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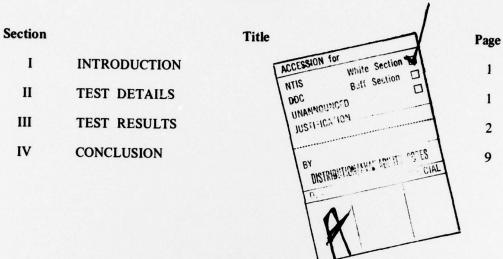
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DEVELOPMENT OF AN ANTIFREEZE EXTENDER AND WATER INHIBITOR FOR AUTOMOTIVE COOLING SYSTEMS

I. INTRODUCTION

A shortage of ethylene glycol for antifreeze usage was brought about as a result of the 1973 energy crisis. The feedstock (petroleum naphtha) used for the manufacture of ethylene glycol was diverted to more lucrative fields, particularly automotive gasoline, both leaded and unleaded. Instead of being cracked to ethylene to manufacture ethylene glycol, a large portion of the naphtha was used directly for formulating gasolines and other solvents that were in short supply. Additionally the soaring demand for ethylene glycol for use in polyester fiber and film further reduced the availability for antifreeze.

Because of the increasing concern to improve energy conservation and the desirability to extend the service life of engine antifreezes, a program was initiated to determine the feasibility of reclaiming or reusing antifreeze. The results of this study are reported in MERADCOM Report 2168¹ and show that it is possible to reclaim used antifreeze and restore it to a usable condition. Four commercial materials were evaluated; three were eliminated because of corrosion of the metal specimens and excessive foaming in the glassware corrosion test ASTM Method D1384.² The one commercial inhibitor found to be satisfactory was tested further in the ASTM-D-2570 simulated service test³ and was found to meet our initial requirements. But since this was a proprietary material and the company did not plan to market it, a program was initiated to formulate an additive package having similar or superior characteristics.

II. TEST DETAILS

Initially the satisfactory commercial material tested previously was analyzed to identify and/or characterize the components. Total solids, infrared spectrophotometry, X-ray diffraction studies, and atomic absorption spectrophotometry were conducted.

Eighteen formulations were compounded based upon the above analysis. Of these eighteen blends, Blends 17 and 18 were further tested in the laboratory.

¹ James H. Conley and Robert G. Jamison, "Reclaiming Used Antifreeze," MERADCOM Report 2168, March 1976.

² ASTM D-1384, "Corrosion Tests for Engine Coolants in Glassware."

³ ASTM D-2570, "Simulated Service Corrosion Testing of Engine Coolants."

Laboratory screening tests were conducted in two phases. The first phase consists of testing in accordance with ASTM Method D-1384, "Corrosion Test for Engine Coolants in Glassware." This method covers a simple beaker-type screening test for evaluating the effects of engine coolants on metal specimens under controlled laboratory conditions. Specimens of metals typical of those present in automotive cooling systems are totally immersed in the test antifreeze solution with aeration for 336 hours at 87.8°C (190°F). The corrosion inhibitive properties of the test solution are evaluated on the basis of the weight changes incurred by the specimens. Each test is run in triplicate and the average weight change is determined for each metal. This method will generally distinguish between coolants that are definitely deleterious from the corrosion standpoint and those that are suitable for further evaluation.

The second phase of the laboratory screening tests was conducted according to ASTM Method D-2570, "Simulated Service Corrosion Testing of Engine Coolants." This method evaluates the effect of a circulating engine coolant on metal test specimens and automotive cooling system components under controlled laboratory conditions. The method specifies cooling system components, type of coolant, and coolant flow conditions that are considered typical of current automotive use. The test temperature specified in this method is $87.8^{\circ}C$ ($190^{\circ}F$), but for the purposes of this study tests were also conducted at $115.5^{\circ}C$ ($240^{\circ}F$) in order to study the cavitation corrosion effect on an aluminum water pump. This test is significant if the results include comparable test information on a coolant of known service performance characteristics such as MIL-A-46153 antifreeze⁴ and O-I-490 corrosion inhibitor⁵ in water.

III. TEST RESULTS

Analysis of a commercial reinhibitor is shown in Table 1. Results show this material is essentially a water solution containing approximately 30 percent by weight solids. Elements identified by infrared, X-ray diffraction, and atomic absorption include boron, sodium, silicon, traces of potassium, calcium, and an unidentified organic antioxidant.

Eighteen formulations were compounded using the analysis in Table 1 as the guide. Problems with solubility of various silicates and organic antioxidants eliminated 16 of the formulations. The two formulations listed in Table 2 were further studied in the laboratory corrosion tests.

⁴ Military Specification MIL-A-46153, "Antifreeze, Ethylene Glycol, Inhibited, Heavy-Duty, Single-Package," 14 August 1973.

⁵ Federal Specification 0-1-490, "Inhibitor Corrosion, Liquid Cooling System," 26 April 1965.

No.	Parameter	Content
1	Total Solids	29.5% by weight.
2	Residue by Infrared	Water solution of borates and silicates.
3	X-ray Diffraction	Silicates with traces of phosphorus. Chloride and iron.
4	Atomic Absorption	2.9% boron; 5.3% sodium; 0.6% silicon with traces of potassium and calcium.
5	Infrared of Water Extraction	Organic Antioxidant, not identified.
6	Concentrated pH	12.7.
7	Reserve Alkalinity	3% solution 8.5.
8	Specific Gravity	1.230.

Table 2. Experimental Inhibitor Formulations

Blend 17

29%	by weight Sodium Metaborate.						
3%	by weight Sodium Mercaptobenzothiazole (50% solution).						
4.6%	4.6% by weight Sodium Silicate.						
63.4%	by weight Distilled Water.						
	Concentrated pH 12.25.						
	3% Reserve Alkalinity 9.8.						
	Specific Gravity 1.193.						
	Blend 18						
29%	by weight Sodium Metaborate.						
3%	by weight Sodium Mercaptobenzothiazole (50% solution).						
4.6%	by weight Potassium Silicate.						

63.4% by weight Distilled Water.

Concentrated pH 12.5.

3% Reserve Alkalinity 11.8.

Specific Gravity 1.192.

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Table 3 shows the results of tests conducted using ASTM Method D-1384. Test No. 6 clearly shows that Blend 18 is superior to Blend 17 in used antifreeze (Test No. 2). Test No. 6 also compares favorably to Test No. 7 which is conducted on new MIL-A-46153 antifreeze. Test No. 5 shows there is no adverse affect when a 3-percent concentration of Blend 18 is added to new MIL-A-46153 antifreeze. Test No. 4 shows that Blend 18 can also be used to reduce corrosion effectively in corrosive water. Foaming tendency of both blends was checked and neither showed an increase in foam formation.

Results of testing according to ASTM-D-2570 are shown in Table 4. Blend 17 produced less corrosion on the solder specimen than did Blend 18 when used at a 3-percent concentration in corrosive water. But during testing the packaged material was found to be unstable. After a storage period of only one month, the material began to precipitate out and testing was discontinued on Blend 17. Although Blend 18 gave higher solder weight losses during testing, there was no adverse affect on the solder in the radiator. All other metals were satisfactory. Blend 18 is effective in reducing corrosion of all metals in used antifreeze and does not adversely affect new antifreeze. Blend 18 was especially effective in corrosive water at 115.5°C (240°F) when compared to 0-I-490 inhibited corrosive water at the same temperature.

Figure 1 shows the cavitation corrosion on the aluminum water pump used in Test No. 7 with 0-I-490 as the corrosion inhibitor, and Figure 2 shows the water pump from Test No. 6 using Blend 18 as the corrosion inhibitor. There is no clear evidence of corrosion of any kind on the pump from the test using Blend 18. In fact, the pump is as good as new. The use of Blend 18 in the cooling system of an aluminum block engine operating at temperatures at $115.5^{\circ}C$ (240°F) with water is a significant improvement over the presently used 0-I-490 inhibitor system.

A field test has been initiated in military vehicles, both facility vehicles and tactical vehicles to confirm the results of the laboratory testing of Blend 18 and will continue for at least one year. Vehicles were checked and those vehicles that were marginally inhibited were used for the field test. Blend 18 was also added to two vehicles with adequate coolant.

A patent covering Blend 18 is being applied for through the MERADCOM Office of Chief Counsel patent attorney.

A further plan has been developed to use this technology in the development of a long-life cooling system. The inhibitor system will be incorporated into a replaceable canister/filter-type conditioner that will require periodic replacement and will allow continued use of the antifreeze, thus reducing the demand for antifreeze replacement.

		Weight Loss per Specimen (mg)					
	Solution	Copper	Solder	Brass	Steel	Cast Iron	Aluminum
1.	3% Blend 17 in corrosive water	8.6	21.4	11.1	2.3	Nil	0.6ª
2.	3% Blend 17 in used antifreeze	14.3	49.9	11.6	181.6 ^b	204.1°	6.3
3.	3% Commercial Reinhibitor in used antifreeze	7.5	33.8	8.0	2.8	250.9°	3.4
4.	3% Blend 18 in corrosive water	8.2	18.6	9.2	2.1	3.7	8.5 ^d
5.	3% Blend 18 in new MIL-A-46153 antifreeze	8.2	21.0	9.0	1.7	2.2	1.1
6.	3% Blend 18 in used antifreeze	10.7	16.9	8.8	5.3	1.7	Gain
7.	New MIL-A-46153 antifreeze	7.9	17.3	8.4	0.5	+1.8	5.9
Ma lim	x. weight loss hit	10.0	30.0	10.0	10.0	10.0	30.0

^a Aluminum specimens pitted.

b Heavy etching at corner of specimen.

c Heavy etching overall.

d Slight etching.

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	Test Temp	Test Duration		We	Weight Loss per Specimen (mg)	per Speci	men (mg)	
Solution	(°F)	(hr)	Copper	Solder	Brass	Steel	Cast Iron	Aluminum
1. 3% Blend 17 in corrosive water	061	1064	23.2	41.8	15.5	2.9	1.2	54.2ª
2. 3% Blend 18 in corrosive water	190	1064	15.3	208.3	15.1	2.8	Gain	29.5
3. 3% Blend 18 in new MIL-A-46153 antifreeze	190	1064	8.1	20.9	9.2	1.1	1.0	4.4
4. 3% Blend 18 in used antifreeze	190	1064	6.6	270.1	12.0	15.0	2.8	421.0
5. 1.5% 0-I-490B in corrosive water	190	1064	2.1	170.8	5.2	1.2	+1.6	620.9
6. 3% Blend 18 in corrosive water	240	700	+22.13b	225.6	+3.7b	2.7	+4.9	14.5
7. 1.5% 0-I-490B in corrosive water	240	555	No specin	No specimens were used in this test	tsed in this	i test		
Max. weight loss limit			20	300	20	20	20	400
 Moderate etching. b Specimens tin plated. 								

Table 4. ASTM D-2570 Simulated Service Test Results

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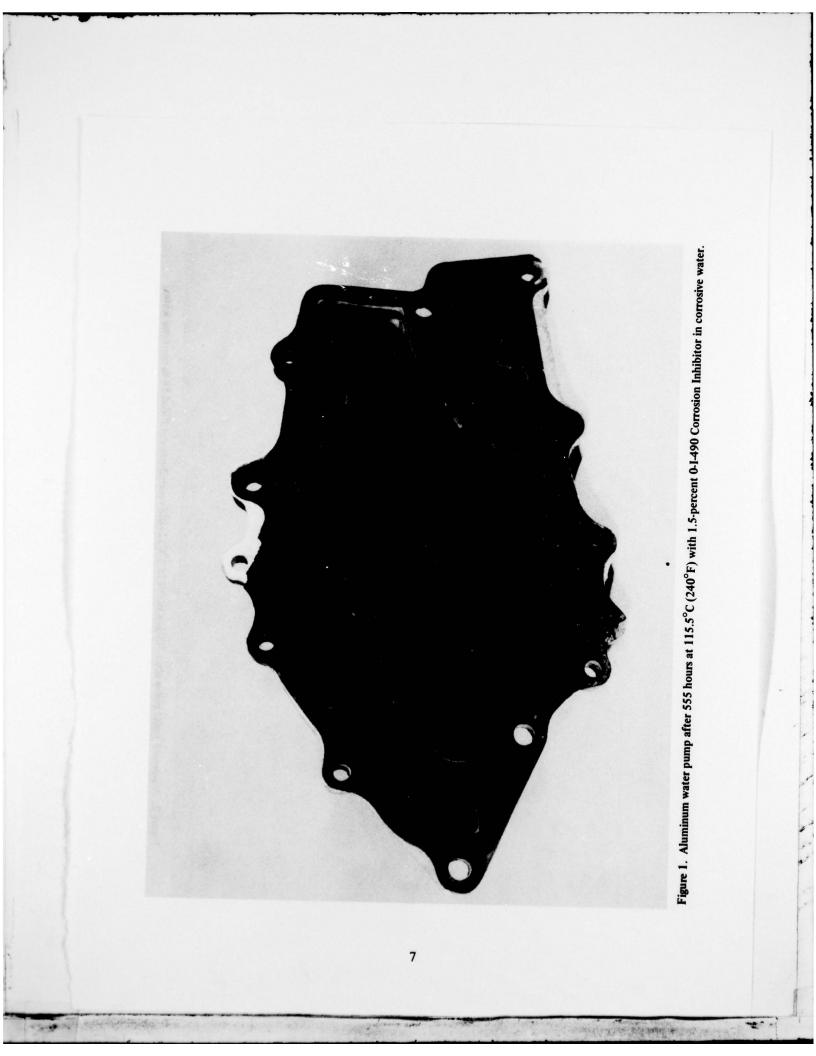
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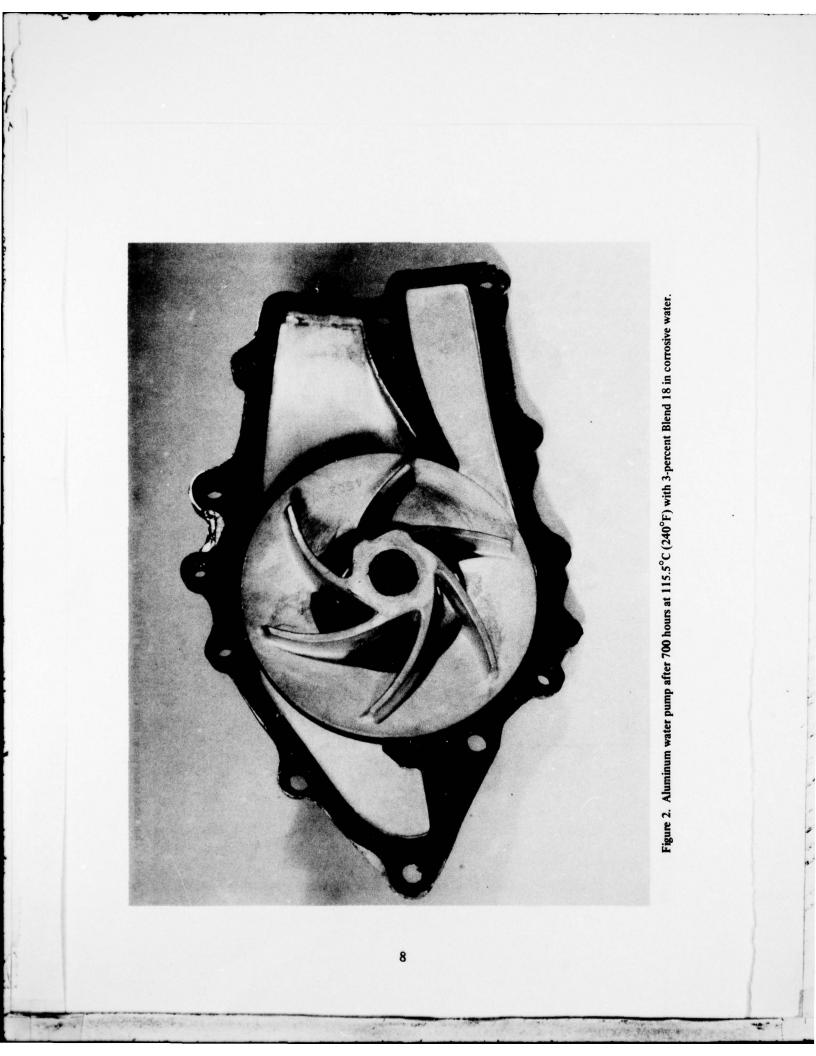
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IV. CONCLUSION

Blend 18 is an effective inhibitor for use in returning used antifreeze to an acceptable level of corrosion protection to allow one year's additional use. It is an excellent replacement for the presently used 0-I-490 inhibitor in systems that require only water as the coolant. It is especially useful in military vehicles containing aluminum components that often operate at temperatures well above the $87.8^{\circ}C$ ($190^{\circ}F$) temperature due mostly to the unusual operating conditions.

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