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A PRESERVATIVE-LUBRICANT FOR SMALL ARMS

H. R. Baker, et al

Naval Research Laboratory
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CONTENTS

ABSTRACT

A protective and lubricating coating has been developed for use on the M16 submachine gun and other small arms. It protects the M16 submachine gun used by SEAL and UDT personnel during immersion in sea water and exposure to sand, dust, and jungle environment. Treated weapons are ready for immediate use. The preservative-lubricant coating which was developed for this application is a compound of arachidyl-behenyl amine and tetrapropenylsuccinic anhydride, with an oil and a solvent. It is packaged in aerosol cans for field use. The spray penetrates and displaces water from wet surfaces. The dried coating is firm and nontacky but breaks down to a greaselike consistency between moving parts. The consistency of the coating material can be completely controlled by adjustment of the formulation. It is compatible with other lubricants. The coating provides excellent corrosion protection, because the entire solid phase consists of a waxy polar inhibitor. The oil phase, carrying the inhibitor in solution, can creep out to reprotect scratches and worn areas in the coating. Metal specimens and complete weapons were protected from damage during exposure to sea water for 3 days.

Coatings of this type offer promise as preservatives for exposed parts of operating machinery, spare parts, tools, etc., where lubricant compatibility or operation without depreservation is desirable.

PROBLEM STATUS

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

AUTHORIZATION

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INTRODUCTION

Due to the nature of their operations, the Navy's SEAL Teams need equipment which is reliable under adverse environmental conditions. Among the most severe of these conditions is immersion in the sea, followed by use in the high humidity and heat of the jungle. One of the most effective weapons in the SEAL's arsenal is the M16 submachine gun, similar in construction to the M16 rifle being used extensively in combat in Southeast Asia. Because of its lightness and small size, it is easier to carry than other rifles or machine guns. This weapon, however, is susceptible to salt-water corrosion because of the variety of metals used in its construction.

In conjunction with its projects in corrosion inhibition, dry-film lucricants, and aircraft machine-gun lubrication, NRL was requested by the Naval Ordnance Systems Command to develop a protective coating for the M16 submachine gun. Such a coating must not interfere with the normal functioning and handling of the weapon. It must provide any lubrication required by the moving parts and protect the weapon against corrosion during immersion at considerable depths in the sea and subsequent use under warm, humid jungle conditions. Finally, it must either be permanent or easy to apply under field conditions.

The materials used in the construction of the weapon must be considered in evaluating the corrosion threat and avoiding incompatibilities with protective coatings. The metals used to fabricate the major components of the M16 are various steel alloys–301, 304, and 321 stainless steels–and aluminum alloys–7075 and 6061. Most springs are made of 177 stainless steel, but two are made of high-tensile music wire. The aluminum parts are anodized, and the steel parts are finished with a manganese phosphate coating. (In some earlier procurements, the bolt and bolt-carrier parts were chromium plated.) Polymeric and composite materials are used extensively in the stock and hand guards. These materials include rag-filled phenolic, fiberglass-reinforced phenolic, urethane foam, Delrin, and a plastisol coating.

Two approaches to the problem of gun protection and lubrication were considered–the use of low-surface-energy polymeric coatings and the use of oil-based or wax-based compositions. Polytetrafluoroethylene (Teflon) films bonded to metal surfaces as dry-film lubricants had been developed by NRL several years ago for use on Marine Corps small arms (1). These coatings have a low coefficient of friction and are repellent to most liquid and solid contaminants. They are, however, not impervious to moisture and are consequently not exceptionally good as corrosion preventatives. Also, for good performance the pure Teflon films must be sintered at 750°F, which would adversely affect the aluminum parts. Composite coatings containing Teflon and a thermosetting resin binder appeared to be a better choice for this application. Although not as durable as sintered Teflon coatings, they provide better corrosion protection with similar friction properties and require a lower curing temperature. It was recognized that even if the polymer coating was completely successful, a preservative which could be applied in the field would be needed to protect the bore, chamber, and any places where the permanent coating became worn off. A sufficiently effective material of this type, applicable to the entire weapon as an alternative to the polymer coating, might offer several advantages:

1. The coating could be applied in the field without complete disassembly of the weapon and without any alteration of weapons already in service in the field.

2. The same material could be used to protect the bore and chamber.

3. Under conditions of wear and abrasion, a semisolid coating can be more tenacious than a hard brittle coating.

Attention was therefore concentrated on the development of an alternative protective system derived from petroleum and other hydrocarbons. The salts of amines and organic acids were of particular interest. Amine salts, amides, and imides have been used previously as corrosion inhibitors in lubricants, preservatives, fuels, and petroleum production and transportation (2-6).

The present report describes the development of both a permanent and a renewable temporary coating and offers suggestions for a Military Specification covering the latter type.

COATING DEVELOPMENT

Composition

Experimental procedures and materials are given in detail in Appendix A.

The polymer coating selected on the basis of past experience for evaluation as a permanent water repellent and low-friction coating was a resin-bonded-fluorocarbon-polymer coating produced by E. I. duPont de Nemours and Company and designated "Durable Release Coating, Green, 100-B-60571." A 0.6 to 0.7-mil-thick coating was applied and cured at 400°F. The coating exhibited water contact angles of 100 to 108 degrees and static and dynamic friction coefficients of 0.17 and 0.12, respectively. It carried a load of 56,000 psi during 1000 repeated passes over the same wear track with no increase in friction coefficients. This coating therefore provided durability and low friction, as well as a lower (400°F) curing temperature than sintered Teflon coatings. Some difficilty was encountered in reassembly, because the original clearances between mating parts left no allowance for the 1.2 to 1.4 mils of coating. The tight fit caused some peeling of the coating from pins and the barrel during assembly. For optimum usefulness, this coating would have to be applied during manufacture of the weapons, and some changes in the clearances between parts would have to be made.

In seeking a renewable coating material which could be applied to the M16 in the field, corrosion protection was given primary attention. Lubrication was a secondary consideration. The consistency of the material was also an important factor to be considered, because it would affect application, handling of the weapon, and malfunctioning under sandy or dusty conditions. Many already available preservative compositions, including those covered by Military Specifications MIL-C-16173C and MIL-C-11796B, were screened for possible use. Those which were effective in preventing corrosion in sea water were all undesirably greasy or tacky. The nontacky coatings were particularly deficient in preventing corrosion in scratched-panel tests, due to their lack of reprotecting ability.

A series of amine-acid salts under study at NRL as corrosion inhibitors appeared to have promise for use in this problem. Not only are they effective in low concentrations as rust inhibitors, but some of them are also waxy in bulk form and capable of forming highly water-resistant coatings. When combined with oils, these inhibitors form nontacky coatings with excellent water resistance and very good salt-water-corrosion protection. The oil softens the coating slightly, provides lubrication, and carries enough of the inhibitor in solution to prevent corrosion in crevices and on rubbing surfaces and to reprotect scratched areas in the coating by oil migration. When the coating is applied from solution, the film formed is firm to the touch and not tacky or oily. Where the film is sheared between moving parts, it breaks down to a greaselike consistency.

The composition of the coating solution which was developed is given in Table 1. The amine and acid anhydride react when dissolved in the alcohol to form a mixed amide-amine salt. The selected amine and acid anhydride were chosen from many available ones to achieve optimum hardness, corrosion inhibition, solubility, and resistance to water emulsification. The acid anhydride, succinic anhydride with a branched dodecenyl side chain, has been used as a corrosion inhibitor in turbine oils and is readily available from commercial sources. The primary amine is a mixture of saturated, unbranched fatty amines. Gas-liquid chromatographic analysis of a typical batch of the undistilled amine indicated the following weight-percent composition: C_{16}-0.4, C_{18}-3.2, C_{20}-47.0, C_{22}-48.4, and C_{24}-1.0. The major variation observed between batches was in the C_{18} content, which could be as high as 7.5 wt-% without affecting the properties of the coating. A sample of the distilled-grade amine had a similar distribution of chain lengths but only traces of secondary amines, while the undistilled material contained as much as 5 wt-% secondary amines.

Table 1
Composition of Preservative-Lubricant Solution

Component	Content (wt-%)
Arachidyl-behenyl primary amine*	5.12
Tetrapropenylsuccinic anhydride	3.93
Symbol 1010 lubricating oil	10.80
Oxidation inhibitor†	0.15
Ethanol‡	25.00
n-Heptane	55.00

*Distilled grade.
†2,6-Di-tert-butyl-4-methylphenol has been used in this study.
‡Maximum of 2% water.

The titration of a typical sample of the amine with the anhydride (Fig. 1) yielded an equivalence point of 0.48 g of anhydride per gram of amine, close to the expected value. The pH at this point was 8.1. To reach pH 7.0, 0.64 g of anhydride per gram of amine was required, or about 1.54 eq of anhydride per equivalent of amine. At first it was thought that the anhydride was being hydrated to form the acid, which then reacted with 2 eq of amine to form the salt. However, hydration of the anhydride under the mild conditions used is unlikely. Instead, 1 eq of amine may add to 1 mole of the anhydride to form an amide and a carboxyl group. A second equivalent of amine can then react with the carboxyl group to form the amine salt. This mechanism is supported by the fact that the anhydride could not be saponified with aqueous KOH without heating to about 70°C. Also, when the resulting potassium salt was hydrolyzed and the freed acid was reacted with the amine, the properties of the resulting di-salt differed from the properties of the anhydride-amine reaction product. The product made from the acid was much more oil soluble than that made from the anhydride. The infrared spectrum of the anhydride-amine product showed amide absorption bands not found in the spectrum of the acid-amine product. Titration of the amine with the acid yielded an equivalence point and pH at equivalence nearly identical to those obtained with the anhydride.

Another method was devised to determine the number of amine-acid salt groups in the products. A sample of the succinic anhydride was hydrolyzed to the acid, which was then reacted with the amine. Anhydrous hydrogen chloride was passed through a heptane solution of this substituted ammonium salt to form the original dibasic acid and twice as many moles of the amine hydrochloride, as indicated by the neutralization equivalent of the isolated acid (calc., 149; found, 146) and the chloride determination of the reaction

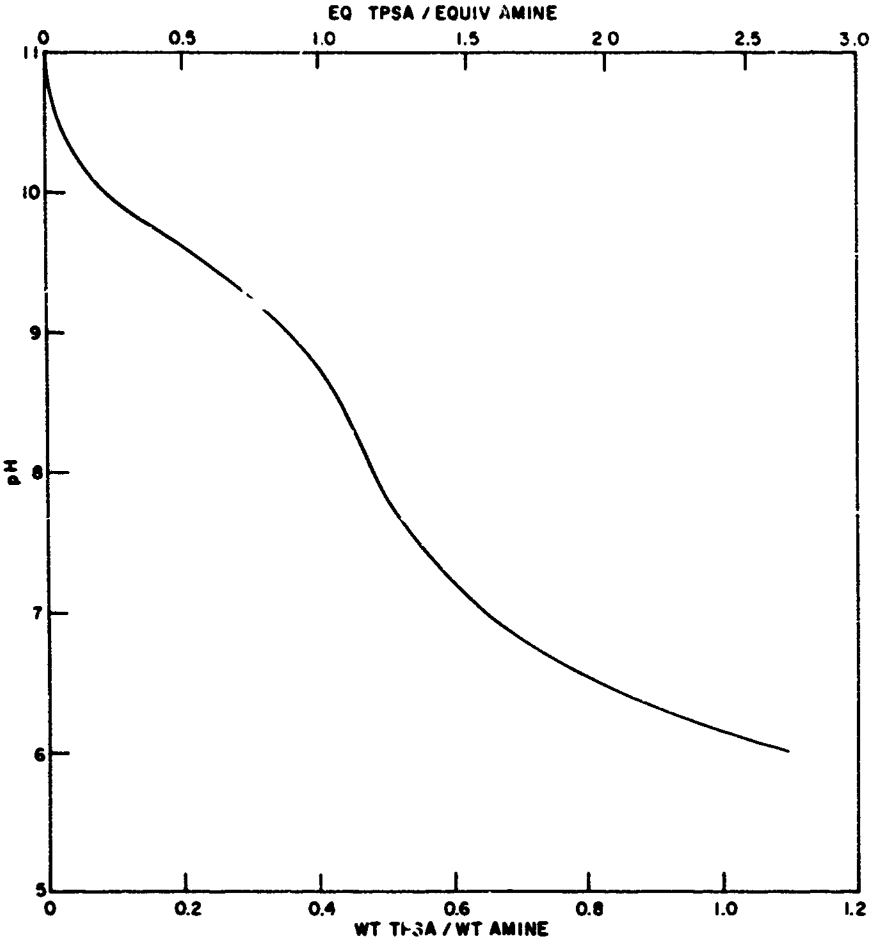
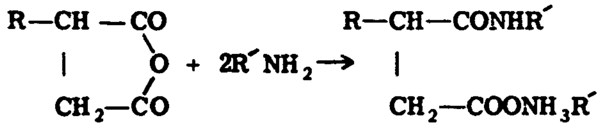


Fig. 1 - Titration of arachidyl-behenyl amine with tetrapropenylsuccinic anhydride (TPSA)

products (milliequivalents of chloride per gram of sample: calc., 2.16; found, 2.38). A similar treatment of the product from the anhydride and the amine yielded only about one half of the previous amount of chloride in the products (milliequivalents of chloride per gram of sample: calc., 1.10; found, 1.18) and a much higher neutralization equivalent for the isolated acid (calc., 586; found, 755). It is reasonably certain, therefore, that the anhydride and the amine react to form the hybrid amide and amine salt:



Alcoholysis of the anhydride with ethyl alcohol, the reaction solvent, can also occur, forming the half ester. In the presence of the amine, however, the alcohol will be displaced, and the amide structure will be formed as before. If the amine-acid salts are heated, water will be driven off and the amide formed (2).

The consistency of the coating material, composed of the amine-acid corrosion inhibitor and a light oil, can be controlled by varying the proportion of oil in the coating and the amount of excess tetrapropenylsuccinic anhydride (TPSA) in the inhibitor. Two measurements of consistency have been used to characterize this coating material. The hardness of the undisturbed coating as it is formed by the evaporation of the solvent is determined by measuring the needle penetration of a bulk sample cast from molten material. (The melting point is about 130°F.) The consistency of the coating after it has been broken down by shearing is then determined by shearing this sample in a grease worker and measuring its cone penetration.

In Fig. 2, typical effects of composition on the consistency of the coating material are shown. As the proportion of oil in the coating material is increased, both the needle and the cone penetrations increase. As the ratio of anhydride to amine is increased, the cone penetration increases, and the needle penetration decreases, reaches a minimum, and increases again. The four ratios shown correspond to 1.5, 1.6, 1.7, and 1.84 eq of anhydride per equivalent of amine, respectively. Variations in the raw materials can have large effects on the consistency of the product. One cannot, therefore, expect to reproduce the data of Fig. 2 with a different batch of starting materials. Their behavior will be similar enough, however, so that this pattern can be used as a guide in preparing trial batches and adjusting the formulation until the proper consistency is obtained.

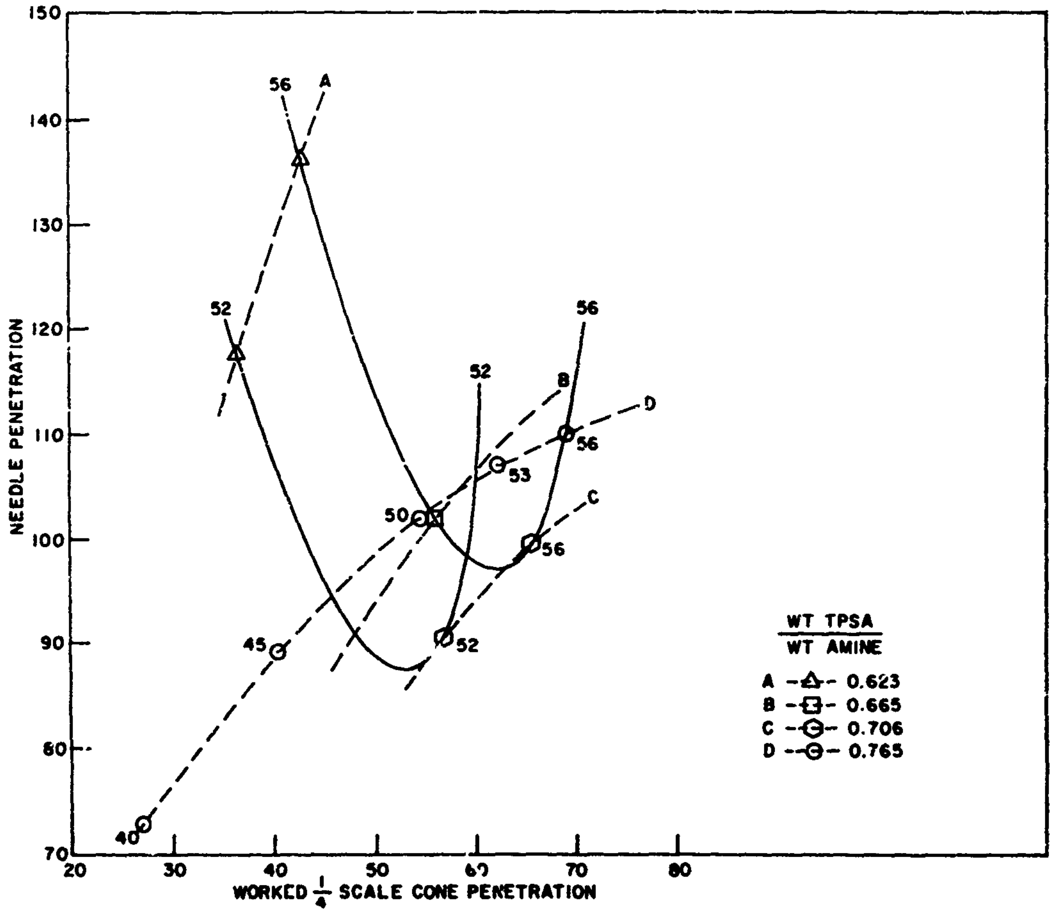


Fig. 2 - The effect of composition on the consistency of the coating material. All numbers on graph refer to weight percent of oil in coating material.

The consistency found to be optimum for the coating material—considering handling, dust and sand adhesion, durability, lubrication, and the force needed for the initial operation of various mechanisms—is characterized by a needle penetration of 130 and a cone penetration of 75. Limits for the acceptable range of consistencies have been established as a needle penetration of 100 to 160 and a worked cone penetration of 55 to 90. To allow for variations in the measurements due to reproducibility errors, the material should be prepared so as to fall within more stringent limits: a needle penetration of 110 to 145 and a worked cone penetration of 62 to 83.

In trial batches and laboratory samples, all of the difficulties encountered in obtaining the correct consistency were traced to impurities in the amine or variations in the oil. The use of a distilled-grade amine should eliminate or reduce the variations from this source. The oil affects the consistency, because the solubility of the inhibitor in the oil is affected by the aromaticity of the oil. Paraffinic oils are preferred, but the formulation can be adjusted to employ other petroleum lubricants and synthetic oils as well.

A mixture of heptane and ethanol was found to be the most satisfactory solvent for the application of the coating material. Mixtures of chlorinated hydrocarbons and ketones were also usable, but precipitation of the solids occurred on cooling the solutions to about 15°C. The heptane-ethanol solutions became cloudy at 7°C (45°F), which is one reason that the solution is recommended for tropical use only. 2-Propanol can be used in place of the ethanol, but the coating dries more slowly. The amount of water in the alcohol must not exceed 0.4% of the entire solution. Blended-hydrocarbon solvents can be used in place of the heptane, but the slowness of evaporation of the heavy ends extends the time required for the coating to reach its proper hardness. Because of the alcohol, the solvent has water-displacing properties (7). The coating may, therefore, be applied to wet surfaces. The water film will be displaced, and a protective film will be formed on the surface. Because of the dilution of the alcohol by the heptane, the high proportion of nonvolatiles, and the rapid evaporation of the solvent, this composition is not as effective in displacing water as some compositions developed specifically for this purpose. Undisplaced water droplets may leave holes in the protective coating, so on wet surfaces a second coat should be applied after the first is dry.

Corrosion and Materials Studies

A number of untreated parts from a M16 rifle were exposed to fresh-water fog for 4 days in a fog cabinet to evaluate the susceptibility of the metal parts to corrosion. Other parts treated with the resin-bonded fluoropolymer were also exposed to determine the permeability of that coating. The untreated steel parts were generally well protected by their conversion coatings and rusted only where bare metal was exposed. The aluminum forgings showed corrosion products blooming through the anodizing in a few spots. The resin-bonded-fluorocarbon coating provided almost complete protection, even where applied over bare metal. The stainless-steel springs were quite resistant to oxidative corrosion, but it was felt that they might be susceptible to stress-corrosion cracking in the presence of sea water. Several springs were therefore immersed, under stress, in synthetic sea water at 60°C for 14 days. No failures occurred. During immersion testing of the entire submachine gun, one detent spring cracked. Several of these springs were obtained from the manufacturer and immersed in synthetic sea water under stress with no failures. The one observed failure therefore may have been due to overhardening.

The extensive use of plastics in the construction of the M16 weapons required that the compatibility of these materials with synthetic lubricants be determined prior to considering the use of such oils. A grease containing a bis(2-ethylhexyl)sebacate oil was applied to the hand guards, stock exterior, stock interior, pistol grip, and sling swivels. After 2 months at room temperature, the parts were examined for softening or cracking.

The coating on the sling swivels was softened slightly but not noticeably weakened. None of the other parts was affected.

To screen renewable coatings for their ability to prevent corrosion, coated and scratched coupons of cold-rolled steel were immersed in synthetic sea water for 3 days and then examined for evidence of rusting. The best protection was provided by soft coatings, such as greases, soft solvent-cutback preservatives, and heavily inhibited oils. These materials are not suitable for use on weapons because of the difficulties in handling and the adhesion of sand and dust which would result from their softness and tackiness. Second in effectiveness was the group of alkyl and alkenylsuccinic anhydride-arachidyl-behenyl amine compounds mixed with various oils. The mixed alkenyl derivative was slightly more effective than a dodecylsuccinate. Petroleum oils were more effective in reprotecting the scratch than were synthetic ester oils. Oils of low viscosity were more effective than those of high viscosity. These effects are caused by the greater solubility of the inhibitor in petroleum oils and the more rapid spreading of the lower viscosity oils. The coupons with the best coatings in this group typically showed one or two spots of rust on the flat surfaces, one spot along the scratch, and a few on the edges and corners. The least effective coatings, particularly in regard to rusting along the scratch, were the hard preservative coatings and the very thin protective films. This would be expected, as they have no mobile phase to reprotect scratches or fill pinholes.

The most promising renewable coatings were applied to three assemblies made up of parts from a M16 rifle. These assemblies provided typical examples of (a) metals, finishes, and galvanic couples, (b) methods of assembly and clearances between parts, and (c) areas not readily accessible to coating sprays. The coated assemblies were immersed in synthetic sea water under pressure, removed and wrapped in plastic bags while still wet, then examined for corrosion after 3 days. These experiments confirmed that the amine-acid coatings with petroleum oil were effective in protecting the actual materials of the weapon under simulated exposure conditions. It was also possible to determine the extent of disassembly of the weapon necessary to obtain adequate coverage. It was found, for example, that all of the parts of the trigger mechanism could be adequately protected without disassembly, if the spray was directed into the receiver until all parts of the mechanism were coated and the parts were operated to ensure penetration of the coating solution around pins and between parts. The extent of disassembly found necessary is described in the instruction sheets which form Appendix C of this report.

Two complete M16 submachine guns were subjected to corrosion testing. The first was coated with the resin-bonded-fluorocarbon polymer. This weapon functioned normally in test firing after a few tight areas were relieved. The weapon, free of oil and grease, was immersed in synthetic sea water under 100 psi pressure for 30 min, then removed and sealed in a plastic bag. After 3 days, considerable rusting had occurred in the bore, chamber, and gas cylinder, which were without any protective film. Slight rusting also occurred around most of the pins. After the bore, chamber, and gas cylinder had been cleaned, the weapon was again test fired and functioned properly without lubricating oil or grease. The ball cartridges (M193) were found to be watertight under the same immersion conditions.

The second submachine gun was used to test several different batches of the preservative-lubricant coating. The partially disassembled weapon was sprayed, and the coating allowed to dry. After the moving parts of the weapon had been operated, it was immersed in synthetic sea water under 100 psi pressure and then exposed to a humid atmosphere for 3 or 4 days as before. This weapon was subjected to two such tests in the laboratory and one immersion in the surf at the Naval Amphibious Base, Little Creek, Virginia, during field trials. Superficial corrosion was noted in a number of places during these tests but appeared twice in only six places: (a) at the point where the hammer and firing pin were in contact, (b) on the extractor spring, (c) on the butt plate, (d) around the front-sight detent spring, (e) in the gas cylinder, and (f) on exterior steel surfaces.

Disassembly of the extractor and front sight before coating is now recommended to improve the protection of these parts. In nearly all cases, the corrosion products were removed by normal cleaning. No parts failed as a result of corrosion, and the weapon always functioned properly.

Service Testing

The preservative-lubricant-coated weapon was also subjected to low temperatures to determine the lower operating-temperature limit. At 0°F, dummy ammunition was chambered from a magazine and ejected normally. All mechanisms operated properly. The coating solution cannot be sprayed at temperatures below 40°F, however, as the inhibitor begins to come out of solution at this temperature. Since the pour point of the oil used in the preservative lubricant is -70°F, the stiffening of the coating at moderately low temperatures is probably due to the hardening of the waxy inhibitor and the decreased solubility of the inhibitor in the oil. If coatings for low-temperature use are desired, changes in the inhibitor will have to be made.

For the first field test of the preservative-lubricant coating, 1 gal of the solution (designated N4002-36) was made up in the laboratory and packaged commercially in aerosol cans. A trip was made to the Naval Amphibious Base, Little Creek, Virginia, to deliver the material to the UDT and SEAL team personnel, to demonstrate the method of application, and to observe the effect of sand exposure and immersion in the surf on the operation of the preservative-coated and polymer-coated weapons.

One M16 submachine gun was completely coated with the preservative-lubricant. Another was coated with the resin-bonded-fluorocarbon polymer, with the preservative sprayed on the areas not polymer coated and on the parts subject to wear. After the coating had dried for 2 hr, the weapons were immersed in the Chesapeake Bay surf on a sandy beach. Neither weapon was rendered inoperable, even though sand was allowed free access to the moving parts. The sand appeared to adhere less to the polymer coating than to the preservative-lubricant, but the polymer coating showed more abrasion damage caused by the sand. Most of the sand adhering to both weapons could be washed off by simply dunking the weapon in the water. When both weapons were dropped in dry sand and picked up gently, an almost complete coating of sand adhered to their exteriors, slightly less complete on the polymer coating. Once again, however, the sand washed off both weapons when they were immersed in the water and agitated. Although no range was available for live firing at this time, both weapons appeared to be functioning properly. Several cans of the preservative-lubricant were left with the UDT and SEAL personnel for use in training exercises and live firing. They reported later that the material had performed very well, creating no problems in simulated combat operations including live firing.

Because of the apparent success of the preservative-lubricant and because use of the polymer coating would require coating application during manufacture as well as alterations in parts clearances, it was decided that the preservative-lubricant should be selected for further development.

A larger batch of the solution (designated N4002-51) was then prepared in the laboratory. This 27-gal batch was commercially packaged in 800 aerosol cans. Most of this batch was shipped to the Atlantic and Pacific coast SEAL and UDT groups and to Navop, Vietnam. Smaller quantities were sent to the Army and Marine Corps for evaluation. The Army evaluated this material along with several other lubricants for the M16 rifle (8). The conclusion of this study was that the semifluid lubricant available under specification MIL-L-46000A was the best lubricant for use on the M16 rifle under the conditions encountered by Army personnel. The NRL preservative-lubricant of batch N4002-51 was found to permit excessive malfunctions after prolonged firing without cleaning and in the dynamic dust test. Unfortunately, at the time this batch of the preservative-lubricant was prepared, the technique of measuring and controlling the consistency of the material had not been developed. The needle and cone penetrations of the material, as measured later, were 225 and 13, respectively, while the currently recommended median values are 130 and 75. This coating was therefore too soft in the undisturbed condition and too stiff after shearing. The former condition may have increased the malfunction rate in the dust tests, while the latter increased the malfunction rates throughout the tests. It is felt that the currently produced material would provide significantly better performance under dusty conditions and in prolonged firing. The oil used in this batch was of grade 1005. The more viscous 1010 oil is now recommended, providing better lubrication and less evaporation loss.

The Army tests showed that both the NRL preservative-lubricant and the MIL-L-46000A lubricant kept M16 rifles in operating condition during a salt-water immersion test. Immersion testing in synthetic sea water under pressure in this laboratory confirmed that the MIL-L-46000A lubricant is effective in preventing corrosion under these static conditions. However, the semifluid lubricant does not have sufficient body to resist erosion by flowing water which would be encountered in surf or result from the motion of a swimmer through the water. Also. repeated immersion, splashing, and spray can quickly deplete a semifluid lubricant film through emulsification and spreading of the lubricant on the water surface. These effects were observed earlier at NRL during a study (9) of the water resistance of the MIL-L-19701 semifluid lubricant, which has a consistency similar to that of MIL-L-46000A.

Water drops formed on the surface of a lubricant film were seen to be immediately covered by oil spreading from the lubricant film onto the surface of the water drop. This oil is carried along as the drop runs off.

The results of these effects can be seen in the Army water-spray firing test. The NRL preservative-lubricant performed quite well; in fact, the malfunction rate was lower than the rate for firing under dry conditions. On the weapons and corrosion test panels in the water-spray tests, the fluid and semifluid lubricants were observed to have been largely washed off, while the NRL coating was practically intact. The NRL coating completely protected the panels against corrosion, while the other materials did not. The NRL preservative-lubricant is therefore thought to offer the best combination of corrosion protection and lubrication where exposure to water is more than just an occasional accident.

The compatibility of the preservative-lubricant with the MIL-L-46000A lubricant was studied by applying the lubricant to metal panels and to the M16 submachine gun after these had been coated with the preservative. No adverse effects were noted. The lubricant simply softened the coating gradually, and the two materials mixed when rubbed, forming a coating with about the same consistency as the applied semifluid lubricant but with better water resistance. It would therefore be possible for amphibious troops to apply the preservative lubricant to their weapons prior to a landing for the maximum protection against corrosion and from then on use the MIL-L-46000A lubricant for relubrication, thus taking advantage of its low malfunction rate during prolonged firing and under dusty conditions. Should subsequent exposure to rain or immersion wash off the semifluid lubricant, the preservative would still protect much of the weapon.

Other synthetic and petroleum lubricants are also compatible with the coating material and may be used for relubrication. More of the coating may of course be sprayed on to replace that which is worn off during use of the weapon. Oil should be removed from weapons before the initial coating is sprayed on, because the excess oil will otherwise cause the coating to be softer than the optimum consistency.

Commercial Production

Four potential suppliers of the preservative-lubricant solution were informed of the formulation, the method of preparing the solution, and the testing procedures. Three have submitted satisfactory products packaged in aerosol cans. It has been found necessary to check the consistency of each batch when different lots of oil or amine are used and often to adjust the formulation to obtain consistency values within the recommended limits. The preferred atomizer nozzle is one providing a narrow spray pattern, about 2 in. in diameter at 1 foot. The pressure provided by the propellants should not be high, because the evaporative cooling effect can cause the inhibitor to come out of solution on spraying. Fine atomization is not desirable in any case.

Freon propellants have been used because of their low flammability. These propellants may not be used aboard submarines with catalytic combustion air purifiers. Liquified petroleum gas might be substituted if this use is contemplated. In any case, it must be noted that the solvent mixture itself is flammable and has a low flash point. (The flash point of heptane is 25°F). The lower explosive limit for the solvent vapors would be reached after spraying the contents of 20 6-oz cans with an inert propellant in a sealed compartment of 1000-cu ft volume. None of the components of the coating solution is highly toxic. The toxicity threshold for continuous exposure would be reached by spraying the contents of one 6-oz can in a sealed compartment of 1000-cu ft volume. Much higher concentrations can be tolerated for short exposures.

NRL has been purchasing the preservative-lubricant in lots of 1000 cans. The material has been checked for consistency, instruction sheets have been placed in the cartons, and field-trial amounts have been shipped to the Atlantic and Pacific UDT and SEAL teams and to ComRivRon 5 in Vietnam. It is being evaluated for use on weapons and other equipment on the patrol boats as well as use on the M16 and other small arms. It is recommended that a specification be written to cover this composition so that procurement can be made through the normal supply system.

CONCLUSIONS

A preservative-lubricant composition has been developed from an amine-acid compound which provides excellent protection against corrosion to small arms exposed to repeated and extended immersion in fresh and sea water. The protective coating is readily applied in the field from aerosol containers, does not interfere with the normal handling and operation of weapons, provides lubrication, and is compatible with other lubricants. This material is being used by the Navy's UDT and SEAL teams and by river-patrol squadrons in Vietnam. Its use by the Marine Corps during the landing phase of amphibious operations would aid in preventing the corrosion of weapons exposed to immersion in the surf.

Many different amines and organic acids can be combined to produce inhibitors with a wide variety of properties. These can be mixed with various oils and solvent blends to make protective coatings for a variety of applications. The formulation developed for use on the M16 submachine gun is a penetrating and water-displacing solution, which dries to leave a nontacky film that repels water, prevents corrosion, lubricates moving parts, is compatible with other lubricants, can be removed with common solvents, and is ashless when burned.

The properties of this family of preservatives make them superior to currently stocked preservatives and lubricants for many applications, such as on spare parts, on wire rope and aircraft control cables, on hand tools and machine tools, and about amphibious vehicles and small boats. Since the coating provides some lubrication and is compatible with lubricants, spare parts could be installed without depreservation; machine tools, weapons, and other mechanisms could be protected from corrosion while remaining ready for immediate use. It is recommended that these materials be studied further to develop the best formulations and methods of application to promote their use as temporary protective coatings and packaging preservatives.

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Appendix A

EXPERIMENTAL PROCEDURES AND MATERIALS

PROCEDURES

Application of the Polymer Coating

The M16 submachine gun was disassembled, and all of the metal parts, including springs, were solvent cleaned and hand-spray coated with a 0.6 to 0.7 mil film of the duPont release coating 100-B-60571. The bore, chamber, gas cylinder, some concealed areas, and some small orifices were not coated. The coating was cured at 400°F in a recirculating oven.

Preparation of the Preservative-Lubricant Solution

The procedure for the preparation of the preservative-lubricant solution is given in paragraph 5.2 cf Appendix B of this report. In laboratory-scale preparations, the amine and anhydride were reacted in the alcohol by warming, the mixture was cooled, the oil, oxidation inhibitor, and heptane were added, and the corrosion inhibitor redissolved by slight warming.

Gas-Chromatographic Analysis of Amines

A Beckman GC-4 instrument was used in the gas-liquid-chromatographic analysis of the amine samples. For determining the chain-length distributions in the primary amines, a 6-ft column of 1/8-in. O.D. packed with 3% SE-30 on 60/80-mesh Chromosorb-W was used. A helium flow rate of 20 ml/min at a temperature of 225°C gave adequate separations from the C_{16} to the C_{22} primary amines. A better elution, particularly of the higher molecular-weight primary amines, was obtained by using a programmed-temperature increase from 200 to 275°C in 10 min, with a flow rate of 30 ml/min. Known C_{16}, C_{18}, and C_{20} primary amines were used as standards. To determine the secondary amine content, a 1-ft column of 1/8-in. O.D. packed with 3% JXR on 60/80-mesh Gas Chrom Q was used. The flow rate was 30 ml/min, and the programmed-temperature increase was from 200 to 350°C in 15 min. A mixture known to consist largely of C_{20} and C_{22} secondary amines was used as a standard.

Titration of Amine with Acid and Anhydride

A weighed sample of the amine was dissolved in a mixture of equal parts of ethanol and n-heptane with 0.5% water. The acid or anhydride was added in weighed increments, and the pH of the solution measured with a pH meter after each addition.

Determination of the Amine-Acid Complex Structure

The number of amine-acid salt structures in the complexes was determined by acidolysis. Anhydrous hydrogen chloride was passed through 0.5 g of the sample dissolved in

10 g of heptane. After standing overnight, the mixture was filtered at 0°C, and the neutralization equivalent of the filtrate residue was determined. For chloride analysis, the heptane solution was reacted with anhydrous hydrogen chloride as before, then evaporated to dryness. Fresh heptane was added, and the evaporation repeated. Finally, the residue was warmed for 30 min in excess alcoholic potassium hydroxide. The alcohol was removed, water was added, and the chloride content was determined by the Volhard method.

Consistency Measurement

The technique developed for characterizing the consistency of the coating material by needle and cone penetration measurements is described in paragraph 4.1 of Appendix B of this report. Careful control of the evaporation process is necessary, since incomplete evaporation of the solvents or excessive evaporation of the oil will affect the penetrations. The rate of cooling of the molten sample must also be standardized, since the rate of cooling affects the structure of the solid and thereby its consistency.

Fog-Cabinet Corrosion Test

The rifle parts were placed on a glass plate on the rotating turntable in the fog cabinet.* The temperature was maintained at 120°F, and the parts were exposed to the distilled-water fog for 4 days.

Scratched-Coupon Corrosion Tests

Cold-rolled-steel (SAE 1010 or 1020) coupons, 1-1/4 by 2 by 1/16 in., were cleaned in solvents, then abraded with 320 Carborundum paper and grade 4-0 emery paper. The coating to be tested was applied to all surfaces of the panel and allowed to dry for 24 hr. A scratch was then made across the width of one side of the panel with a sharp-pointed scriber. The coating was allowed 2 hr to recover the scratch, then the coupon was hung on insulated wire in synthetic sea water conforming to ASTM Specification D-141-52 Formula A. After having been suspended in the sea water at room temperature for 3 days, the coupon was removed and examined for the presence of rust spots.

Corrosion Test Assemblies

Three assemblies of parts from the M16 rifle were used in studying the protection provided by the more promising coatings. Assembly A consisted of the barrel, front sight, one front-sight tapered pin, and the front swivel pin. Assembly B consisted of the bolt carrier, bolt, bolt cam pin, firing pin, firing-pin retaining pin, extractor, extractor spring, and extractor pin. Assembly C consisted of the lower receiver, trigger guard, automatic sear with spring and pivot tube, automatic sear pin, selector lever, selector-lever detent and spring, pistol grip, and pistol-grip screw-and-lock washer. These parts were of the same materials and surface finishes as the current submachine gun parts, except that the bolt carrier, bolt, and extractor were chrome plated while the currently produced ones are phosphate-conversion coated.

The parts were cleaned with solvents, and any rust from previous tests was removed. After coating and drying, the assemblies were immersed in synthetic sea water conforming to ASTM Specification D-141-52 Formula A in a pressure tank. A pressure of 95 to 100 psig (equivalent to that at a depth of over 200 ft in the sea) was applied for 30 min.

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*Federal Test Method, Standard 791, Method 5312.1.

The assemblies were then removed from the synthetic sea water, wrapped in a plastic bag to prevent evaporation of the water remaining on the parts, and kept at room temperature for 3 days. The parts were then examined for evidence of corrosion.

Corrosion Test of Entire Weapon

The corrosion tests of the coated M16 submachine guns were conducted in the same manner as the corrosion tests using the three assemblies of parts. The weapons were placed in the pressure tank, and totally submerged in synthetic sea water, with the muzzles up, chambers empty, bolts closed, and dummy cartridges in the magazines.

MATERIALS

The tetrapropenylsuccinic anhydride (alkenylsuccinic anhydride) was obtained from the Monsanto Chemical Company and Union Carbide Corporation. Other possible sources are Allied Chemicals, Archer Daniels Midland, and the Oronite Division of Chevron.

The arachidyl-behenyl primary amine was obtained from the Humko Products Chemical Division of the National Dairy Products Corporation as Kemamine P-190 and Kemamine P-190D. The P-190D (distilled grade) was found to be preferable for use in this application.

The military symbol 1010 oil is a light petroleum lubricant for aircraft turbine engines described by Specification MIL-L-6081C. Its viscosity is 10 cs minimum at 100°F and 3000 cs maximum at -40°F. Suppliers of this oil include Bray, Cities Service, Humble, MacMillan Ring Free, Royal, Sinclair, and Texaco.

The oxidation inhibitor 2,6-di-tert-butyl-4-methylphenol is available as Ionol from the Shell Chemical Corporation and as Parabar 441 from the Enjay Chemical Company.

The n-heptane was of commercial grade, with an approximate boiling range of 203 to 209°F. Absolute and 190-proof ethanol were used. Suitable denaturants are other alcohols, ketones, and esters. Petroleum solvents of wide boiling range may adversely affect the drying of the coating.

Appendix B

SUGGESTED TECHNICAL REQUIREMENTS FOR A MILITARY SPECIFICATION FOR PRESERVATIVE-LUBRICANT, TROPICAL (SMALL ARMS)

1. Scope

1.1 This specification covers a spray-applied coating material which provides lubrication and corrosion protection for M16 and other small arms in service where exposure to fresh or sea water is encountered.

2. Applicable Documents

2.1 The following documents of the issue in effect on the date of invitations for bids form a part of this specification to the extent specified herein: Book of ASTM Standards - 1967, Part 17 and Book of ASTM Standards - 1967, Part 18.

3. Requirements

3.1 Composition. The composition of the coating solution shall be as specified in Table B1.

Table B1
Composition of Preservative-Lubricant Solution

Component	Weight Percent	
Arachidyl-behenyl primary amine*	5.12	±0.70†
Tetrapropenylsuccinic anhydride	3.93	±0.80†
Lubricating oil, symbol 1010‡	10.80	±1.00†
Oxidation inhibitor§	0.15	±0.02
Ethanol¶	25.00	±3.00
n-Heptane**	55.00	±5.00

*Distilled grade.
†Adjust to obtain the proper consistency.
‡As specified in MIL-L-6081C.
§Hindered phenol type, such as 2,6-di-tert-butyl-4-methylphenol.
¶Maximum 2% water.
**Commercial grade.

3.2 Consistency. The needle and cone penetrations of the coating material shall be measured in accordance with 4.1. The needle penetration shall be no more than 160 and no less than 100. The cone penetration shall be no more than 90 and no less than 55.

3.3 Nonvolatile content. The nonvolatile portion of the solution shall be 20 ± 3.0% based on the weights of the solution and the nonvolatile residue determined in accordance with 4.1.

4. Test Methods

4.1 Consistency and nonvolatile content. The special equipment required consists of (a) a 50-ml erlenmeyer flask with ground-glass stopper, (b) a brass plate, as described in ASTM Test Method D 1321-65, (c) a cylindrical sample container of brass tubing (open at both ends) of inside diameter 3/4 in., wall thickness 1/8 in., and height 11/16 in., (d) a water bath controlled at 25 ± 0.1°C, (e) a penetrometer as described in ASTM Test Methods D 1321-65 and D 1403-62, (f) a needle and plunger of 100 g total weight as described in ASTM Test Method D 1321-65, and (g) a 1/4-scale cone and 1/4-scale grease worker as described in ASTM Test Method D 1403-62.

40 ± 0.5 ml of the coating solution from a bulk sample or sprayed from an aerosol can shall be placed in the tared 50-ml flask, and the weight determined to 0.01 g. The flask shall then be immersed to 1/3 of its height in boiling water. A flow of clean air at 2 l/min shall be directed onto the surface of the solution. After 60 min, the flask shall be removed from the boiling water, dried, and weighed. Oil condensed on the walls of the flask shall be returned to the sample by tilting and rotating the flask. The sample container shall be placed on a carefully flattened piece of aluminum foil on the brass plate. This assembly shall be preheated and placed in an oven at 60 ± 3°C along with the stoppered flask. After 30 min or more, the brass plate with the sample container shall be removed from the oven and placed on corks in a draft-free room at 25 ± 3°C. The molten sample shall be poured from the flask into the sample cup until a convex meniscus forms above the top of the sample container. The sample shall be left to cool undisturbed for 40 to 60 minutes. The material above the end of the sample container shall be cut off level with the end using a spatula; the container and sample shall be removed from the plate, the foil peeled off, and the container and sample replaced on the plate in the inverted position. The 1/4-scale cup, the sample container, and the plate are placed in the water bath at 25 ± 0.1°C, with the top of the sample container above the surface of the water. After 30 to 60 min, the needle penetration of the sample shall be measured once in the center of the container using the technique in paragraphs (c) and (d) of section 6 of ASTM Test Method D 1321-65. The sample shall then be pushed out of the sample container, any water drops on its surface shall be blotted off, and it shall be placed in the grease-worker cup. Working and measurement of the cone penetration shall be done as described under "Procedure for Worked Penetration" in ASTM Test Method D 1403-62. The sample shall then be removed from the worker to the flask. The flask, sample container, foil, and plate shall then be returned to the 60°C oven, and the procedure repeated from that point to obtain another pair of measurements. The results of the two runs shall be averaged to obtain the reported needle and cone penetrations. The data from the two runs should not differ by more than 10%.

5. Method of Preparation

5.1 The amounts of the amine, anhydride, and oil used must be adjusted, as noted in Table B1, to compensate for variations in the raw materials. Small samples should be made up, tested for consistency, and adjustments made before preparing large batches.

5.2 The following procedure has been found to be satisfactory for the preparation of the preservative-lubricant solution. Dissolve the anhydride and the amine in half of the alcohol by warming to about 70°C. Mix this solution into a mixture of the remaining ingredients. The pH of the finished solution should be between 6.5 and 7.0. The pH can be measured with a pH meter with a glass electrode and sleeve-type calomel reference electrode. The electrodes can be cleaned by rinsing with a mixture of alcohol and aliphatic naphtha or heptane.

CAUTION: The heptane and alcohol are very flammable solvents. The amine is alkaline and will irritate the skin. Precautions should be taken to avoid skin contact with this material. Once reacted with the anhydride, it is not irritating.

5.3 Packaging. The solution shall be packaged in pressurized aerosol cans. Nonflammable propellants shall be used. The nozzles shall produce a spray pattern approximately 2 in. wide at a distance of 12 in.

The following label information is recommended:

PRESERVATIVE-LUBRICANT, TROPICAL, FOR SMALL ARMS.

DIRECTIONS

Apply to all metal parts of partially stripped weapon. Operate moving parts before coating dries.

Nontoxic.

CAUTION: Flammable mixture. Do not spray near open flames or while smoking. Heat may burst the pressurized can. Do not expose the can to temperatures over 120°F.

Appendix C

USERS' INSTRUCTIONS

INSTRUCTIONS FOR THE USE OF NRL PRESERVATIVE-LUBRICANT ON THE M16 SUBMACHINE GUN

1. Disassembly

Remove and field-strip the bolt carrier group.
Separate the upper and lower receiver groups.
Upper receiver group: remove the hand guards.
Lower receiver group: remove action spring and spring guide, stock-locking lever screw, washer, locking lever, butt-plate screw, butt plate, and stock.

Magazine: depress the catch and slide the floor plate part way out.

Remove dirt and oil from all parts.

This procedure is the minimum recommended. More complete disassembly will permit more complete protection against corrosion. The parts most susceptible to corrosion are the front sight adjustment assembly, the extractor spring, and the stock-locking mechanism.

2. Coating

The coating may be sprayed on wet surfaces if necessary, since it will displace water.

Do not spray at temperatures below 40°F. Do not use on weapons to be used at temperatures below 0°F.

CAUTION: The coating contains flammable solvents. Do not smoke while spraying. Do not spray near open flames. This material is effective in very thin films. Apply only enough spray to fully wet all surfaces. Do not respray to build up coating.

Upper receiver group:

Spray - into the chamber until the bore is coated and the coating drips from the muzzle.
- into the flash hider and the silencer.
- into the receiver.
- on the entire exterior.

Operate all moving parts to distribute the coating while it is wet. Depress sight detents several times to work the coating material into the holes.

Lower receiver group:

Spray - into the interior, coating trigger mechanism parts thoroughly. Operate all parts several times before the coating dries.
- into the receiver extension. Depress the action-spring detent several times to work the coating into the hole.

- into the stock-locking mechanism. Depress the ball detent to work the coating into the hole.
- onto the remainder of the assembly, inside and out, moving all pins and catches.

Hand guards: spray both sides of the metal heat shields.

Stock: spray into both holes, onto metal parts.

Bolt carrier: spray inside and out, into all holes and gas passages.

Bolt: spray inside and out. Depress the ejector several times. Remove the extractor if possible and spray separately.

Magazine: spray inside and out.

Other parts: spray all surfaces: operate any moving parts.

Reassemble the weapon. preferably after allowing the coating to dry for about 2 hr.

No additional oil or grease is necessary for lubrication. Under dusty conditions or for relubrication, semifluid Lube Oil MIL-L-46000A may be added. (Stock 9150-889-3522).

After firing. lightly recoat the bore, chamber, and bolt-carrier group. Do not buildup a heavy coating on moving parts.

3. Cleaning

After immersion in sea water, the weapon should be thoroughly rinsed in fresh water at the first opportunity.

Where it is possible to disassembly the weapon, the parts should be rinsed in fresh water and dried. Spray again to cover areas where the coating has worn off.

The coating can be removed by scrubbing the parts in common solvent, such as dry-cleaning solvent (type 1), Varsol 1, Stoddard solvent, naphtha, mineral spirits, or diesel fuel.

A more effective solvent can be prepared by mixing 2 parts of any of these solvents with 1 part alcohol (ethyl alcohol or isopropyl alcohol). CAUTION: this mixture is more flammable than the solvent alone.

The coating can also be removed by 1,1,1-trichloroethane (methyl chloroform) in a vapor degreaser.

GENERAL INSTRUCTIONS FOR THE USE OF NRL PRESERVATIVE-LUBRICANT

1. Intended Use

This material was developed to provide corrosion protection and lubrication for small arms and other equipment exposed to salt or fresh water and jungle conditions.

2. Application

The equipment to be coated should be disassembled so that the spray can reach all parts.

Dirt, grease, and oil should be removed before coating.

Spray the metal parts so that all surfaces are coated.

Operate moving parts to work the coating solution into the crevices between the parts.

Allow to dry for about 2 hr.

CAUTION: The coating solution contains flammable solvents. Do not smoke while spraying. Do not spray near open flames.

3. Lubrication

Although the coating is intended primarily for corrosion prevention, it will also provide sufficient lubrication for most equipment.

Additional lubricants may be applied if necessary.

Do not apply this coating on equipment which will be required to operate at temperatures below 0°F; do not spray at temperatures below 40°F.

The coating will melt at temperatures above 130°F but will continue to provide corrosion protection and lubrication.

4. Cleaning

After being exposed to salt water, the equipment should be rinsed thoroughly in fresh water and dried. Spray again to cover areas where the coating has worn off.

The coating can be removed by scrubbing the parts in common solvents, such as dry-cleaning solvent (type 1); Varsol 1, Stoddard solvent, naphtha, mineral spirits, or diesel fuel.

A more effective solvent can be prepared by mixing 2 parts of any of these solvents with 1 part alcohol (ethyl alcohol or isopropyl alcohol). CAUTION: this mixture is more flammable than the solvent alone.

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13. ABSTRACT

A protective and lubricating coating has been developed for use on the M16 submachine gun and other small arms. It protects the M16 submachine gun used by SEAL and UDT personnel during immersion in sea water and exposure to sand, dust, and jungle environment. Treated weapons are ready for immediate use. The preservative-lubricant coating which was developed for this application is a compound of arachidyl-behenyl amine and tetrapropenylsuccinic anhydride, with an oil and a solvent. It is packaged in aerosol cans for field use. The spray penetrates and displaces water from wet surfaces. The dried coating is firm and nontacky but breaks down to a greaselike consistency between moving parts. The consistency of the coating material can be completely controlled by adjustment of the formulation. It is compatible with other lubricants. The coating provides excellent corrosion protection, because the entire solid phase consists of a waxy polar inhibitor. The oil phase, carrying the inhibitor in solution, can creep out to reprotect scratches and worn areas in the coating. Metal specimens and complete weapons were protected from damage during exposure to sea water for 3 days.

Coatings of this type offer promise as preservatives for exposed parts of operating machinery, spare parts, tools, etc., where lubricant compatibility or operation without depreservation is desirable.

Security Classification

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Preservatives						
Lubricants						
Small-arms lubricants						
Corrosion inhibitors						
Amine-acid compounds						
M16 submachine gun						
Weapons lubricants						

Security Classification