
K Oleate Paste, S-461	1.00	0.62	1.62
Improved K O paste, S-210	0.56	0.18	0.74

** Assuming density of S-210 and RH-195 is 0.5 and of S-461 is 0.4.

62. These calculations show that the paste systems would require as much or more space to store the necessary ingredients than is the case with TCE/RH-195. Furthermore, if S-210 were used in the emulsion paste somewhat more paste would be required per unit area. Also it should be pointed out that for a lighter contamination of H, it might be possible to apply the TCE/RH-195 more thinly. It is doubtful that the emulsion paste could be effective in a much thinner layer because the paste would dry too quickly. The improved potassium oleate paste could be used in a somewhat thinner layer because the special wax retards the evaporation of Perclene.

C. Preparation and Application

63. The TCE/RH-195 solution is readily mixed and easily applied by spraying or with swabs. The prepared solution is stable for several weeks at moderate temperatures, but should ordinarily be prepared shortly before use. The residues should be hosed off with water, but this offers no difficulty as the residues are readily washed away.

64. The emulsion paste is readily mixed and may be applied by spraying or brushing. It is more apt to clog any spray nozzle than is the TCE/RH-195 solution, and it is more difficult to clean from the 3-gallon sprayers. The prepared paste is stable for several months at moderate temperatures, but it is not recommended that it be prepared in advance. The residues are usually readily dislodged by hosing with water but may require scrubbing with a brush in some cases. The residues are much more voluminous than for TCE/RH-195, and disposal of them will require more attention to drainage facilities.

65. The Perclene/potassium oleate/S-210 paste is easily mixed but requires more stirring than do the other two systems. The solvent mixture, before adding the S-210, will separate to some extent on storage and is difficult to remix when cold (35° - 40°F.). The prepared paste will lose much of its active chlorine within twenty-four hours and must be prepared fresh shortly before use. This paste also is more apt to clog the spray nozzle and is difficult to remove from the 3-gallon sprayer. The residues are more difficultly removed than for the other two systems and require scrubbing in addition to hosing with water. The residues from this paste are likewise voluminous.

D. Other Considerations

66. Both the emulsion paste and potassium oleate paste would require the addition of dyes or pigments to camouflage the white color of the pastes or paste residues. Satisfactory means have been worked out for doing this. TCE-RH-195 solution requires no camouflage. Also, caking of the chloroamide would increase somewhat the work of stirring up the pastes, as compared to the TCE-RH-195 solution.

E. Summary of Comparison of Systems

67. The comparisons of the three best systems under discussion are summarized in Table XX. From this table it is seen that the chief disadvantages of the TCE/RH-195 solution are its toxicity, corrosive effects on metals and harmful effect on plastics. The pastes are much better in these respects but suffer from

lower covering power, somewhat greater storage requirements and greater difficulty in application and removal. It should be noted also that S-210, which is used in the pastes, is not in production at present although it can be manufactured on a large scale.

68. The potassium oleate paste is a better decontaminant for the nitrogen mustards than are the other two systems. The potassium oleate paste will decontaminate CN, for which the other two systems are ineffective.

TABLE XX

Comparison of Decontamination Systems for Use on Shipboard

	TCE/RH-195 Solution	Improved K Oleate/S-210	Emulsion/S-210 Paste
Decontamination of H	Good	Good	Good
Decontamination of L	Good	Good	Good
Decontamination of HN	Fair	Fair	Poor
Decontamination of CN	Poor	Good	Poor
Decontamination of BBC	Poor	Fair	Poor
Decontamination of wet surfaces	Poor	Good	Good
Decon. at high temp. (95°F)	Good	Good	Good
Decon. at low temp. (50°F.)	Good	Good	Good
Inflammability of Materials	None	None	None
Availability of Materials	Good	Good *	Good *
Lowest usable temperature	About -30°F	About -4°F	About 32°F
Camouflage necessary	No	Yes	Yes
Toxicity of vapors	Bad	Good	Good
Skin irritation	Bad	Bad, if hot	Bad, if hot
Ease of Preparation	Good	Fair	Fairly good
Stability of Mixture	Good	Fair	Good
Ease of Application	Good	Fair	Fair
Ease of Removal	Good	Requires scrubbing	May need scrubbing
Disposal of Residues	Good	Bad	Fair
Effect on deck paint	Bad	Little effect	Little effect
Corrosion	Fair	Good	Good
Effect on plastics	Bad	No effect	No effect
Relative vol. of materials required	1.0	1.4	1.1
Covering power, yd^2/3-gal. sprayer	57	40	19

* S-210 is not in production at present date.

SUMMARY AND CONCLUSIONS

69. A new decontamination system, the emulsion paste, has been developed which consists essentially of a dispersion of a chloroamide in a viscous emulsion of water in Perclene. Water makes up 65% of the total weight of the system. The chloroamide giving best practical results is S-210. With the exception of its inflammability, S-461 is better than S-210. Span 20 (sorbitan monolaurate) and Span 80 (sorbitan monooleate) were found to be the best emulsifying agents for this purpose.

70. This system will satisfactorily decontaminate H in deck paint under various conditions. It will decontaminate L and HV readily, and will partially decontaminate HN-3 and HN-1, being as effective as TCE/RH-195 in this respect.

71. Cellulose acetate and methyl methacrylate clear plastics are readily decontaminated from H by the emulsion paste without further damage than that caused by H. Aircraft paints are fairly readily decontaminated from H, although M485 lacquer is softened and loosened by the paste. Cellulose nitrate doped fabric is decontaminated and cellulose acetobutyrate doped fabric partially decontaminated without damage by the paste to the coating or fabric.

72. The emulsion paste system is much less toxic, less corrosive to metals and less harmful to paint and clear plastics than is TCE/RH-195. The emulsion paste is not quite as easy to handle as TCE/RH-195 and would require slightly more storage space. The mixed paste has about one-third as much effective covering power per gallon as does the TCE/RH-195 solution.

73. An improved modification of the NDRC potassium oleate paste has been examined and found to be about twice as efficient as the earlier potassium oleate paste. This improved paste would require more storage space than either the TCE/RH-195 solution or the emulsion paste, and is more difficult to remove after decontamination. The improved potassium oleate paste has about twice as much covering power per gallon as does the emulsion paste.

RECOMMENDATIONS

74. None. The TCE/RH-195 solution is more efficient and is easier to handle than is any paste system thus far examined. The paste systems warrant consideration if the toxicity and deleterious effect of the TCE/RH-195 solution on many surfaces should ever make it necessary to use some other system.

APPENDIX A

TABLE I

Manufacturer	Agent	State	Solubility of 1 gm. Agent in 10 gm. Perclene	Ease of Emulsification	Emulsion Type	Stability of Emulsion	Thickening Action
Amer. Cyan.	Aerosol OT	solid	completely, slowly	easy	O/W	-	none
DuPont	Alkanol SA	solid	almost completely	easy	O/W	bad	none
-	Aluminum mahogany sulfonate	liquid	completely	no emulsion	-	-	-
-	Aluminum naphthenate	solid	completely on heating	" "	-	-	-
-	Aluminum oleate	liquid	completely	" "	-	-	-
-	Alum. palmitate	solid	completely on heating	"	-	-	-
-	Alum. sebacate	solid	"	" "	-	-	-
-	Alum. stearate	solid	"	" "	-	-	-
-	Aresklene 375	liquid	completely, slowly	easy	O/W	bad	none
Atlas	Arlacel A	liquid	completely, quickly	"	W/O	good	good
"	Arlacel C	liquid	completely, quickly	"	W/O	good	good
"	Arlex	solid	insoluble	-	-	-	-
DuPont	Avitex SF	soft paste	disperses readily	easy	O/W	bad	none
	Barium oleate	solid	partially	no emulsion	-	-	-
Ohio Apex	Butoxyethyl stearate	liquid	completely, quickly	difficult	-	bad	none
Kessler	Butyl laurate	liquid	completely, quickly	difficult	-	bad	none
Kessler	Butyl stearate	liquid	completely, quickly	difficult	-	bad	none
	Calcium oleate	solid	partially	no emulsion	-	-	-
DuPont	Chlorinated alkyl sulfonic acid	liquid	partially	easy	-	bad	none
	Cobalt naphthenate	solid	slowly when heated	no emulsion	-	-	-
	Copper oleate	solid	slowly when heated	difficulty	-	bad	none

TABLE I (Cont'd.)

Manufacturer	Agent	State	Solubility of 1 gm. Agent in 10 gm. Perclene	Ease of Emulsification	Emulsion Type	Stability of Emulsion	Thickening Action
Vanderbilt	Darvan No. 1	solid	insoluble	-	-	-	-
"	Darvan No. 2	"	"	-	-	-	-
Dewey & Almy	Daxad No. 23	"	"	-	-	-	-
Glyco	Diethyleneglycoldicaricinoleate	liquid	complete	fair	W/O	fair	good
"	Diethyleneglycol monoethyl ether laurate	paste	completely	difficult	-	bad	none
"	Diethyleneglycol monoethyl ether stearate	solid	completely	difficult	-	bad	none
"	Diglycol laurate	liquid	completely	fair	W/O	fair	some
"	Diglycol ricinoleate		completely, cloudy	difficult	W/O	bad	-
"	Diglycol stearate	solid	partially	fair	W/O	bad	slight
Gen.Dyestuff	Emulphor A	liquid	completely	easy	O/W	fair	slight
" "	Emulphor AG oil soluble	"	completely, cloudy	"	O/W	bad	slight
" "	Emulphor ELA	"	" "	"	O/W	-	none
" "	Emulphor ON	solid	" "	"	O/W	fair	slight
Glyco	Ethyleneglycol monoethyl ether laurate	liquid	completely	difficult	-	bad	none
	Gelatin	solid	insoluble	none	-	-	-
Glyco	Glyceryl oleyl triricinoleate	liquid	complete	fair	W/O	-	good
Kessler	Glyceryl monolaurate	solid	partially	"	W/O	good	"
Glyco	Glyceryl monoricinoleate	"	completely, turbid	easy	W/O	"	"
Kessler	Glyceryl monostearate	"	partial	fair	W/O	"	"
Glyco	Glyceryl trilevulinyl triricinoleate	liquid	complete	difficult	W/O	poor	-

TABLE I (Cont'd.)

Manufacturer	Agent	State	Solubility of 1 gm. Agent in 10 gm. Perclene	Ease of Emulsification	Emulsion Type	Stability of Emulsion	Thickening Action
Eimer & Amend	Gum tragacanth	solid	insoluble	no emulsion	-	-	-
	Hercose C High	"	"	" "	-	-	-
Horn	Hornkem No. 3	liquid	"	" "	-	-	-
Rohm & Haas	Hydrocide 10-X	"	none or slight	easy	O/W	bad	none
Gen.Dyestuff	Igepal CA	"	" " "	"	"	-	"
" "	Igepon T gel	gel	partial	"	"	bad	"
	Imerial TX	paste	"	"	"	"	slight
Mallinkrodt	Lanolin	"	complete but slow	fair	W/O	"	none
	Lead oleate	"	" if heated	no emulsion	-	-	-
	" stearate	solid	" " "	" "	-	-	-
	" resinate	"	partial	fair	W/O	good	some
	" ricinoleate	paste	complete, if heated	no emulsion	-	-	-
	Magnesium oleate	solid	complete, if heated	no emulsion	-	-	-
	Manganese naphthenate	"	complete	" "	-	-	-
Atlas	Mannitan diricinoleate	liquid	complete	easy	W/O	good	good
Atlas	Mannitan monolaurate	liquid	complete	easy	W/O	good	good
Atlas	Mannitan monooleate	liquid	complete	easy	W/O	good	good
Atlas	Mannitan tetraricinoleate	"	complete	easy	W/O	good	good
Atlas	Mannitan triricinoleate	"	complete	easy	W/O	good	good
Atlas	Modified sorbitan monooleate	liquid	complete, turbid	easy	O/W	fair	none

TABLE 1 (Cont'd)

Manufacturer	Agent	State	Solubility of 1 gm. Agent in 10 gm. Perclene	Ease of Emulsification	Emulsion Type	Stability of Emulsion	Thickening Action
Allied	Nacconol EP	solid	partial	easy	O/W	bad	none
Allied	" NR	"	"	"	"	"	"
Glyco	Monoethylene-glycol diricinoleate	liquid	complete	"	"	-	"
Wolf	Orotol N	solid	partial	"	"	-	"
Proctor & Gamble	Orvus	paste	insoluble	"	"	bad	"
Parke, Davis	Phemerol	solid	partial	"	"	-	"
A.D. Mackay	Potassium laurate	paste	"	"	-	bad	"
DuPont	" oleate	paste	"	"	-	"	"
	" salts of linseed oil acids	solution	"	"	-	"	"
Beacon	Potassium stearate	paste	"	"	W/O	"	"
	Ramapol WF	liquid	complete	difficult	"	"	some
Gen. Dyestuff	Ramasol NA	solid	insoluble	no emulsion	-	-	-
	Rosin	"	partial	difficult	-	poor	-
Monsanto	Santomerse #3	"	complete	easy	O/W	"	slight
"	Santomerse S	liquid	none to slight	"	-	"	"
Sherwood Ref'g. Co.	Sherosope L	"	complete	difficult	-	"	"
" "	Sherosope N	"	"	"	-	"	"
"	Sherosope T	"	"	"	-	"	"
Ciba Co.	Solvadine NC conc.	"	none to slight	easy	O/W	"	none
Atlas	Sorbide dipalmitate	solid	complete	fair	W/O	fair	some
Atlas	Sorbide monolaurate	liquid	"	easy	"	good	good
Atlas	Sorbide monostearate	solid	"	"	"	"	"
Atlas	Sorbitan monolaurate (Span 20)	liquid	"	"	"	"	"

TABLE I (Cont'd)

Manufacturer	Agent	State	Solubility of 1 gm. Agent in 10 gm. Perclene	Ease of Emulsification	Emulsion Type	Stability of Emulsion	Thickening Action
Atlas	Sorbitan mono-oleate (Span 80)	liquid	complete	easy	W/O	good	good
Atlas	Sorbitan mono-palmitate (Span 40)	solid	"	"	"	"	"
Atlas	Sorbitan mono-ricinoleate	liquid	"	"	"	"	"
Atlas	Sorbitan mono-stearate (Span 60)	solid	"	"	"	"	"
Atlas	Sorbitan tri-laurate	liquid	"	difficult	"	"	slight
Atlas	Sorbitan trioleate	"	"	fair	"	fair	some
Atlas	" tetralaurate	"	"	difficult	-	poor	-
Glyco	Sorbitol laurate	"	"	easy	W/O	fair	good
Glyco	Soya lecithin	"	"	difficult	-	poor	slight
Glyco	Tetraethyleneglycol monostearate	solid	"	easy	O/W	-	none
Glyco	Tetraethyleneglycol distearate	"	"	fair	W/O	poor	slight
Rohm & Haas	Triton B 1956	liquid	"	easy	W/O	good	good
Rohm & Haas	" B-2994	solid	"	"	O/W	fair	none
Rohm & Haas	" K-60	paste	partially	difficult		poor	"
Rohm & Haas	" NE	liquid	complete, turbid	easy	O/W	-	none
Eimer & Amend	Turkey red oil	"	insoluble	"	"	-	"
Atlas	Tween 60	"	partial	"	"	-	"
Gen.Dyestuff	Vultramine	solid	insoluble	no emulsion	-	-	-
	Zinc oleate	"	partial	difficult	-	-	-

TABLE II

Classification of Emulsifying Agents

These agents were arbitrarily divided on the basis of ease of emulsification of water in Perclene by hand stirring, and their stability in the presence of S-461.

Most Promising	Intermediate
Arlacel A	Diglycol laurate
Arlacel B	Glyceryl monoricinoleate
Mannitan diricinoleate	Mannitan monooleate
Mannitan monolaurate	Mannitan tetraricinoleate
Mannitan triricinoleate	Sorbide monolaurate
Sorbitan monooleate (Span 80)	Sorbide monostearate
Sorbitan monopalmitate (Span 40)	Sorbitan monolaurate (Span 20)
Sorbitan monostearate (Span 60)	Sorbitan monoricinoleate
	Sorbitol laurate
	Triton B1956

Least Promising
Glyceryl monolaurate
Glyceryl monostearate
Glyceryl oleyl triricinoleate
Lead resinate
Sorbide dipalmitate
Sorbitan trioleate

TABLE III

Decontamination of H in Deck Paint

The ratios 20/30, 20/50 and 20/80 refer to the Perclene/water ratio. Tests were repeated 24 hours later if positive one hour after decontamination.

Emulsifying Agent	Paper Test Times (Minutes) 20/30 S-461 1 Hr.	20/30 S-461 24 Hrs.	20/30 RH-195 1 Hr.	20/30 RH-195 24 Hrs.	20/30 S-210 1 Hr.	20/30 S-210 24 Hrs.	20/50 S-461 1 Hr.	20/50 S-461 24 Hrs.	20/80 S-461 1 Hr.	20/80 S-461 24 Hrs.
Arlacel A	neg.		neg.		24 neg.	neg.	6 neg.	neg.	-	-
Arlacel C	neg.		neg.		neg.		neg.		neg.	
Mannitan Diricin-oleate	neg.		2 1	neg. neg.	neg.		12 neg.	neg.	-	-
Mannitan Monolaurate	neg.		neg.		neg.		neg.		-	-
Mannitan Triricin-oleate	15 1	neg. neg.	2 1	neg. neg.	neg.		1 5	neg. neg.	-	-
Span 80	neg.		22 37	neg. neg.	19 neg	neg.	1 neg.	neg.	neg.	
Span 40	neg.		neg.		24 3	neg. neg.	neg.		neg.	
Span 60	neg.		neg.		2	neg.	neg.		neg.	
Typical Blank	< 1 < 1	6 5								

TABLE IV

Decontamination of H in Deck Paint with 20/80 Emulsion Pastes of S-210

Emulsifying Agent	% of Weight of Perclene	Paper Test Times 1 Hr.	Paper Test Times 24 Hrs.
Span 40	1%	neg.	
	5%	2	neg.
		2	neg.
	10%	2	neg.
		5	neg.
Span 80	1%	2	neg.
		3	neg.
	10%	2	neg.
		neg.	

TABLE V

Decontamination of H in Deck Paint

Perclene/Water Ratio	Percentage of Chloroamide	Paper Test Times: With Span 20 Immed.	With Span 20 24 Hrs.	With Span 80 Immed.	With Span 80 24 Hrs.
20/50	5% S-330	18	neg.	neg.	
		10	neg.	neg.	
20/50	10% S-330	1	6	neg.	
		1	9	neg.	
20/50	5% S-222	1	2	1	30
		1	2	1	9
20/50	10% S-222	1	3	1	3
		1	neg.	1	3
20/50	10% S-436	1	9	1	11
		1	8	1	neg.
20/30	10% S-330	neg.		neg.	
		6	neg.	neg.	
20/30	10% S-222	1	neg.	3	neg.
		1	12	3	neg.

TABLE VI

Decontamination of H in Deck Paint

Emulsion Paste	Application Time (Min.)	Test Time: Immed.	Test Time: 3 Hrs.	Test Time: 24 Hrs.
Span 20/S-210	20	1 1	5 2	neg. 15
Span 20/S-210	40	9 30	neg. neg.	
Span 20/S-210	60	neg. neg.		
Span 80/S-210	20	1 1	5 10	neg. neg.
Span 80/S-210	40	neg. neg.		
Span 80/S-210	60	neg. neg.		

TABLE VII

Decontamination of H on Deck Paint

Emulsion Paste	Paper Test Time (Min.): Immed.	Paper Test Time (Min.): 24 Hrs.
* HCB/Span 20/S-461	<1 <1	2 4
HCB/Span 20/RH-195	<1 <1	1 1
HCB/Span 20/S-436	1 3	8 neg.
* HCP/Span 20/S-436	3 5	21 30
* CP/Span 20/RH-195	<1 <1	
CP/Span 20/S-461	<1 <1	
CP/Span 20/S-436	<1 <1	
* BL/Span 20/S-461	<1 <1	

*HCB = Hexachlorobutadiene
HCP = Hexachloropropene
CP = Chloropropane
BL = Butyl laurate

TABLE VIII

Solubility of Chloroamides in Hexachlorobutadiene and Hexachloropropane

Solvent	Chloroamide	Solubility GM./100 cc. Sol'n.
Hexachlorobutadiene	S-461	0.015
"	RH-195	0.82
"	S-436	4.85
Hexachloropropene	S-461	0.03
"	RH-195	1.00
"	S-436	5.40

TABLE IX

Decontamination of H in Wet Deck Paint Panels

The pastes were prepared with S-461, Perclene, Water and the emulsifying agents listed.

Emulsifying Agent	Paper Test Times (Minutes) Wet with Fresh Water	Wet with Sea Water
Arlacel C	neg.	neg.
Span 80	neg.	neg.
Span 40	neg.	neg.
Span 60	neg.	neg.
Mannitan monolaurate	-	neg.

TABLE X

Decontamination of H in M485 Airplane Lacquer

Emulsifying Agent	Application Time (Min.)	Paper Test Time (Minutes) S-210 Immed.	S-210 2 Hrs.	S-461 Immed.	S-461 2 Hrs.
Span 20	20	2	28	1	3
		4	42	1	1
Span 80	20	6	neg.	1	4
		4	42	2	neg.
Span 20	60	2	8	neg.	
		6	neg.	neg.	
Span 80	60	2			
		neg.			

TABLE XI

Decontamination of H in Doped Fabrics

Emulsion Paste	Application Time (Hrs.)	CN *Fabric Immed.	CAB *Fabric Immed.	CAB *Fabric 24 Hrs.
Span 20/S-210	1	5	1	4
	2	-	1	9
Span 20/S-461	1	neg.	1	4
	2	-	1	neg.
Span 80/S-210	1	neg.	2	5
	2	-	1	6
Span 20/S-461	1	neg.	1	5
	2	-	1	29

*CN = cellulose nitrate doped
*CAB= cellulose acetate butyrate doped

TABLE XII

Tensile Strength Measurement of Doped Fabrics after 3 Successive Treatments and 2 Weeks' Outdoor Weathering

Treatment	Tensile Strength (Lbs.) Cellulose Nitrate	Cellulose Acetobutyrate
1. Blank	113	127
2. Span 20/S-210 Emulsion	107	120
3. Span 20/S-461 Emulsion	115	131
4. Span 80/S-210 Emulsion	113	117
5. Span 80/S-461 Emulsion	106	120
6. H + (2)	116	130
7. H + (3)	113	129
8. H + (4)	110	129
9. H + (5)	114	125

TABLE XIII

Decontamination of HV and L on Deck Paint with S-461 20/30 Perclene/water Pastes

Emulsifying Agent	Paper Test Time (Min.) HV Immed.	HV 24 Hrs.	L Immed.
Arlacel C	23 15	neg. neg.	neg.
Mannitan monolaurate	neg.		neg.
Span 80	neg.		neg.
Span 40	neg.		neg.
Span 60	neg.		neg.

TABLE XIV

Decontamination of HN-3

Decontamination System	Paper Test Times (Min.) Immed.	24 Hrs.	48 Hrs.
Span 80/S-461 emulsion paste	10		24
	10		27
Span 80/S-210 emulsion paste	10		21
	10		21
Spray twice, TCE/RH-195	10		24
	10		27
Spray twice, TCE/S-436	26	neg.	
	26	neg.	
Blank	3	10	20
	3	10	19

TABLE XV

Decontamination of HN-1

Decontamination System	Paper Test Times (Min.) Immed.	3 Hrs.	48 Hrs.
Span 80/S-461 emulsion paste	3	6	16
	4	6	16
Span 80/S-210 emulsion paste	5	6	16
	5	6	17
Span 20/S-436 emulsion paste	neg.		
	neg.		
Span 80 emulsion, no chloroamide	4	5	16
	5	6	20
Spray twice, TCE/RH-195	4	4	11
	3	4	9
Spray twice, TCE/S-436	5	23	neg.
	6	23	neg.
Blank	2	1	5
	2	1	5

TABLE XVI

Storage of Perclene/Span Solutions

Perclene Solutions	% Span	Separation on Storage (4 Wks.) R.T.	5°C	45°C
Span 20	10	None	V. slight	None
Span 20	5	None	None	None
Span 40	5	V.slight*	Considerable	V. slight
Span 60	5	V.slight	Considerable	V. slight
Span 80	5	V.slight	V. slight	V. slight

*V. slight separation appears to be due to insoluble impurities such as sorbitol.

TABLE XVII

Stability Of Emulsion Pastes on Storage

Storage Temp.	Emulsifying Agent	Chloroamide	% Cl+ Orig.	1 Wk.	2 Wks.	4 Wks.	8 Wks.
R.T.	Span 20	S-461	2.80	2.79	2.85	2.83	2.82
	" 20	S-210	1.85	1.86	1.87	1.90	1.87
	" 20	RH-195	3.01	3.03	2.98	2.96	2.62
	Span 80	S-461	2.56	2.62	2.62	2.54	2.41
	" 80	S-210	1.78	1.78	1.79	1.78	1.80
	" 80	RH-195	1.85	1.82	1.79	1.74	1.57
45°C	Span 20	S-461	2.80	2.74	2.62	1.91	0.28
	" 20	S-210	1.85	1.91	1.94	-	1.67
	" 20	RH-195	3.01	2.30	1.49	-	-
	Span 80	S-461	2.56	2.44	2.41	2.31	1.53
	" 80	S-210	1.78	1.77	1.78	-	1.62
	" 80	RH-195	1.85	1.34	0.87	-	-

TABLE XVIII

Storage of S-461/Span 80/Porclene Mixtures

Storage Time	% Cl+ R.T.	% Cl+ 45°C	% Cl+ 60°C
Original	16.52	16.37	16.30
3 days	15.62	15.20	14.80
7 days	16.10	15.58	14.34
22 days	16.14	15.00	13.43

TABLE XIX

Tests of Oil Soluble Dyes in Emulsion Pastes

Manufacturer	Dyestuff	Solubility in Perclene	Color in Perclene	Color of Paste on Panel, Wet	Color of Paste on Panel, Dry
National Aniline	Oil Black 38226 PDR	soluble	Bluish-black	Chocolate	Chocolate
" "	AZP Oil Blue Black B	Soluble	Red-black	Dark purple	Dark blue-purple
" "	Nubian Resin Black	Sl.soluble	Brown-purple	Pink	Cream
" "	Oil Black 5115 PDR	Soluble	Purple-black	Brown	Dark brown
" "	Alizarine Blue	Soluble	Dark blue	Light blue	Light blue
" "	Spirit Nigrosine R	Insoluble	-	-	-
" "	Oil Black 24087	Soluble	Dark blue	Brown	Dark brown
Gen. Dyestuff	Induline Base R	Soluble	Dark red	Light brown	Light brown
" "	Bismarck Brown TSS Base, 35356	Sl.soluble	Red-brown	-	-
" "	Sudan Blue GA, 247459	Soluble	Blue black	Blue	Blue
" "	Induline Base B 24002	Sl.soluble	-	-	-
" "	Alizarine Sky Blue B, 34272	Soluble	Dark blue	Blue	Light blue
Calco	Calcofast Black Green Toner	Insoluble	-		
"	Calco Oil Black B-3804	Soluble	Gray	Brown	Light brown
"	Calco Nigrosine Base	Soluble	Red-black	Pink	Pink
"	Victoria Blue B Base	Soluble	Red-black	Dark blue	Light blue
"	Calcofast Spirit Black R	Insoluble	-	-	-
"	Calco Oil Black 8603	Soluble	Purple-black	Blue-gray	Blue-gray
"	Calco Oil Blue 10641	Soluble	Dark bluegreen	Blue-green	Light blue
"	Calco Oil Blue IR Base	Soluble	Purple	Purple	Violet
"	Calco Oil Blue NA	Soluble	Blue	Dark blue	Blue
"	Calco Oil Blue Goo	Soluble	Blue green	Light blue	Light blue
"	Calcochrome Alizarine Blue AWR	Sl.soluble	-	-	-
"	Calco Oil Blue B5199	Soluble	Blue-black	Steel gray	Steel gray
"	Calco Oil Black F4160	Soluble	Blue-black	Brown	Light brown

APPENDIX B

Dedontamination Efficiency of TCE/RH-195 and Paste Systems

1. A series of decontamination experiments was made with the paste systems using 6" x 12" blue deck painted steel panels. Each panel was contaminated on 2-inch squares with H at a density of about 20 g./yd^2. The temperature during the tests was 95°F. One hour after the H was applied, the pastes were brushed on, left for one hour then removed by hosing with water and brushing if necessary. The results of the tests for residual H are given in Table I.

TABLE I

Efficiency of Decontaminating Systems (Small Panel)

Paste	Weight of Paste Used Lb./yd^2	Paper Test Time (Minutes) Immed.	3 Hrs.	24 Hrs.
E.P. S-461	1.6	neg.	-	-
" "	1.1	8	-	neg.
" "	0.7	1	-	neg.
E.P. S-210	1.7	5, neg.	neg.	-
" "	1.1	2. 2	14, 28	neg.
" "	0.8	<1	4	neg.
K Oleate/S-461	0.9	4	-	neg.
" "	0.6	2	-	neg.
" "	1.2	11	-	neg.

2. A series of experiments using several decontaminating systems were run in which 36" x 72" blue deck painted panels were used. Data were obtained for TCE/RH-195, the potassium oleate paste and emulsion pastes. The H contamination was about 20 g./yd^2 The temperature during the tests was 85 to 95°F. The tests for residual H are given in Table II.

TABLE II

Efficiency of Decontaminating Systems (Large Panels)

System Used	Application Method	Weight Used Lb/yd	Paper Test Time (Min.) Immed.	3 Hrs.
E.P. Span 80/S-210	Spray	2.7	<1(8)	2 to neg.
E.P. Span 80/S-210	Brush	0.5	<1(4)	1,<1(3)
E.P. Span 80/S-461	Brush	0.4	3,1,1,2	5,3,2,3
E.P. Span 80/S-461	Brush	1.25	2,4,6,neg.	-
K. Oleate/S-461	Spray	1.2	15,20,neg.(2)	-
TCE/RH-195	Spray	2.7	5, neg.(3)	-
TCE/RH-195	Spray	0.7	9,30,20,neg.	30,neg(3)
TCE/RH-195	Spray	0.7	7,26,neg.(2)	-
TCE/RH-195	Spray	0.8	10,15,20,neg.	-

Note: Figures in parentheses indicate the number of tests which gave the same paper test readings.

3. Ingredients for the improved potassium oleate paste were obtained from the NDRC group at the DuPont Experimental Station. The potassium oleate suspension had the following composition by weight:

Tetrachloroethylene (Perclene)	67.3 parts
Potassium Oleate (28% water)	18.0 parts
Barium hydroxide octahydrate	2.75 parts
Aristowax 160/165	1.6 parts

To prepare the paste, 11.5 parts of S-210 were mixed with 100 parts of the above dispersion. Decontamination tests for H using the paste were made on 6" x 12" deck painted steel panels. The H contamination was approximately 20 g./yd^2. The tests for residual H are given in Table III.

TABLE III

Decontamination of H with Improved Potassium Oleate Paste

Amount Used Lb/yd^2	Paper Test Time (Minutes)		
	Immed.	3 Hrs.	24 Hrs.
0.8	4, 4	40, neg.	neg.
0.9	10, neg.	neg.	
0.9	neg.		
1.0	neg.		
1.1	neg.		
1.5	neg.		

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