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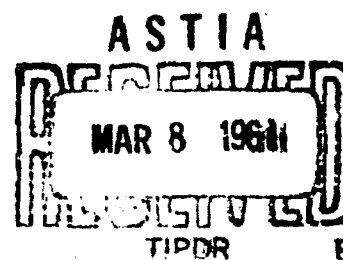
SURFACE CHEMICAL METHODS OF DISPLACING WATER AND/OR OILS AND SALVAGING FLOODED EQUIPMENT

PART 1 - PRACTICAL APPLICATIONS

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ABSTRACT

The effectiveness of surface-active substances in displacing Navy Special fuel oil and water from solid surfaces has been demonstrated. A general mechanism is outlined for the rapid displacement of one liquid by another as a result of unbalanced surface forces. Two formulations have been developed, one for the displacement of fuel oil and the other for the removal of water from electrical, electronic, or mechanical equipment. Both formulations are relatively harmless to most electrical and electronic insulation. The fuel-oil-cleaning emulsion retards rusting of the ferrous metals with which it comes in contact, while the water-displacing composition leaves a persistent and effective rust inhibiting film over the surfaces of the equipment.

Electrical and electronic components which had been submerged in a mixture of Navy Special fuel oil and sea water were effectively salvaged by treatment with the fuel-oil-cleaning emulsion and the water-displacing composition. An integrated procedure is outlined for the recovery of electrical, electronic, or mechanical equipment after such submersion. A method of sealing the insulated parts of electromotive equipment against the penetration of flooding water is also presented.

PROBLEM STATUS

This is an interim report; work on this problem is continuing.

AUTHORIZATION

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SURFACE CHEMICAL METHODS OF DISPLACING WATER AND/OR OILS AND SALVAGING FLOODED EQUIPMENT

PART I - PRACTICAL APPLICATIONS

INTRODUCTION

As a result of equipment damage in floods caused by hurricanes, tornados, heavy rains, or rapid thaws, millions of dollars of flood damage are done annually to homes, factories, and equipment of all kinds. The resulting immersion in water is particularly serious for electrical equipment, power sources, and complex machinery. Similar damage results from the accidental flooding of basements and ship compartments or from the materials used in fighting a fire. Insurance company figures show that water damage losses from accidental and fire-fighting flooding now run into many millions of dollars annually.

Damage caused by flooding can be greatly reduced by prompt application of adequate salvage procedures. The damage could be still further reduced if equipment in hazardous locations were properly conditioned before flooding conditions occurred. The procedures described in this report are the result of a 10-year investigation of methods for salvaging naval equipment from flooded ship compartments. However, the fundamentals of cleaning and water displacement involved apply to many types of equipment in any location.

Some of the results of our investigations on water-displacing fluids have been presented in earlier reports (1-3). Other investigators have also been active. Galvin and McAuley have patented a water-displacing composition in which the active components are dispersed in a hydrocarbon solvent (4). Snell and associates have described the use of emulsion-type diphase cleaners for simultaneous removal of water-soluble and hydrocarbon-soluble soil by solution or solubilization mechanisms (5-11). Some uses of water-displacing fluids have been reviewed by D. L. Samuel (12,13). The materials and procedures reported here differ in mode of action and in range of applicability from those of the references.

Success in preventing corrosion in flooded equipment depends upon (a) complete removal of all sea water salts (or corrosion-promoting chemicals often present in flooding waters), (b) removal of all water, and (c) application of a rust preventive, all these steps occurring soon after removal of the equipment from the water. However, flooding of equipment is often accompanied by contamination with oil and grease resulting from spillage, flooded gear boxes, crankcases, or fuel-oil tanks, or from breaks in oil lines. In such cases the viscous, adherent oil residues resist removal by water and also prevent effective action of the water-displacing compositions which have been developed. Similar difficulties are encountered with the tar-like residues deposited by the smoke of a severe fire.

This report is concerned with the development (a) of a cleaning emulsion for rapidly removing oily contaminations from mechanical or electronic equipment and (b) of a water-displacing composition that will rapidly displace water from all surfaces, parts, and crevices accessible to a liquid spray and thus accelerate the drying process. It outlines a successful integrated salvaging procedure and indicates suitable equipment for field

applications to flooded and/or oil-fouled electrical, electronic, or mechanical equipment. It also presents a method of preconditioning existing electrical equipment to render it more capable of salvage should it be flooded.

The liquid displacement with which this report is concerned involves surface chemical phenomena which have not been fully appreciated before. The surface chemistry of the process, which provides clear guidance for the selection of displacing agents for a wide variety of liquids besides water, will be presented in a separate report (Part 2).

THE FORMULATION OF WATER- AND OIL-DISPLACING COMPOSITIONS

Water-Displacing Compositions

A practical water-displacing composition must displace water efficiently and resist rewetting, must do no significant damage to electrical insulation, and must protect salvaged metal surfaces from rusting. It is also desirable that it should offer low fire and health hazards, be stable in storage, and be low enough in cost to justify widespread stocking and use. The three essential components of such compositions are the displacing liquid, the surface active rust inhibitor, and a suitable oxidation inhibitor; these must be chosen in the light of the above requirements. The effectiveness of the water-displacing liquid was assessed by a water displacement test whose results are tabulated in Table 1. In this test a 4 by 4 inch stainless steel coupon was supported horizontally so that the upper surface of the plate could be adjusted to either 1 or 2 mm below the surface of the water in a waxed hydrophil tray. Waxed glass barriers were used to scrape the water surface clean, then 0.025 ml of the organic liquid was placed gently on the surface of the water using a blood pipette. If the liquid was effective in displacing water, a hole formed in the water, revealing the bare surface of the steel plate. The maximum diameter of the hole formed was measured and recorded as a criterion of the water-displacing ability of the fluid under study.

The list of effective water-displacers includes alcohols from C_3 to C_6 , methyl amyl ketone, acetylacetone, amyl acetate, cellosolve acetate, ethyl aceto acetate, butyl or propyl lactate, possibly ethyl or methyl carbonate, and the cellosolves (2,3). Higher cellosolve derivatives, carbitols, and a few other compounds which gave good water displacement were omitted from consideration here because their evaporation rates were so low as to create a problem of removal from the cleaned equipment. Lower molecular weight members of the alcohol, ester, and ketone series, while active in displacement, were not considered because their volatility created fire or toxicity hazards or because the displacement effect was too transitory. Some otherwise promising liquids were ruled out because they caused excessive softening or disintegration of common types of electrical insulation. In the light of the requirements outlined in the preceding paragraph, 1-butanol was selected for use in a practical formulation and has been a component of the compositions used in field tests. On the basis of their less obnoxious odor, the pentanols and hexanols may be preferable to the butanols for some applications.

The rust inhibitor in a water-displacing composition plays a dual role; it must act during the water-displacing action to deposit a hydrophobic film which the displaced water will not rewet, and it must protect the salvaged equipment from subsequent rusting during service or storage. In addition, it must be compatible with the other components of the formulation and satisfy the general requirements outlined previously.

The mechanism of action and the characteristics of polar-nonpolar rust inhibiting compounds have been discussed in earlier reports of this Laboratory (14-16). Compounds tested in experimental water-displacing fluids (2) included naphthenic and arylstearic acids and their alkali, alkaline earth, and amine salts, the corresponding salts of petroleum and synthetic sulfonic acids, acid phosphate esters, and polyesters such as the glycerol oleates. Rust inhibiting properties were compared by using the fog-cabinet corrosion test (17). Of the compounds examined, basic barium dinonylnaphthalene sulfonate was chosen as the most suitable for use in water-displacing fluids because, in addition to being soluble in 1-butanol, this basic salt is compatible with the oxidation inhibitor chosen, 2,6-di-tertiarybutyl, 4-methylphenol, imparts excellent rust inhibition, and is commercially available as a reproducible chemical. Because of its alkaline reaction it has a basic reserve which is essential for corrosion inhibitors which must operate in atmospheres where minute quantities of acid vapors may be encountered or where personnel handling the equipment may leave corrosive finger prints.

Water-displacing compositions with good stability against oxidation and deterioration during storage are desirable because military use often requires long distance shipments and extended storage times, frequently under adverse conditions. Oxidation during storage may cause the formation of objectionable acids, gums, and precipitates. As in the storage of gasoline and oils, the rate of oxidation accelerates rapidly with increasing temperature, so that tropical storage is a serious problem.

The oxidation stabilities of several water-displacing fluids and compositions were examined using the induction-period test for the oxidation stability of gasoline (18). The usual 50-ml sample was held at 100 psi and 100°C and the length of the induction period was observed by means of a pressure recorder. The fact that 1-butanol has an induction period of 8 hours is indicative of fair storage stability. However, 1-butanol containing 0.1 wt-% of 2,6-di-tertiarybutyl, 4-methylphenol (PX441) or the same concentration of 2,4-dimethyl, 6-tertiarybutylphenol (24M6B) gave induction periods greater than 216 hours. The addition of 3.0 wt-% of glyceryl mono- and dioleate or the same concentration of basic barium dinonylnaphthalene sulfonate did not appreciably alter the induction period. Formulations containing 1-butanol, PX441, and either glyceryl mono- and dioleate or basic barium dinonylnaphthalene sulfonate as rust inhibitors have shown no deterioration in water-displacing ability after storage periods of 10 and 6 years respectively.

Considerations of cost make the possible use of a nonpolar diluent interesting. A study was made of the effect of several such diluents on the equilibrium spreading pressure and on the practical water-displacing efficiency of 1-butanol. The results are summarized in Fig. 1. It can be seen from the figure that the incorporation of Stoddard solvent (104°F-flash-point naphtha) in 1-butanol has only minor effects on the equilibrium spreading pressure until the proportion of hydrocarbon in the mixture exceeds 75 wt-%. This is because the surface tension of the naphtha and the butanol are almost the same and because the latter is so strongly adsorbed at the oil/water interface that the interfacial tension is also little changed as long as the alcohol remains a major constituent of the mixture.

The effect of the same nonpolar diluent on the water-displacing effect, on the other hand, is marked. The addition of as little as 20 wt-% causes a 40% reduction in diameter of the displacement effect. Compositions containing between 20 and 80 wt-% diluent have equivalent displacing powers, but further reductions in alcohol content give liquids of negligible displacing ability.

Table 1
Water-Displacing Ability and Related Properties of Various Organic Fluids

Fluid	Specific Gravity (20/20°C)	Solubility of Water in Fluid (wt-% at 20°C)	F_e (dynes/cm at 20°C)*	Maximum Diameter of Area of Displaced Water Layer (in.)†	
				One-mm-Thick Layer	Two-mm-Thick Layer
Alcohols					
Methanol	0.7924 (a)	Complete	-	0.75	Does not penetrate
Ethanol	0.7905 (a)	Complete	-	0.75	Does not penetrate
1-Propanol	0.8044 20/4	Complete	-	1.00	Does not penetrate
2-Propanol	0.7854 20/4	Complete	-	1.00	Does not penetrate
1-Butanol	0.8109 (a)	20.1 (a)	48.5	2.00	1.50, recovers immediately
2-Butanol	0.808 20/4	56.0	50.4	1.75	1.50, recovers immediately
2-Methyl-1-propanol	0.8169	-	49.3	1.75	1.50, recovers immediately
1-Pentanol	0.82 (d)	-	39.5	1.75	0.50, recovers immediately
2-Methyl-1-butanol	0.816 (d)	-	40.1	1.75	0.50, recovers immediately
3-Methyl-1-butanol	0.81 (d)	-	41.6	1.75	0.50, recovers immediately
2-Methyl-2-butanol	0.81 (d)	18 (d)	43.6	2.50	2.00, recovers in 15 seconds
3-Pentanol	0.815	-	40.8	2.00	1.00, recovers immediately
1-Hexanol	0.8208 (a)	7.26 (a)	36.9	1.75	Does not penetrate
2-Ethyl-1-butanol	0.8328 (a)	4.56 (a)	39.1	2.00	Does not penetrate
2-Methyl-1-pentanol	0.831	5.8	39.2	1.75	Does not penetrate
1-Heptanol	0.8210	-	37.3	1.50	Does not penetrate
2-Heptanol	0.8187 (a)	5.07 (a)	36.3	1.50	Does not penetrate
2,4-Dimethyl-3-pentanol	0.829	-	38.8	1.75	Does not penetrate
1-Octanol	0.8246	-	35.5	1.00	Does not penetrate
2-Ethyl-1-hexanol	0.8304 (a)	2.57 (a)	35.0	2.00	Does not penetrate
1-Nonanol	0.8274	-	35.5	0.75	Does not penetrate
2-Methyl-7-ethyl-4-undecanol	0.8363 (a)	2.11 (a)	22.0	0.50	Does not penetrate
2-Heptyl-1-nonanol	0.8355 (a)	0.66 (a)	14.1 25°C	Floats	Floats
3,9-Diethyl-6-tridecanol	0.8475 (a)	0.46 (a)	19.6	Floats	Floats
Benzyl alcohol	1.250 15/15	-	27.4 25°C	1.25	1.00, recovers immediately
Cyclohexanol	0.9449 25/4	-	35.1	0.75	0.50, recovers in 30 seconds
Furfuryl alcohol	1.134	-	Sinks	2.50	0.50, recovers immediately
Tetrahydrofurfuryl alcohol	1.109 25/4	-	Sinks	1.25	0.50, recovers immediately
Ether Alcohols from Glycol					
Methyl cellosolve (Glycol monomethyl ether)	0.9663 (a)	Complete	41.2	1.00	Does not penetrate
Cellosolve (Glycol monoethyl ether)	0.9311 (a)	Complete	40.2	1.25	Does not penetrate
Butyl cellosolve (Glycol monobutyl ether)	0.9019 (a)	Complete	39.6	1.75	0.75, recovers immediately
2-Ethyl butyl cellosolve (Glycol 2-ethyl butylether)	0.8954	10.0	41.0	1.50	Almost penetrates
Diethyl cellosolve (Glycol diethyl ether)	0.8424	3.4	46.7	1.25	0.50, recovers immediately

* F_e , equilibrium spreading pressure, 10.025 ml fluid laid gently on the surface of the water layer.

(a) Carbide and Carbon Chemicals Corp.

(b) Hercules Powder Company.

(c) E. I. du Pont de Nemours and Co., Inc.

(d) Sharples Chemicals, Inc.

Table 1 (Continued)
Water-Displacing Ability and Related Properties of Various Organic Liquids

Fluid	Specific Gravity (20/20°C)	Solubility Water in Fluid (wt-% at 20°C)	F _e (dynes/cm at 20°C)*	Maximum Diameter of Area of Displaced Water Layer (in.)†	
				One-mm-Thick Layer	Two-mm-Thick Layer
Ether Alcohols from Diethylene Glycol					
Methyl carbitol (Diethylene glycol monomethyl ether)	1.0211 (a)	Complete	41.6	1.00	Does not penetrate
Carbitol (Diethylene glycol monoethyl ether)	0.9898 (a)	Complete	40.8	1.25	Does not penetrate
Butyl carbitol (Diethylene glycol monobutyl ether)	0.9536 (a)	Complete	40.1	1.25	Does not penetrate
Ketones					
Acetone	0.7911 (a)	Complete	-	1.50	Does not penetrate
Methyl ethyl ketone	0.805 20/4	-	46.2	1.00	Does not penetrate
Acetyl acetone	0.975	4.5	31.9	0.50	Does not penetrate
Acetonyl acetone	0.973	Complete	Dissolves	1.00	0.25, recovers immediately
Methyl-n-amyl-ketone	0.8166 (a)	1.5	35.8	2.75	0.50, recovers immediately
Diisobutyl ketone	0.8089 (a)	0.15	26.6	2.50	Does not penetrate
Diacetone alcohol (4-hydroxy-4-methyl pentanone-2)	0.941	Complete	Dissolves	1.25	0.50, recovers immediately
Esters					
Methyl lactate	1.092	Complete	Sinks	1.50	0.25, recovers immediately
Ethyl lactate	1.031	Complete	Sinks	2.00	0.50, recovers immediately
Butyl lactate	0.979	-	43.5	2.50	0.50, recovers immediately
Dimethyl carbonate	1.069	-	40.1	1.25	Does not penetrate
Diethyl carbonate	0.975	-	33.1	2.50	Does not penetrate
Diethyl oxalate	1.077	-	29.3	2.25	Does not penetrate
"Cellosolve" acetate (Ethylene glycol monoethyl ether acetate)	0.975	6.5	43.2	1.25	0.50
Miscellaneous					
n-Octane	0.7036	1	0.2	Does not penetrate	Does not penetrate
n-Dodecane	0.786 20/4	1	-0.0	Does not penetrate	Does not penetrate
Benzene	0.8794	1	9.9	Does not penetrate	Does not penetrate
Toluene	0.866 20/4	1	8.7	Does not penetrate	Does not penetrate
Varsol No. 1 (Boiling Range 155-195°C)	-	1	9.7	Does not penetrate	Does not penetrate
Solvesso No. 1 (Boiling Range 93-135°C)	0.821	1	11.9	Does not penetrate	Does not penetrate
Solvesso No. 2 (Boiling Range 135-175°C)	0.858	1	10.5	Does not penetrate	Does not penetrate
Furfural	1.160	-	Sinks	2.00	Does not penetrate
Tetrahydrofuran	0.854	-	40.5	1.50	0.25
Terpene methyl ethers (b)	0.912	-	28.3	0.50	Does not penetrate
Pentamethylene glycol (c)	0.994	-	Dissolves	0.75	Does not penetrate
Ethylhexylene glycol (a)	0.942	11.7	41.9	1.25	Does not penetrate

*F_e, equilibrium spreading pressure.
†0.025 ml fluid laid gently on the surface of the water layer.
(a) Carbide and Carbon Chemicals Corp.
(b) Hercules Powder Company.
(c) E. I. du Pont de Nemours and Co., Inc.
(d) Sharples Chemicals, Inc.

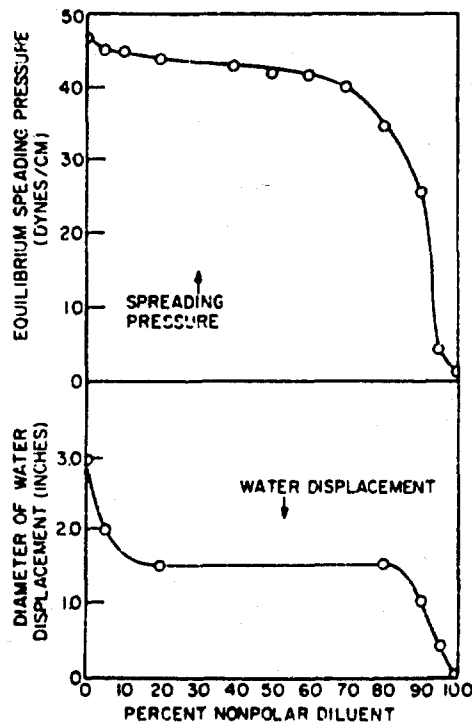


Fig. 1 - Effect of dilution with hydrocarbon (Stoddard solvent, 104 F-flash-point naphtha) on equilibrium spreading pressure and on water-displacing activity of 1-butanol

The use of a hydrocarbon of higher vapor pressure might improve the displacement efficiency, but would also increase the fire hazard. Dilution with mineral spirits is thus not a promising economy, although it might be acceptable for some specialized applications.

In the light of the technical requirements and of the foregoing experimental results, formulations falling within the following ranges have been of interest for the practical displacement of water during salvage:

- Displacing liquid: 90 to 95 wt-% 1-butanol or 1-pentanol, or mixtures of the two,
- Oxidation inhibitor: 0.1 to 0.5 wt-% 2,6-di-tertiarybutyl, 4-methylphenol,
- Rust inhibitor: 1.0 to 5.0 wt-% basic barium dinonylnaphthalene sulfonate.

Oil-Displacing Compositions

A suitable oil-removing composition must have the following characteristics:

1. It should remove oil residues rapidly and gently, with little or no mechanical treatment or brushing of the surface required.
2. It should not cause significant damage to the common insulating materials found in electrical or electronic equipment.
3. It should be no more flammable than the oil it removes.

4. It should be noncorrosive and should retard the rusting of steel surfaces to which it is applied.

5. It should be nontoxic, and nonirritating to the skin.

Preliminary experiments showed that an oil-in-water emulsion cleaner would be the most effective material meeting these requirements. The dispersed organic solvent component of the emulsion removes the hydrocarbon contaminant while the continuous water phase dissolves any water-soluble contaminants as well as flushes away the oily materials. Furthermore, the continuous water phase reduces the flammability and health hazard of the cleaner formulation and also lowers the cost of the composition. In using such a system, the cleaning emulsion and contaminant could be flushed away with sea water or fresh water, depending on availability.

It should be clearly understood that cleaning emulsions of the type discussed here are intended only to remove mobile contaminants; i.e., they are not expected to dissolve films of varnish, lacquer, and gums deposited as a result of oxidative deterioration of lubricants or fuel oils.

Two organic solvents which approximately satisfy the requirements outlined for the dispersed phase are 1,1,1-trichloroethane (methyl chloroform) and 140°F-flash-point aliphatic naphtha. In suitable formulations both solvents gave excellent removal of fuel oil. The aliphatic naphtha was selected for field testing because of its ready availability on shipboard, its moderate cost, and its lower toxicity. The other ingredients (besides water) required with the 140°F-flash-point naphtha are a suitable emulsifying agent and a minor amount of a hydrocarbon of special type which assists in oil displacement.

A surface-active agent is needed to hold the hydrocarbon and water in a useful emulsion as well as to assure the complete displacement of oil from contaminated surfaces. Oil-displacing effectiveness of specific surfactants in emulsion formulations was determined by a fuel-oil-displacement test in which a film of Navy Special fuel oil about 0.007 inch thick was applied to the surface of a clean SAE 1020 cold-rolled steel coupon placed horizontally on a leveling device. Onto this film 0.625 ml (one drop) of the cleaning emulsion containing the surfactant was dropped from a height of 1 cm. The maximum area from which the cleaning emulsion cleared away the fuel oil to expose bare metal was taken as an indication of the oil displacement ability (Fig. 2). One percent of the surfactant to be tested was dissolved in two parts by volume of Stoddard solvent and one part by volume of 1,1,1-trichloroethane, then this solution was emulsified with water in proportions of 35 vol-% of solution to 65 vol-% of water. The emulsion was prepared immediately before testing. The results of these tests are given in Table 2.



Fig. 2 - Penetration and displacement of Navy Special fuel oil from steel coupon (6 by 3 inches) by one drop of oil-displacing emulsion

Table 2
 Effectiveness of Emulsions Containing Various Surfactants in the Displacement of Navy Special Fuel Oil From a Horizontal Steel Plate (0.0625 ml of a 35-vol-%-solvent, 65-vol-%-water emulsion containing 1.0% surfactant - the solvent used is a 2-to-1 (vol) mixture of Stoddard solvent and 1,1,1-trichloroethane)

Surfactant Used		Area of Displacement, After 5 Minutes (cm ²)
Type	Trade Name	
Control, organic solvent water emulsion (without additive)		1.6
A condensate of ethylene oxide with a hydrophobic base formed by condensing propylene oxide with propylene glycol	Pluronic L-63	3.7
Polyethylene glycol 400 monooleate S1006	As in name*	3.5
A polyethylene glycol ester of lauric acid	Nonisol 100	3.3
An alkyl aryl polyether alcohol	Triton X-155	3.2
A condensate of ethylene oxide with a hydrophobic base formed by condensing propylene oxide with propylene glycol	Pluronic L-43	3.1
Nonylphenyl ether of polyethylene glycol	Tergitol nonionic NPX	2.9
Sorbitan trioleate	Span	2.9
A condensate of ethylene oxide with a hydrophobic base formed by condensing propylene oxide with propylene glycol	Pluronic L-62	2.8
A condensate of ethylene oxide with a hydrophobic base formed by condensing propylene oxide with propylene glycol	Pluronic L-42	2.8
Nonylphenoxy polyethylene ethanol	Igepal CO-630	2.5
A substituted oxazoline	Alkiterge C	2.4
Nonylphenoxy polyethylene ethanol	Igepal CO-530	2.0

*Supplied by Glycol Products Co.

Emulsions containing each of the ten most effective surfactants found by the above screening method were then compared in the emulsion cleaner spray test under conditions more closely simulating actual salvage operations. For this test an SAE 1020 steel plate 3 by 8 by 1/8 inch was uniformly coated with Navy Special fuel oil by dipping the plate in the oil, inclining it 45 degrees, and allowing it to drain for exactly 5 minutes before spraying. An oil spray gun operated with compressed air was used to spray the emulsion cleaner against the plate from a distance of two feet, using an air supply at 80 psi. This test showed that emulsions of highest stability did not cut the fuel oil film most effectively; the most complete removal of the film took place when the emulsion broke readily on contact with the surface of the fuel oil, thus releasing the solvent to attack the contaminating film. A desirable surfactant keeps the solvent mixture dispersed in an oil-in-water emulsion only until the solvent droplets touch the surface of the fuel oil film. Of the emulsions containing the surfactants listed in Table 2, polyethylene glycol 400 monooleate S1006 gave optimum results. It was not only effective with sea water as well as with fresh water but also exhibited considerably better rust inhibition than any of the other applicable surfactants.

The rust inhibiting property of these emulsions was determined by observing the corrosion on a grease-free SAE 1020 cold-rolled steel coupon after partial immersion in the emulsion cleaner for 24 hours at $100 \pm 2^\circ\text{F}$. The most effective surfactant kept the steel coupon free from rust in both the aqueous and hydrocarbon phases of the broken emulsion.

Early observations revealed that the original formulation containing only naphtha and an emulsifier was able to push back the film of fuel oil only after this film had been ruptured by strong spray impact which put the emulsion droplets in contact with bare metal. Once the molecules of surfactant reached the metal, adsorption and wetting effects caused rapid and complete displacement of the oil from the surface. Such a displacement mechanism is more efficient and more economical in the consumption of the clearing agent than a progressive solution or emulsification of the contaminant in layer-by-layer fashion. Composition based on trichloroethane did not exhibit this difficulty in penetrating the film. It was suspected that the 140°F -flash-point aliphatic naphtha was not a good enough solvent to dissolve the fuel oil instantaneously and an attempt was made to find a blending agent which would promote penetration of the film. These efforts were successful. Experiments with a variety of organic materials revealed that an effective and cheap component for this purpose was the fuel oil itself. Other compounds which promoted rapid penetration of fuel oil films were: naphthenic base turbojet oil (Spec. Mil-O 6081b, Grade 1005) and diesel fuel oil (Spec. Mil-F 16884C, Ships, Type 1). During this investigation it became evident that many kinds of oily contaminant could be removed if a penetration assistant having similar properties was utilized in the cleaning formulation.

The compositions employed to salvage electrical or electronic equipment from contaminated sea water should not materially affect insulating tapes, fabrics, plastics, or varnish under the conditions of salvage. Experience has shown that for a given solvent type the greater the volatility, the less will be the effect of solvent on electrical insulation. For the same volatility, the rate of attack will depend on the molecular structure. For use in the cleaning emulsion, aliphatic hydrocarbon solvents are less harmful to electrical insulation than the aromatic or chlorinated hydrocarbons. In the water-displacing compositions aliphatic alcohols appear to be the least harmful, with the cellosolves, acetates, lactates, and ketones following in order of increasing damage to insulation.

These conclusions are based on observations of the effects of each solvent on selected samples of insulating fabrics, tapes, plastics, and varnishes. The cleaning composition and water-displacing formulation ultimately chosen were evaluated for insulation damage in an exposure simulating the salvage operation. Samples were partially immersed in sea water covered with a layer of fuel oil for 24 hours, then removed and cleaned with the

cleaning composition, flushed with water, blown with air, and sprayed with the water-displacing formulation. On the following day, the Wilkinson Pencil Hardness Test Method (15) was used to determine the effect the soaking and recovery procedure had had on the insulating materials. The insulating materials studied are representative of those found in present day electrical and electronic equipment.

The results are reported in Table 3, which shows that all of the samples amenable to hardness testing returned to their original hardness within 24 hours after salvage. None of the insulating materials were affected by either the cleaning composition or the water-displacing composition; however, a few of the fiber reinforced materials were softened by the water or permitted fuel oil to penetrate between the layers of insulating materials so that it was necessary to use the cleaning emulsion in an ultrasonic field in order to remove the last traces of the fuel oil. The fiber-containing samples studied were strips and pieces cut from rolls of tape or cloth so that the edges were not protected with the bonding material. The rag paper was not affected after surface and edge treatment with grade CA varnish (Specification Mil-V-1137). Likewise, none of the materials which allowed penetration of the fuel oil were affected when the cut edges were coated with the same varnish and allowed to dry before being submerged in the fuel oil and sea water. In actual applications the entire assembly, including the edges of these materials, would be treated during the impregnating process, thus reducing the tendency toward fuel oil penetration.

Substitute fuel-oil displacing compositions may be prepared by employing similar quantities of either Navy Special fuel oil or turbojet engine oil in place of the diesel fuel oil. Pluronic L-63* or Nonisol 100† can be substituted for the polyethylene glycol 400 monooleate S1006‡ in the formulation. If the cleaning emulsion is to be used on mechanical equipment only, where damage to insulation is not a problem, chlorinated or aromatic hydrocarbons can be used instead of the aliphatic solvent. In this case the displacing activity is lower but the solvent action is increased. In water-displacing compositions, 1-pentanol or mixtures of butanol and pentanol may be used instead of the 1-butanol. Also 2,4-dimethyl, 6-tertiarybutyl phenol (24M6B) may be substituted for 2,6-di-tertiarybutyl, 4-methylphenol (PX441) as oxidation inhibitors. The basic salt (base No. 50) of barium, calcium, or strontium petroleum sulfonate made from a sulfonic acid having a molecular weight in excess of 460 may be substituted for the basic barium dinonylnaphthalene sulfonate as the rust inhibitor.

SALVAGE EXPERIENCE

The cleaning and water-displacing compositions whose development has been described have been utilized in simulated salvage of a wide variety of electrical and electronic units, and on the basis of this experience an integrated system of physical reconditioning or salvage has been established. Briefly, the salvage procedure involves the following steps:

1. Mobile fuel oil or grease contaminant is removed by spraying the equipment with cleaning emulsion, or by subjecting the equipment to ultrasonic radiation while immersed in the emulsion. The latter procedure is preferred for complex electronic assemblies or for components bearing adherent contamination or corrosion products.
2. The equipment is thoroughly flushed with fresh water to remove cleaning emulsion and traces of salt, using spray application or ultrasonic treatment as may be appropriate.

*Supplied by Wyandotte Chemical Corporation.

†Supplied by Geigy Industrial Chemicals.

‡Supplied by Glyco Products Company.

Table 3
 Effect of the Ultimately Chosen Cleaning Composition
 and Water-Displacing Fluid on Electrical and Electronic
 Equipment Insulation After Partial Immersion in Sea
 Water Covered with Fuel Oil

Material	Mil. Spec.	Detrimental Effect
Electrical insulating board - laminated phenolic, type PBG	Mil-P-3115	None
Electrical insulating board - laminated melamine, type GMG	Mil-P-15037	None
Electrical insulating fiber board	Mil-F-1148	None
Mica mat, silicone bonded	-	None
Mylar film	-	None
Mica mat, glass cloth silicone bonded	-	None
Varnished cambric cloth	Mil-I-3374	None
Organic varnished glass cloth	Mil-I-3190	None
Mylar-glass cloth-silicone bonded	-	None
Mylar-glass cloth-organic varnish bonded	Mil-I-3190	None
Mica-glass cloth-epoxy bonded	-	None
Fish paper F	Mil-I-695	None
Vinyl impregnated sleeving	-	None
Laminated phenolic insulating sheet, type PBG	Mil-P-3115	None
Glass tape, high temp. class B, type MG	Mil-I-3505	*
Glass tape, high temp. class H, type GMG	Mil-I-3505	*
Mylar film on rag paper	-	*

*Fuel oil penetrated between the layers of insulating material. It was necessary to use the cleaning composition in an ultrasonic transducer cleaning apparatus in order to remove the last traces of the fuel oil - no detrimental effect suffered.

3. Bulk water is blown from the equipment with oil-free compressed air and all parts of the equipment are sprayed with the water-displacing composition, which causes most of the film-forming water to drain away.

4. The remaining water and water-alcohol azeotrope are then evaporated by blowing with warm air or by placing in a moderately warm oven for several hours.

The detailed salvage procedures are set forth in Appendix A of this report. These procedures have been successfully applied to equipment which had been exposed to damage as indicated below.

1. AC motors to 5 horsepower, ac-dc motor-generators, and dc motors and controllers to 1-1/2 horsepower were repeatedly immersed in sea water covered by fuel oil, soaked in sea water for 72 hours, and then returned to their initial operating characteristics by the salvage procedure in Appendix A (Figs. 3 and 4). After salvage two different 3/4-horsepower dc motors were operated under full load for 300 hours without difficulties.

2. A radio transmitter (Type COL-52245; Figs. 5 and 6), receiver (Type CKP-46159A), and a rectifier power unit (Type COL-20218) were similarly exposed and recovered, using ultrasonic radiation to promote the action of the emulsion cleaner and the fresh water rinse. After the replacement of two plastic-base radio tubes which had trapped sea water, both transmitter and receiver operated satisfactorily, and passed a 30-day continuous operation test with no signs of difficulty. Corrosion damage was negligible.

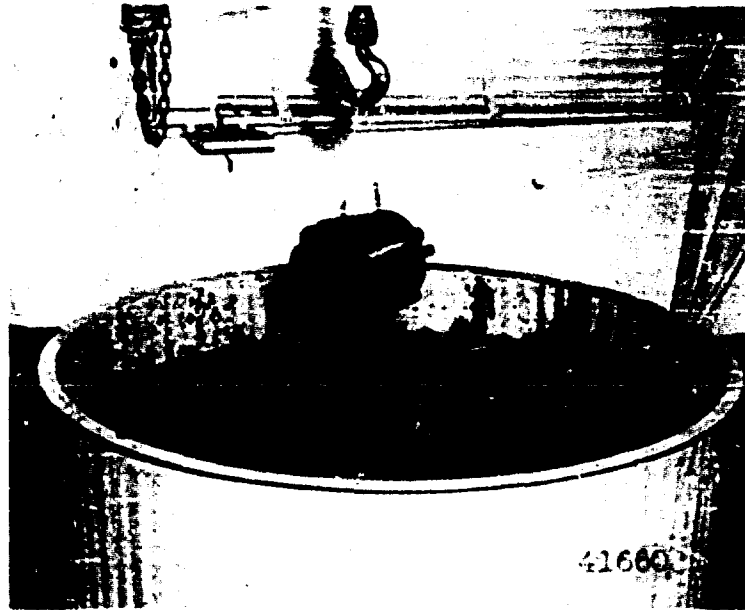


Fig. 3 - Electric motor immersion in sea water covered with Nay special fuel oil

Fig. 4 - Electric motor being cleaned after submersion in sea water covered with Navy Special fuel oil

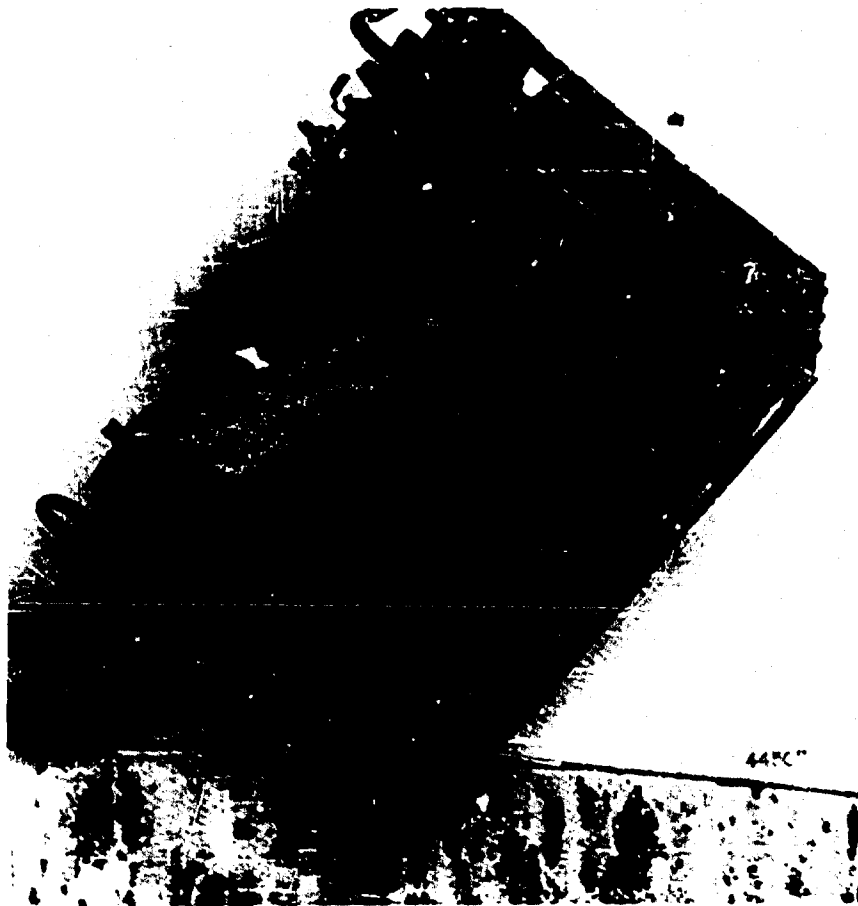
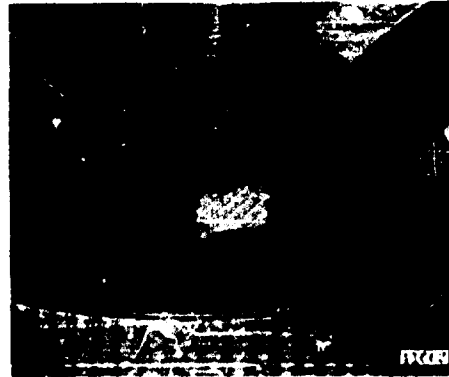


Fig. 5 - Radio transmitter after immersion in sea water covered with Navy Special fuel oil



Fig. 6 - Radio transmitter shown in Fig. 5 being cleaned after submersion in sea water covered with Navy Special fuel oil

3. A large airborne radar unit which had been subjected to a 50-hour salt water spray test in the course of acceptance testing was cleaned and reconditioned by the salvage procedure with ultrasonic treatment. More than 99% of the components were found to be operational after salvage. They are thus available for training use, whereas former sets subjected to the salt spray test were found to deteriorate rapidly to a total loss. It should be noted that while no oil contamination existed in this case the emulsion cleaner and ultrasonic agitation were effective in removing the salt deposits and loose corrosion that had been responsible for the previous progressive deterioration of equipment exposed to the salt spray test.

4. Electronic and electrical equipment damaged in a recent fire aboard a naval vessel under construction are being reconditioned by these salvage techniques. Although circumstances prevented immediate treatment and allowed marked corrosion of some components to occur, it was found that judicious use of selected metal cleaning compounds, followed by the standard cleaning, gave excellent results. It is estimated that the application of these procedures will restore to usefulness several million dollars worth of equipment which would otherwise have been scrapped.

In the course of the salvage experiences outlined above several factors influencing the practical performance of the cleaning system were studied. The results are summarized here as background information for practical application of the techniques.

When the cleaning formulation was used on equipment covered with burner fuel oil, grade heavy (Bunker C), ease of removal was greatly increased by warming the formulation to about 100°F before spraying the equipment. The higher temperature also hastens the removal of burner fuel oil, Navy Special, although in this case it is not essential. Removal of either fuel oil from equipment at temperatures below 40°F would be extremely difficult.

The efficiency of the cleaning formulation was studied at spraying pressures ranging from 20 to 80 psi. In general, it was found that cleaning could be effected at the lower pressures only with the expenditure of larger quantities of cleaning fluid. A fine spray ejected at a higher pressure cleans better and uses less fluid. Tests have also shown that even the most delicate insulation in use in electrical motors or in most electronic equipment is not damaged by spraying with a fine spray at 80 psi air pressure at a nozzle-to-surface distance of a few inches.

Ultrasonic agitation is almost essential for satisfactory and economical cleaning of electronic assemblies. It is difficult and time-consuming to get the last traces of fuel oil from deep crevices and inaccessible places near small components in electronic equipment by the spray method. However, most equipment can be cleaned quickly by immersing it in an ultrasonic cleaning tank containing the cleaning composition. Direct-current electrical motors and motor-generators, which are difficult to salvage by simple spraying procedures, were successfully salvaged when the ultrasonic equipment was used in conjunction with the spraying method for the removal of the fuel oil and sea water.

When contaminants are encountered which resist removal by the cleaning emulsion, alternate cleaning compositions may be employed. For example, the heavy tarnish or green discoloration often encountered on copper, bronze, or brass fittings after being in contact with sea water can be removed readily by immersing the component for about one minute in a solution of an inhibited sulfamic acid in warm water (about 140°F). This treatment should then be followed with an alkaline rinse such as a detergent solution of ammonium hydroxide for about one minute. Both cleaning compositions should be used in separate ultrasonic cleaning baths. Fresh water flushing, preferably with hot water (150° to 180°F) should be utilized after each cleaning step in order to prevent contamination of the next cleaning bath. After all special cleaning has been accomplished the apparatus salvage is completed by the method outlined in Appendix A. In many cases resistant contaminants may be removed by brush application of special cleaners without immersing the whole assembly in the aggressive cleaning solution.

During the submersion tests it was observed that the submersion times of 18 hours, 72 hours, or even 30 days did not change the pattern of salvage. The equipment is as wet in 18 hours as it is likely to be if submerged for longer times. Slight corrosion does occur when equipment is submerged for extremely long periods, but as long as the equipment is completely submerged, corrosion progresses very slowly.

It was noted that most of the difficulties experienced in the salvage of equipment containing electrical coils would have been eliminated if the coils had been adequately sealed against moisture before immersion. It was found that existing equipment could be readily treated with such a sealant during overhaul with a resulting marked decrease in susceptibility to flooding damage. The details of this phase of the investigation are reported in Appendix B.

CONCLUSIONS

Formulations have been developed for removing Navy Special fuel oil and water from electrical, electronic, and mechanical equipment which has been submerged in sea water subject to contamination with the oil. An integrated procedure has been developed for salvaging the equipment after submersion in fuel-oil-contaminated sea water. This cleaning technique has also been found to be effective in reconditioning electronic and electrical equipment which has been exposed to smoke and salt water as a result of a fire. A method for sealing the insulated parts of electromotive equipment against the penetration of flooding water has also been developed.

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* * *

APPENDIX A

RECOMMENDED PROCEDURE FOR RECOVERY OF ELECTRICAL, ELECTRONIC, AND MECHANICAL EQUIPMENT AFTER SUBMERSION IN WATER

WORKING FORMULATIONS

The oil-displacing composition adopted and used for salvaging equipment is formulated as follows:

The cleaning organic solvent concentrate is emulsified with an equal volume of water to give the following formulation:

- 44.5 vol-% 140° F flash-point aliphatic solvent
- 5.0 vol-% diesel fuel
- 0.5 vol-% polyethyleneglycol 400 monooleate S1008*
- 50.0 vol-% water.

The water-displacing composition currently in use for salvaging equipment is formulated as follows:

- 93.75 vol-% 1-butanol
- 0.25 vol-% 2,6-di-tertiarybutyl, 4-methylphenol
- 6.00 vol-% rust inhibitor concentrate composed of 3.0 vol-% basic barium dinonylnaphthalene sulfonate and 3.0 vol-% naphtha diluent to render the rust inhibitor more easily dispersed in the 1-butanol.

SALVAGE PROCEDURE

Before removing any equipment from fresh or sea water be sure that preparations have been made to proceed immediately with salvage operations. When equipment wet with water is moved into an oxygen-rich atmosphere such as air, the ferrous surfaces rust rapidly, causing severe damage to sensitive parts. If the equipment cannot be cleaned immediately, as may be the case when damage is extensive and the overall salvage operation is complex, it should be flushed with fresh water and thoroughly sprayed with the water-displacing fluid at the earliest possible moment. This will reduce the rate of corrosion on the equipment as it dries out, thus making complete salvage at a later date possible.

*Supplied by Glyco Products Co.

If the only surface contamination is fuel oil or similar material and sea water, the equipment may be flushed off with fresh water and carried immediately into step 1 below. In the case of fire damaged equipment, or if equipment has been allowed to corrode before cleaning is begun, certain supplementary procedures may be required before step 1 is applied. If severe tarnishing or corrosion is encountered on small areas of the equipment, the corrosion products may be removed by a treatment of a minute or less in an ultrasonic bath containing inhibited sulfamic acid solution. This should be followed immediately by a fresh water rinse, an alkaline neutralizing bath, and a final flushing with fresh water. The equipment should be carried immediately into step 1 of the salvage procedure to remove the final traces of the stronger cleaning agents used in the auxiliary treatment. Heavy sludges and greases may require the use of alkaline cleaners such as polyphosphates or silicates before step 1. Dense soot or carbon deposits from fire exposure are more readily removed if the affected parts are scrubbed with a stiff bristle brush during the ultrasonic treatment.

Blistered or discolored paint may be removed by sandblasting or by the use of a paint stripper, with appropriate care to prevent the stripper reaching insulation it might damage. For paint stripping, an alkaline solution containing sodium gluconate and chelating agents is effective, particularly when the treatment is combined with ultrasonic agitation. These auxiliary cleaning chemicals are most effective in hot water. The concentrations used are usually in the range of 4 to 8 ounces per gallon of water. Exposure time should be held to one minute or less, and the cleaning solution should be flushed off in fresh water immediately after use. This prevents contamination of the cleaning emulsion of step 1, and also safeguards the equipment from possible damage by prolonged exposure to traces of these stronger chemicals.

Step 1. Spray the exterior of the equipment thoroughly with the cleaning emulsion to remove as much contaminant as possible. Flush the equipment with fresh or sea water. If access can be gained to the interior of the equipment, spray the interior thoroughly, then flush it with water. If the construction of the equipment prevents access to the interior, disassemble and spray the interior thoroughly, then flush with water.

Step 2. After most of the oily contaminant has been removed by the flushing process, the last traces of contaminant and sea water can be removed from the equipment by subjecting it to ultrasonic radiation while immersed in the cleaning emulsion in an ultrasonic cleaning tank. This treatment is particularly effective in removing contaminants from crevices and narrow clearances. The ultrasonic cleaning treatment is carried out as follows:

- a. Disassemble the equipment as far as necessary to gain ready access of liquid to all remote locations.
- b. Immerse the parts in the tank of an ultrasonic cleaning apparatus containing the cleaning emulsion and operate according to instructions supplied by the manufacturer of the ultrasonic apparatus. An intensity of about five watts of radiated energy per square inch is effective.
- c. After the ultrasonic treatment in a bath of the cleaning emulsion, flush with fresh water; then repeat the ultrasonic treatment using fresh water to remove excess cleaning emulsion.

Step 3. Blow as much water as possible from the equipment with clean compressed air at not more than 50 psi pressure.

Step 4. Spray all parts of the equipment with the water-displacing fluid. Remember that this fluid must penetrate to all parts of the equipment that have been wet with water. After spraying, allow 20 minutes for the water-displacing fluid to penetrate, displace, and combine with the water remaining in the equipment.

Step 5. The residual mixture of water and water-displacing fluid should now be evaporated from the equipment by blowing with clean, heated, compressed air or heated air from an electric blower, by storage of the equipment in an electric oven with good air exchange, or by simply allowing sufficient time for the equipment to air dry. (No further corrosion will take place on the equipment after it has been sprayed with the water-displacing fluid.)

If there is no fuel oil or lubricant contamination, omit steps 1 and 2 and start the salvage procedure as indicated in step 3.

Mechanical equipment is ready for operation after a thorough lubrication. Electronic equipment can be operated as soon as the drying process has progressed to where all components are operable. On electric motors and motor-generators, operation should not be attempted until the field-to-ground resistance exceeds 8000 ohms. After starting any equipment it should be operated for several hours under no load or very light load to secure gentle internal heating to complete the drying. This treatment should return the equipment to its original electrical characteristics. Operation should continue until field-to-ground resistance exceeds 1 megohm before applying a capacity load. If in an unusual case the equipment cannot be returned to operational condition, it should be sprayed again with the water-displacing fluid and the drying process repeated as before.

The following precautions should also be observed in all salvage operations:

Journal bearings - remove waste packing in bearing chambers, clean as directed for other equipment above, add new packing, and saturate with suitable lubricant.

Ball and roller bearings - remove grease either by disassembly and cleaning or by forcing new grease through bearings until all old grease has been displaced.

Carbon brushes - replace carbon brushes in generators and dc motors.

Contact points in switches and relays - remove the rust-inhibiting film left from the water-displacing fluid by wiping points with lintless cloth soaked in naphtha solvent or ethyl alcohol.

APPENDIX B

WATER RESISTANT SEALANT FOR WINDINGS OF ELECTROMOTIVE EQUIPMENT

One of the most troublesome problems encountered in the salvage of electrical power equipment is the removal of water from deep closed-end voids in electrical windings. The difficulties are frequent and serious when the windings have been taped and varnished in such a manner as to permit access of salt water to labyrinth voids within. Serious trouble arises when de equipment containing such coils is returned to service with traces of salt water still present. Electrolysis is set up at insulation flaws and produces corrosive chemicals which enlarge the insulation breaks so that electrolytic short circuits and overheating lead to complete failure of the windings. An effective method for sealing such voids in electromotive units would reduce flooding damage and simplify salvage. It seems certain that such sealing could be accomplished during manufacture if it were required by specifications. However, there is a tremendous amount of equipment now in service with the fleet which is vulnerable to flooding because of labyrinth voids in the windings, and a method is needed by which such equipment can be upgraded in flooding resistance at overhaul. This Appendix summarizes the investigation leading to such a method and outlines an effective impregnation procedure using available sealants and simple auxiliary equipment.

For the use planned, it is essential that the sealant and the method of application should not be such as to affect the electrical or mechanical integrity of the insulations and insulating varnishes now in service. The sealant must also be capable of wetting and spreading smoothly over the coatings currently in use.

In general, the windings of equipment now in service will have been treated with one of the varnishes listed under Military Specification Mil-V-1137A, or some equivalent material. The specification covers four types: Grade BA, black air-drying; Grade BB, black baking; Grade CA, clear air-drying; and Grade CB, clear baking. The baking grades are required to be phenolic; the air-drying grades may also be phenolic but this is not specified.

These varnishes are supplied in Type M and Type AN. Type M, which has less stringent requirements on solvent flammability and toxicity than Type AN, is commonly used by manufacturers, while Type AN is used in naval repair facilities. The properties of the cured varnishes obtained from the two types are similar.

Attempts to make the windings impervious to water by an encapsulating procedure that placed a 1/8-inch layer of epoxy or rubber sealant over the exterior of the winding were abandoned because the encapsulated windings overheated when operated at rated load. Attention was therefore turned to treatments that would seal the voids in the winding, without impairing heat dissipation.

Examination of windings in service equipment showed that present methods of applying varnish left uncoated and unsealed spaces within the winding, and experimental coating tests demonstrated that the winding voids would almost never be adequately sealed if the varnish were applied by brushing, spraying, or dipping at atmospheric pressure. On the other hand, it was found that vacuum impregnation led to effective sealing and filling of the winding voids by any of the varnishes of Specification Mil-V-1137A.

A high vacuum is not required for successful impregnation; a final pressure of 15 to 25 mm Hg (such as is obtainable from a water operated aspirator pump) is satisfactory. It is necessary, however, to employ a two-step process in which the first application of varnish is cured by air-drying or baking, as may be appropriate, before the next vacuum impregnation. This stepwise buildup of the sealant coating is especially important when air-drying varnish is used. In this case it is necessary between coats to allow time for complete drying and hardening of the varnish in deep pores to prevent trapping of solvent in the deeper voids when the next coat is applied. Air-drying sealants occasionally require a third treatment for complete water exclusion.

The choice of varnish type among those available under Mil-V-1137A depends on the nature of the insulation and varnish already on the windings. If these are known to be baked on, then a baking enamel is permissible and often preferable because of the smaller shrinkage during cure and the shorter curing time. If the varnish already present is an air-drying type it is likely to blister or decompose during the baking of BB or CB varnishes. Hence if the equipment is not known to be insulated entirely with baking materials it is necessary to do the auxiliary sealing with an air-drying varnish such as BA or CA. Class H motors coated with a silicone varnish may be waterproofed by a two-stage reduced-pressure impregnation with the varnish supplied under Military Specification Mil-L-2707, "Insulation Electrical, Liquid Impregnating, High Temperature." This or other silicone varnishes should not be used on any equipment not already containing silicone insulation, because the vapors may cause damage to carbon brushes and commutator assemblies.

Equipment protected from water penetration by this method has been shown to retain its original heat dissipation characteristics, so that it does not overheat in service. AC motors, dc motors, and ac-dc motor-generators whose windings had been coated with Mil-V-1137A AN type varnishes by two-stage impregnation have been salvaged from sea water immersion and operated successfully under rated loads for 300 hours or more. They showed no evidence of overheating or water damage.

To summarize, it has been found that vacuum impregnation of windings for electromotive equipment can prevent trapping of salt water by voids inaccessible to the salvage procedure. This allows simple and quick salvage of flooded units and could be an important contribution to the recovery potential of a damaged vessel. The method is applicable to equipment in service as well as to that in production; it employs presently specified varnishes in simple and relatively inexpensive equipment. It does not significantly change the operating characteristics of electromotive units to which it is applied. The results suggest the possibility of developing shipboard electromotive equipment which will be nearly immune to flooding damage if simple salvage procedures are promptly applied.

* * *

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-- DISPLACEMENT OF ONE LIQUID BY ANOTHER AS A RESULT OF UNBALANCED
-- SURFACE FORCES. TWO FORMULATIONS HAVE BEEN DEVELOPED, ONE FOR THE
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-- FROM ELECTRICAL, ELECTRONIC, OR MECHANICAL EQUIPMENT. BOTH
-- FORMULATIONS ARE RELATIVELY HARMLESS TO MOST ELECTRICAL AND
-- ELECTRONIC INSULATION. THE FUELOIL-CLEANING EMULSION RETARDS
-- RUSTING OF THE FERROUS METALS WITH WHICH IT COMES IN CONTACT, WHILE
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-- RUST INHIBITING FILM OVER THE SURFACES OF THE EQUIPMENT. ELECTRICAL
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-- NAVY SPECIAL FUEL OIL AND SEA WATER WERE EFFECTIVELY SALVAGED BY
-- TREATMENT WITH THE FUEL-OIL-CLEANING EMULSION AND THE WATER-
-- DISPLACING COMPOSITION. AN INTEGRATED PROCEDURE IS OUTLINED FOR THE
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