CCL REPORT NO. 206

PROGRESS REPORT

INVESTIGATION OF ORGANIC PHOSPHATES AS CORROSION INHIBITORS FOR COOLANTS

BY

JAMES H. CONLEY

JUNE 1966

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U. S. ARMY COATING & CHEMICAL LABORATORY

Aberdeen Proving Ground
Maryland

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INVESTIGATION OF ORGANIC PHOSPHATES AS CORROSION INHIBITORS FOR COOLANTS

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JAMES H. CONLEY

JUNE 1966

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U.S. ARMY COATING AND CHEMICAL LABORATORY
ABERDEEN PROVING GROUND
MARYLAND

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ABSTRACT

The object of this study was to investigate the use of organic phosphates as corrosion inhibitors in coolant formulations. Glassware bench corrosion tests and simulated service tests were conducted on various organic acid phosphates in combination with other inhibitors such as sodium tetraborate (borax), sodium metaborate and sodium mercaptobenzothiazole (NACAP). Test data showed that these phosphates when exposed to hard water form non-crystalline precipitates that offer no deleterious effect to cooling systems. Test data also showed that these phosphates are effective corrosion inhibitors when used with a sodium borate buffer.

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I. INTRODUCTION

Aberdeen Proving Ground, Maryland was directed by AMC program directive AMCMS Code 5025.11.803 dated 3 September 1965 to investigate improved antifreeze mixtures.

Data in CCL Reports No. 125, 145, 155, 156 and 190 showed that the addition of an inorganic phosphate increased the inhibitor effectiveness, especially in a system containing cast iron and aluminum components. Even though the inorganic phosphate is an excellent inhibitor a crystalline precipitate of insoluble calcium phosphate often forms in the cooling system in the presence of hard water. The organic acid phosphates were investigated as replacements for the inorganic phosphates since the manufacturers had claimedthese phosphates formed non-crystalline precipitates or no precipitates at all with hard waters.

This report contains the results of tests utilizing ethylene glycol base antifreeze compounds inhibited with organic acid phosphates in combination with sodium tetraborate (borax), sodium metaborate, and sodium mercaptobenzothiazole (NACAP).

II. DETAILS OF TEST

A. Glassware Bench Corrosion Test

Bench corrosion tests were conducted in accordance with the procedure outlined in LSD Report No. 205. This procedure involves the immersion of a set of six galvanically coupled metal test specimens (cast iron, aluminum, copper, solder, brass, steel) in a glass flask containing the test solution. The solution is aerated and refluxed at 180°F. for 192 hours, after which the metal test specimens are examined for evidence and extent of corrosion.

B. Simulated Service Test

This test involves variations of the test outlined in LSD Report No. 205. The test consists of mechanical units arranged to permit the test solution to be circulated in a closed system at controlled circulation rate and temperature.

In this study the unit contained an aluminum or cast iron reservoir and aluminum or brass radiator. The pump was driven by an electric motor. The test temperature was maintained at 180°F. ± 5°F.

Metal test specimens as described in ASTM Method D1384 were arranged on a bracket and suspended in the reservoirs. After 2000 hours of operation or upon failure of the radiator the test was terminated and the metal specimens examined for evidence and extent of corrosion.

C. Precipitation Test

A synthetic hard water made up of 1.5270 gms $Ca(C_2H_3O_2)_2 \cdot H_2O$ + 1.0496 gms $MgSO_4 \cdot 7\ H_2O$ diluted to 1 gallon with distilled water. The resultant "hard water" has an equivalent hardness of 20 grains per gallon or 342 ppm as $CaCO_3$.

Two antifreeze solutions containing inorganic phosphates and three antifreeze compounds containing organic acid phosphates were diluted with the "hard water" and the volume of precipitate recorded. 100 ml. graduated centrifuge tubes were used to determine the precipitate volume.

D. Test Solutions

Solutions used in the glassware bench corrosion tests included water and/or ethylene glycol inhibited with ethyl acid phosphate, tri lauryl phosphate, octyl phenyl acid phosphate, phenyl acid phosphate, iso octyl acid phosphate, the sodium salt of octyl phenyl acid phosphate, or the sodium salt of iso octyl acid phosphate. The minimum acid numbers of the organic acid phosphates range from 165 to 565.

Solutions used in the simulated service test included ethylene glycol-water mixtures inhibited with iso octyl acid phosphate in combination with sodium tetraborate (borax), sodium metaborate, and sodium mercoptobenzothiazole (NACAP).

Solutions used in the precipitation tests included Federal Specification O-A-548a, Antifreeze, Type I with disodium phosphate added, ethylene glycol-borax-NACAP with iso octyl acid phosphate or di-2-ethyl hexyl phosphoric acid, and a commercial antifreeze containing inorganic phosphate. Each solution was prepared with "hard water" containing 342 ppm as $CaCO_3$.

E. Numerical Rating System

A numerical rating system has been devised (see table V) which allows a comparison of test results based on weight loss and visual evidence of corrosion of the metal test specimens. An arbitrary value of 21 was selected as the point of demarkation between satisfactory and unsatisfactory results in the bench corrosion test and a value of 35 was chosen as the point of demarkation in the simulated service test. The arbitrary values were derived from results of glassware tests, simulated service tests, and field tests on various antifreeze formulations. The majority of the coolants tested that have a rating of 21 or less in the bench test and 35 or less in the simulated service test are expected to be satisfactory in the field.

III. RESULTS OF TESTS

Results of tests are listed in Tables II thru IV of the Appendix. Data listed in Table II shows that in glassware bench corrosion tests phenyl acid phosphate, octyl phenyl acid phosphate and iso octyl acid phosphate are effective corrosion inhibitors offering increased protection to aluminum. The first five tests were preliminary screening tests conducted to decide which of the organic acid phosphates had promise as inhibitors for aluminum. The minimum acid numbers of the organic acid phosphates tested had a range from 165 to 565. The three organic acid phosphates that performed best had minimum acid numbers that ranged from 200 to 345. The sodium salts of iso octyl acid phosphate and octyl phenyl acid phosphate were prepared in the laboratory and were found to be adequate inhibitors as shown in Tests Nos. 6 thru 11. The organic acid phosphates offer protection to aluminum even when diluted with ASTM corrosive water containing 100 ppm of Cl^-, $SO_4^=$ and HCO_3^- (Tests 16 and 17). Data shows that an antifreeze formulation with iso octyl acid phosphate buffered with borax is equally effective in a 30% and 50% solution while an antifreeze formulation with iso octyl acid phosphate buffered with sodium metaborate is more effective in a 50% solution than in a 30% solution (Tests Nos. 12 thru 15). Test results of the coolant that is presently in use in government vehicles are listed in Tests Nos. 18 and 19.

Simulated service test data listed in Table III of the Appendix shows that iso octyl acid phosphate in both the low pH range (borax buffered) and the high pH range (sodium metaborate buffered) is an effective inhibitor for aluminum although it is more effective in the low pH range. Tests Nos. 2 and 3 developed pump difficulty which could not be attributed to a faulty pump. Test No. 5 developed pump trouble early in the test which was attributed to a faulty pump.

Table IV shows that precipitation of inhibitors containing phosphates with "hard water" is reduced tenfold by replacing the inorganic phosphate with an organic acid phosphate. Iso octyl acid phosphate from two sources was used. Both showed 1% precipitate by volume and both were white soapy solids. In one the precipitate settled to the bottom and in the other it rose to the top.

The solution of Federal Specification 0-A-548a Antifreeze, Type I with 0.12% disodium phosphate had 10% precipitate by volume. This was ten times the volume of precipitate from either of the two formulations containing iso octyl acid phosphate and from the formulation containing di-2-ethyl hexyl phosphoric acid. The commercial antifreeze is a 50% glycol/water solution as packaged and contains an inorganic phosphate. This formulation was diluted to 30% with the "hard water" and showed 3% (by volume) crystalline precipitate.

IV. DISCUSSION AND CONCLUSIONS

Previous studies showed that corrosion may be experienced in a cast iron-aluminum system after extended use and that the addition of disodium phosphate would provide improved protection to the aluminum components without imparting serious corrosion problems to the other cooling system metals. Tests conducted during this investigation show organic phosphates with minimum acid numbers in the range of 200 to 345 are equally as efficient as the disodium phosphate.

There has been some objections to the use of disodium phosphate in a system exposed to hard water due to the formation of crystalline precipitates. This would be eliminated by using an organic acid phosphate. The organic phosphate inhibited coolants in this study when exposed to "hard water" formed precipitates of finely dispersed soapy materials that measured one tenth that formed from the inorganic phosphate inhibited coolant.

V. RECOMMENDATIONS

It is recommended that the use of organic phosphates as inhibitors for coolants be investigated in the field since they overcome a major objection found in the use of inorganic phosphate inhibitors.

VI. REFERENCES

1. Authority: AMC Program Directive, AMCMS Code 5025.11.803 dated 3 September 1965.
2. Federal Specification O-A-548a, Antifreeze, Ethylene Glycol, Inhibited, dated 30 December 1958.
3. Laboratory Service Division Report No. 205 - Development of a Suitable Laboratory Bench Corrosion Test for Antifreeze Compounds and Inhibitors, dated 26 February 1954.
4. CCL Report No. 125 - Compatability of Coolants with Automotive Cooling Systems Containing Aluminum Components 1st Report, dated 21 June 1962.
5. CCL Report No. 145 - Final Report on Compatability of Coolants with Automotive Cooling Systems Containing Aluminum Components dated 14 June 1963.
6. CCL Report No. 155 - New Corrosion Inhibitors for Antifreeze, dated 16 January 1964.
7. CCL Report No. 156 - The Development of an Improved Cooling System Corrosion Inhibitor, dated 10 February 1964.
8. CCL Report No. 190 - Extended Use of Improved Cooling System Inhibitor - Field Evaluation - Interim Report, dated 3 December 1965.
9. ASTM Method D1384 Standard Method of Test for Engine Antifreeze-Glassware Corrosion Test.

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APPENDIX A

TABLE I

COOLANT SOLUTIONS FOR BENCH TESTS

A - 50.0% Ethylene Glycol, 47.75% Water, 2.00% Borax, 0.20% NACAP, 0.05% Ethyl Acid Phosphate

B - 50.00% Ethylene Glycol, 47.72% Water, 2.00% Borax, 0.20% NACAP, 0.08% Phenyl Acid Phosphate

C - 50.0% Ethylene Glycol, 47.60% Water, 2.00% Borax, 0.20% NACAP, 0.20% Iso Octyl Acid Phosphate

D - 50.00% Ethylene Glycol, 47.90% Water, 2.00% Borax, 0.10% Iso Octyl Acid Phosphate

E - 47.80% Ethylene Glycol, 49.40% Water, 2.50% Borax Condensate, 0.20% NACAP, 0.10% Iso Octyl Acid Phosphate

F - 100% distilled water + 1.0% Borax, 0.2% MBT, 0.12% Sodium Iso Octyl Phosphate

G - 100% distilled water + 1.0% Borax, 0.2% MBT, 0.12% Sodium Octyl Phenyl Phosphate

H - 50% OA548a antifreeze, water contains 50 gms/gal - 75.7% Borax, 15.14% MBT, 9.16% Sodium Iso Octyl Phosphate

I - 30% OA548a Antifreeze, Water contains 50 gms/gal - 75.7% Borax, 15.14% MBT, 9.16% Sodium Iso Octyl Phosphate

J - 50% O-A-548a Antifreeze, Water contains 50 gms/gal - 75.7% Borax, 15.14% MBT, 9.16% Sodium Octyl Phenyl Phosphate

K - 30% O-A-548a Antifreeze, Water contains 50 gms/gal - 75.7% Borax, 15.14% MBT, 9.16% Sodium Octyl Phenyl Phosphate

L - 90.7% Ethylene Glycol, 5.0% Water, 4.0% Borax, 0.3% Iso Octyl Acid Phosphate diluted to 50% with distilled water

M - 90.7% Ethylene Glycol, 5.0% Water, 4.0% Borax, 0.3% Iso Octyl Acid Phosphate diluted to 30% with distilled water

N - 93.0% Ethylene Glycol, 3.5% distilled water, 3.0% Sodium Metaborate, 0.3% Iso Octyl Acid Phosphate, 0.2% NACAP diluted to 30% with distilled water

TABLE I - Cont'd

COOLANT SOLUTIONS FOR BENCH TESTS

O	-	93.0% Ethylene Glycol, 3.5% Water, 3.0% Sodium Metaborate, 0.2% NACAP, 0.3% Iso Octyl Acid Phosphate diluted to 50% with distilled water
P	-	Same as "N" except ASTM corrosive water used for dilution
Q	-	Same as "O" except ASTM corrosive water used for dilution
R	-	30% OA548a Antifreeze, Water contains 48.6 gms/gal 75.7% Borax, 15.14% MBT, 9.16% Na_2HPO_4
S	-	50% OA548a Antifreeze, Water contains 48.6 gms/gal - 75.7% Borax, 15.14% MBT, 9.16% Na_2HPO_4

TABLE II

GLASSWARE BENCH CORROSION TESTS

Test No.	1	2	3	4	5
Coolant	A	B	C	D	E
pH Before	7.50	7.40	7.48	7.40	7.42
pH After	7.50	7.40	7.30	7.30	7.30
R.A. Before	11.20	11.20	11.00	10.80	3.30
R.A. After	10.90	10.90	10.80	10.20	3.30
Visual inspection and wt. change in mg/sq. cm.					
Aluminum	H. Black Coating	Black	O.K.	O.K.	O.K.
Copper	Stained	Stained	V.Sl.Stain	Sl. Stain	V.Sl.Stain
Solder	O.K.	O.K.	O.K.	O.K.	O.K.
Brass	Sl.Coating	Sl.Stain	V.Sl.Stain	Sl.Stain	V.Sl.Stain
Steel	O.K.	O.K.	O.K.	O.K.	O.K.
Cast Iron	O.K.	Sl.Stain	O.K.	O.K.	O.K.
CCL Rating	--	--	--	--	--

Remarks: Preliminary tests conducted without weighing the strips.

TABLE II - Cont'd.

GLASSWARE BENCH CORROSION TESTS

Test No.	6		7		8		9	
Coolant	F		G		H		I	
pH Before	8.90		8.85		7.48		7.82	
pH After	8.88		8.75		7.48		7.70	
R.A. Before	5.50		5.50		10.60		8.70	
R.A. After	5.40		5.50		10.60		8.30	
Visual Inspection and Wt. Change in Mg/sq cm								
Aluminum	Yellow	-3.33	Yellow	-3.38	O.K.	-0.05	Greenish Black	-0.81
Copper	Sl.Stain	+0.06	Mod.Stain	+0.11	V.Sl.Stain	0	Tinned & Stained	+0.05
Solder	Stained	-0.08	Stained	-0.04	O.K.	-0.02	Sl.Stain	-0.04
Brass	V.Sl.Stain	+0.09	V.Sl.Stain	+0.15	V.Sl.Stain	-0.01	Tinned,Sl. Stain	+0.07
Steel	Sl.Stain	+0.07	Sl.Stain	+0.06	O.K.	0	Sl.Stain	+0.01
Cast Iron	Sl.Stain	+0.11	Sl.Stain	+0.14	O.K.	-0.01	Sl.Stain	+0.02
CCL Rating		19		19		8		17

TABLE II - Cont'd.

GLASSWARE BENCH CORROSION TESTS

Test No.	10		11		12		13	
Coolant	J		K		L		M	
pH Before	7.52		7.80		7.45		7.82	
pH After	7.52		7.70		7.43		7.82	
R.A. Before	10.60		8.70		11.50		6.90	
R.A. After	10.30		8.40		11.35		6.90	
Visual Inspection and Wt. Change in Mg/sq cm								
Aluminum	Grey	-0.15	Black	-0.22	O.K.	-0.01	O.K.	-0.01
Copper	Sl.Stain	+0.01	V.Sl.Stain	+0.02	Mod.Stain	-0.15	Sl.Stain	-0.02
Solder	O.K.	-0.02	O.K.	-0.01	V.Sl.Stain	+0.02	O.K.	-0.35
Brass	V.Sl.Stain	+0.02	V.Sl.Stain	+0.01	Mod. Stain	-0.03	Stained	0
Steel	O.K.	-0.01	O.K.	0	Mod. Stain	0	O.K.	+0.02
Cast Iron	O.K.	0	O.K.	-0.08	Mod. Stain	-0.22	O.K.	-0.11
CCL Rating		10		11		14		12

TABLE II - Cont'd.

GLASSWARE BENCH CORROSION TESTS

Test No.	14		15		16		17	
Coolant	N		O		P		Q	
pH Before	8.88		8.58		8.89		8.53	
pH After	8.78		8.52		8.89		8.53	
R.A. Before	7.50		12.30		7.50		12.35	
R.A. After	7.30		12.10		7.45		12.10	
Visual Inspection and Wt. Change in Mg/sq cm								
Aluminum	Greenish Black	-0.77	Sl.Stain	-0.02	Golden Light Pitting	-0.67	Black	-0.41
Copper	Mod.Stain Tinned	+0.09	St.Stain	+0.01	Sl.Stain	+0.01	Sl.Stain	-0.06
Solder	Mod.Stain	-0.45	O.K.	-0.02	Mod.Stain	-1.35	V.Sl.Stain	-0.38
Brass	V.Sl.Stain	+0.12	V.Sl.Stain	+0.02	Sl.Stain	-0.02	V.Sl.Stain	-0.06
Steel	O.K.	+0.03	O.K.	0	Sl.Stain	+0.04	O.K.	+0.01
Cast Iron	V.Sl.Stain	+0.07	O.K.	+0.01	Sl.Stain	+0.05	O.K.	-0.01
CCL Rating		20		9		23		17

TABLE II - Cont'd.

GLASSWARE BENCH CORROSION TESTS

Test No.	18		19	
Coolant	R		S	
pH Before	7.78		7.50	
pH After	7.78		7.50	
R.A. Before	9.00		10.70	
R.A. After	8.70		10.50	
Visual Inspection and Wt. Change in Mg, sq cm				
Aluminum	Dark Grey	-0.15	Light Grey	-0.06
Copper	V.Sl.Stain	0	V. Sl. Stain	-0.01
Solder	O.K.	-0.08	O.K.	-0.02
Brass	V.Sl.Stain	+0.02	V.Sl.Stain	0
Steel	O.K.	-0.03	O.K.	-0.02
Cast Iron	O.K.	-0.07	O.K.	-0.03
CCL Rating		10		9

TABLE III

SIMULATED SERVICE TESTS

Metal Components	Cast Iron Reservoir-Brass Radiator		Cast Iron Reservoir-Brass Radiator		Cast Iron Reservoir-Brass Radiator	
Test No.	1		2		3	
Coolant Solution	90.7% Ethylene Glycol 5.0% Water 4.0% Borax 0.3% Iso Octyl Acid Phosphate Diluted to 50% with distilled water		93.0% Ethylene Glycol 3.5% Water 3.0% Sodium Metaborate 0.2% NACAP 0.3% Iso Octyl Acid Phosphate Diluted to 50% with distilled water		93.0% Ethylene Glycol 3.5% Water 3.0% Sodium Metaborate 0.2% NACAP 0.3% Iso Octyl Acid Phosphate Diluted to 30% with distilled water	
Total Hrs. of Operation	2003		1655*		2002*	
pH Before	7.43		8.58		8.88	
pH After	6.93		8.35		8.68	
RA Before	11.60		12.30		7.50	
RA After	7.50		10.90		8.20	
Visual Inspection and Wt. Change mg./sq.cm.						
Aluminum	Light Grey	-0.01	Green-Pitted	-3.12	Golden	-1.71
Copper	H. Stain	-8.33	H. Stain	+0.15	Mod. Stain	+0.04
Solder	O.K.	+0.01	Sl.Stain	-0.92	Mod. Stain	-4.44
Brass	H.Stain	-0.01	Sl.-Mod.Stain	-0.02	Sl. Stain	+0.05
Steel	V.Sl.Stain	-0.11	V.Sl.Stain	+0.01	Sl.Stain	+0.02
Cast Iron	Sl.Stain	-0.03	V.Sl.Stain	+0.07	Sl.Stain	+0.07
CCL Rating		20		24		25
*Remarks			Test stopped at 1655 hrs. Solution leaked badly at the pump.		Pump replaced at 1209 hrs.	

TABLE III - Cont'd.

SIMULATED SERVICE TESTS

Metal Components	Cast Iron Reservoir-Aluminum Radiator		Aluminum Reservoir-Brass Radiator		Aluminum Reservoir-Aluminum Radiator	
Test No.	4		5		6	
Coolant Solution	90.7% Ethylene Glycol 5.0% Water 4.0% Borax 0.3% Iso Octyl Acid Phosphate Diluted to 50% with distilled water		90.7% Ethylene Glycol 5.0% Water 4.0% Borax 0.3% Iso Octyl Acid Phosphate Diluted to 50% with distilled water		90.7% Ethylene Glycol 5.0% Water 4.0% Borax 0.3% Iso Octyl Acid Phosphate Diluted to 50% with distilled water	
Total Hrs. of Operation	2005		2005*		2053	
pH Before	7.49		7.43		7.48	
pH After	7.28		6.90		7.33	
R.A. Before	11.70		11.60		11.75	
R.A. After	8.90		7.50		9.80	
Visual Inspection and Wt. Change mg/sq.cm.						
Aluminum	V.Sl.Stain	-0.02	V.Sl.Stain	+0.31	V.Sl.Stain	-0.03
Copper	Etched	-5.92	H.Stain	-0.68	H.Stain	-3.47
Solder	V.Sl.Stain	-0.01	V.Sl.Stain	+0.02	O.K.	-0.02
Brass	Mod. Stain	-2.13	Mod.Stain	-0.34	H.Stain	-1.26
Steel	V.Sl.Stain	+0.01	V.Sl.Stain	+0.02	V.Sl.Stain	+0.02
Cast Iron	V.Sl.Stain	+0.06	V.Sl.Stain	+0.09	Sl.Rusting	+0.03
CCL Rating		25		20		24
*Remarks			Replaced faulty pump at 555 hrs.			

TABLE IV

INHIBITOR PRECIPITATION IN HARD WATER

Coolant Solution[3]	Precipitate % by Volume
0-A-548a Antifreeze + 0.12% Na_2HPO_4	10% (crystalline)
90.7% Ethylene Glycol, 5.0% water, 4.0% Borax, 0.3% Iso Octyl Acid Phosphate	1%
90.7% Ethylene Glycol, 5.0% Water, 4.0% Borax, 0.3% Iso Octyl Acid Phosphate.[1]	1% - Floats on top
90.7% Ethylene Glycol, 5.0% Water, 4.0% Borax, 0.3% Di-2-Ethyl Hexyl Phosphoric Acid	1% Yellowish
Commercial Antifreeze containing Na_2HPO_4 [2]	3% (Crystalline)

[1]Phosphate free of pyro and poly phosphates

[2]Antifreeze as packaged 50% solution
Diluted to 30% with hard water

[3]50% solutions with hard water

Synthetic Hard Water

1.5270 gms $Ca\ (C_2H_3O)_2 \cdot H_2O$
1.0496 gms $MgSO_4$
Dissolved in 1 gallon distilled water
Hardness = 20 grains = 342 PPM as $CaCO_3$

TABLE V

NUMERICAL RATING SYSTEM FOR METAL TEST SPECIMENS

mg loss/sq.cm.	Rating
.00 - .10	1
.11 - .20	2
.21 - .30	3
.31 - .40	4
.41 - .50	5
.51 - 1.00	6
1.01 - 3.00	7
3.01 - 7.00	8
7.01 - 14.00	9
14.01 - 50.00	10
50.01 +	11

All weight gains regardless of how much have a rating of 1
Add 1 to each strip that is not visually perfect.

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13. ABSTRACT

The object of this study was to investigate the use of organic phosphates as corrosion inhibitors in coolant formulations. Glassware bench corrosion tests and simulated service tests were conducted on various organic acid phosphates in combination with other inhibitors such as sodium tetraborate (borax), sodium metaborate and sodium mercaptobenzothiozole (NACAP). Test data showed that these phosphates when exposed to hard water form non-crystalline precipitates that offer no deleterious effect to cooling systems. Test data also showed that these phosphates are effective corrosion inhibitors when used with a sodium borate buffer.

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14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
Antifreeze Organic Phosphate Inhibitor Hard water precipitates Inhibition of aluminum						

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