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Serial No. <u>370,236</u>____ Filing Date <u>21 April 1982</u> Inventor<u>Charber T. Lynch</u>

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(8-76)	NATIONAL TECHNICAL INFORMATION SERVICE
INVENTION EV	ALUATION QUESTIONNAIRE
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		KNOWLEDGE OF THE	INVEN	TION AND INDUSTRY	
•	WHA	T IS YOUR RELATIONSHIP TO TH	HE IN	VENTION ?	
	1.	Inventor/Co-inventor	4.	Other patent attorney	
	2.	Inventor's Technical Supervisor	5.	NTIS Invention Evaluator	
	(3.)	Patent attorney who prepared/ prosecuted the invention	6.	Other (please specify)	
3.	HOW QUE	FAMILIAR WERE YOU WITH THE STIONNAIRE ?	INVE	NTION PRIOR TO RECEIVING TH	IS
	1.	Intimately familiar	3.	Was aware of it	
	2.	Moderately familiar	4.	No previous knowledge	
2.	HOW gen	FAMILIAR ARE YOU WITH THE D eral structure) TO WHICH TH	INDUS E INV	TRY (manufacturing, marketi ENTION RELATES ?	ng, and
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	2.	Moderately familiar	-		
		STATUS OF IN	VENTI	ON DEVELOPMENT	
).	WHA	T IS THE CURRENT STATUS OF 1	THE I	NVENTION ?	
	1.	Not in use and not being developed, last use or development on	3.	Currently in use	
	\bigcirc	Still being developed	4.	Unknown	
2.	HOW	FAR HAS THE DEVELOPMENT OF	THE	INVENTION BEEN CARRIED ?	
	1.	No development beyond preparation of patent application	n 4.	Full scale production	
	″(2.)	Experimentation models, bread boards, prototypes	5.	Unknown	
	3.	Limited production			
P.	WHA	T IS THE EXTENT OF CURRENT (COMME	RCIAL USE OF THE INVENTION	?
	1.	Being considered for commercial development (specify company)	3.	Currently in commercial use (specify company)	
	2.	Under commercial development (specify company)	4.) No known commercial interest	

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SIGNIFICANCE OF INVENTION IN ITS FIELD WHAT IS THE RELATIVE SIGNIFICANCE OF THE INVENTION IN ITS FIELD OF G. **TECHNOLOGY** ? (4.) Significant advance 1. Known in existing technology 2. Slight modification 5. Major improvement 3. Modest advance 6. Pioneer discovery WHAT ARE THE PRINCIPAL ADVANTAGES OF THE INVENTION OVER THE PRIOR ART ? Ħ. Multifunctionality of the inhibitor composition. **RELATED DISCLOSURES** ARE THERE OTHER PATENTS/PATENT APPLICATIONS THAT DIRECTLY RELATE TO THIS I. INVENTION ? 4. Other (please specify) Appl. Ser. No. 265, 734 files May 1981 1. No 2. Divisional ser. nos. 3. Continuation-in-part ser. nos. 5. Unknown PLEASE CITE ANY PUBLISHED TECHNICAL REPORTS OR JOURNAL ARTICLES THAT J. DESCRIBE THE INVENTION AND INDICATE PUBLICATION DATES. "Materials and Process Opplications for Land, Sea, air, Space" Vol 26, pp 52-64 (1981) COMMERCIAL POTENTIAL OF INVENTION WHAT IS THE LIKELIHOOD OF ULTIMATE COMMERCIAL USE OF THE INVENTION ? κ. 1. None Good 4. Excellent 5 Poor 2. Unknown 3. Fair 6. FORM NTIS-202 (8-76) USCOMM-DC SP01-P76 -3-

	1.	Not applicable		5.	\$500,000 to \$1,000,000	
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	1.	None		5.	\$1,000,000 to \$5,000,000	
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AF INV 14985 Charles T. Lynch et al

HIGH PERFORMANCE MULTIPUPCTIONAL CORROGION INHIBITORS

RIGHTS OF THE GOVERNMENT

The invention described herein may be manufactured and used by or for the Government of the United States for all governmental purposes without the payment of any royalty.

BACKGROUND OF THE INVENTION

This invention relates to corrosion inhibiting compositions and to a process for inhibiting the corrosion of metals. In particular, this invention relates to a multifunctional inhibitor that provides both anodic and cathodic corrosion inhibition for a broad spectrum of metallic materials and structures in aggressive media such as brine, bilge solution and high-chloride contaminated water.

The financial loss due to the degradative effects resulting from corresion reactions amounts to billions of dollars annually. In an attempt to combat the problem of corresion and minimise its economic disadvantages, a U.S. Air Porce research effort was initiated to develop improved inhibiting compositions. As a part of this research effort a survey and screening of conventional inhibitor compositions such as the polyphosphates, silicates, orthophosphates, chromates, nitrites, and combinations thereof was undertaken to determine their effectiveness in inhibiting corresion of aircraft structures. Film-forming inhibitors, such as emulsified or soluble oils, long chain amines, alcohols and carbonylic acids were also studied.

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Unfortunately, anodic inhibitors, such as the chromates, may chune accelerated corrosion when in contact with a metal in too low a concentration, such as where the concentration decreases during was. The result may be a metal surface protected in most areas,

5 but giving rise to accelerated correction in small, highly anodic areas of the metal surface. On the other hand, a significant advantage of chromate inhibiting formulations is their broad protective ability against general correction of many metals and alloys.

For somehromate systems, a rather complex mixture is required 10 to achieve such broad-based protection. The simple borar-mitrite system is, for example, effective for many steels, but must be complemented by other inhibiters to provide adequate protection for high strength aluminum alloys, particularly in the presence of corsesive contaminants such as sedium chloride.

15 Other inhibitor systems are applicable only to a limited number of alloys or lack the degree of protection for satisfactory and adequate protection for accorpans and other high performance (high strength, high strength:weight ratio, high fatigue resistance) structural alloys. Still other inhibitor systems have not been

20 found satisfactory for use with high performance alloys in the presence of sodium chloride. Commercial formulations which have been tested on acrospace alloys, such as 7075-T6A1, 2024-T3A1, and 4340 steel, in the presence of sodium chloride in aqueous solutions have

ranged from totally ineffective to partially effective in immersion tests.

Toxicity has become an increasingly important consideration in recent years, both with respect to handling of the compounds prior to use, and to the effects of disposal on humans, animals and plants. Consequently, it is necessary to develop substitutes for such popular inhibitors as chromate based formulations and high phosphate based formulations.

A previous study on corrosion prevention of carrier-based aircraft revealed that a considerable savings could be realized in 10 terms of corrosion maintenance by merely rinsing the aircraft with water to remove detrimental particles, such as salt and ash. How, ever, in rinsing aircraft, a very good possibility exists that the water will be trapped in crevices or so-called dry-bay areas. The trapped water, often chemically hard, can cause serious corrosion 15 problems, hence completely jeopardizing the advantage of water rinsing as a corresion-control method. Therefore, the incorporation of a low concentration of a nontoxic, water-soluble inhibitor into the rinse water becomes a desirable means for improving corrosion resistance.

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The value of borax-nitrite as a corrosion inhibitor has long been recognized. Earlier work has shown this combination to be very effective in controlling general corrosion as well as crevice

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corrosion of high strength steels. However, the borax-nitrite combination was not found to be effective against the corrosion of other ferrous and nonferrous metals and alloys. For example, nitrite inhibitors are more effective at higher pH ranges (e.g., 8-9) than at more acidic levels. Very high pR levels, however, can be deleterious to some aluminum alloys since aluminum is amphoteric, subject to attack by strong basic solutions.

In our copending application Serial No. 265,734, filed May, 1981, we disclose corrosion inhibiting compositions which are bio-10 degradable, contain no chromates, and offer important and unique advantages over chromate-based inhibitor combinations. The compositions are multifunctional, providing both anodic and cathodic protection. The compositions are nontoxic, low in cost, soluble in aqueous solution and provide protection for a broad spectrum of metallic structures. Concentration of the inhibitor composition in 15 aqueous rinsing solution is nominally 0.3 to 0.5 percent, by weight, of the rinse solution. The inhibiting compositions include sodium borate, sodium nitrite, sodium hexametaphosphate, sodium metasili-

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20 range of concentrations.

> We have found that the effectiveness of the compositions disclosed by us in the aforesaid application Serial No. 265,734 can be improved by the addition thereto of selected surfactant compounds.

cate, sodium nitrate and mercaptobensothiasole in a predetermined

These improved corresion inhibiting formulations are particularly useful in very aggressive environments containing chloride ion in excess of 1000 ppm (0.1 weight percent). Such high levels may be found in coastal areas and in urine, which contains approximately one weight percent sodium chloride, or about 6000 ppm of chloride ion.

Accordingly, it is an object of the present invention to provide an improved multifunctional corrosion inhibiting composition. Other objects and advantages of the present invention will be readily apparent to those skilled in the art from a consideration of the following disclosure.

SUMMARY OF THE INVENTION

In accordance with the present invention there is provided an improved corrosion inhibiting composition consisting essentially of a mixture of an alkali metal borate, an alkali metal nitrite, an alkali metal nitrate, an alkali metal metasilicate, an alkali metal phosphate, mercaptobensothiazole (MBT), at least one selected surfactant. In some formulations, zinc sulfate and bensotriasole (BT) are also required. The alkali metal can be either sodium or 30 potassium.

BRIEF DESCRIPTION OF THE DRAWINGS

In the drawings;

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Figure 1 illustrates the anodic-polarization behavior of type 7075-T6 aluminum in local top water, distilled water, 0.1 N NaCl and the basic inhibitor solution;

Figure 2 illustrates the effect of increasing chloride concentration upon the breakdown of passivity of type 7075-T6 aluminum;

Figure 3 illustrates the effect of increasing chloride concentration upon the breakdown of passivity of type 4340 steel;

Figure 4 illustrates the effect of adding a surfactant to an inhibitor solution in preserving passivity of type 7075-T6 aluminum;

Figure 5 illustrates the effect of adding a surfactant to an inhibitor solution in preserving passivity of type 4340 steel;

Figure 6 illustrates the anodic polarization of type 7075-T6 10 aluminum in natural and in synthetic urine: and

Figure 7 illustrates the effects of adding the inhibitor composition of this invention for preserving the passivity of various metals in synthetic urine solution.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

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The amounts of each component of the inhibiting composition of this invention are given in Table I, below.

TABLE I

	Component	Concentration solution (weil Broad	ight percent) Preferred	Concentration of Dry Ingredients (percent)		
5	Borate	.20 -2.00	0.25 -1.40	68.0 -70.0		
	Nitrite	.0425	.0520	8.5 -14.0		
	Nitrate	.0450	.0540	13.5 -17.5		
	Silicate	.001505	.00504	0.5 - 1.75		
	Phosphate	.0025025	.00502	0.8 0.9		
10	MBT	.0008015	.003012	0.25 - 0.5		
	BT	.0008015	.003012	0.25 - 0.5		
	ZnSO4	.00306	.00505	1.0 - 2.0		
	Surfactant	.00603	.0050025	1.0 - 2.0		

The surfactant is a selected avionic or nonionic surface 15 active material. The selected surfactants employed in the corrosion inhibitor of the present invention are, in general, proprietary materials. Table II, below, lists the surfactants employed according to the present invention by (1) an arbitrary designation, (2) a brief description of the composition of the surfactant, (3) the 20 commercial name of the surfactant, and (4) the source for such surfactant.

TABLE II

Desig-(1) nation	Description ⁽²⁾	Commercial ⁽³⁾ Name	Source (4)
5AR	Sodium Dodecylbenzene Sulfonate	Richonate	The Richardson Company Des Plaines, Illinois
SAD	Sodium salt of Phosphonic acid	Dequest	Monsanto Company St. Louis, Nissouri
SAB	Corrosion inhibitor (commercial formulation) with complex sulfonate compound	Boeshield T-9	Oxy Netal Industries Corp. Nadison Heights, MI
Sam	Dialkyl alkyl phosphonate		Mobil Chemical Company Phosphorous Division Richmond, Virginia
SAP	Eigh molecular weight phosphate		Monsanto Company St. Louis, Missouri
SAT	Octylphenoxy polyethoxy ethanol	Triton X-114	Rhohm and Haas Co. Industrial Chemicals - NJ Philadelphia, Pennsylvani
8 80	Bigh molecular weight calcium sulfonate	100 011	The Southland Corp. Arthur C. Trask Chemical Division Summit, Illinois
8 λ Ε	Righ molecular weight Barium sulfonate sulfonate	Estersulf	The Southland Corp. Arthur C. Trask Chemical Division Summitt, Illinois
BA G	Sodium salt of a complex Phos- phate ester		GAF Corporation New York, New York

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Proprietary Surface Active Agents

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Referring now to the drawings, Figure 1 shows the anodic polarisation behavior of type 7075-T6 aluminum in distilled water, local tap water, a 0.1 molar solution of sodium chloride and local tap water containing the corrosion inhibitor disclosed in the aforementioned application Serial No. 265,734. This figure illustrates a very high corrosion current and breakdown in passivity in tap water and in 0.1 M NaCl, as well as illustrating the protection afforded by the aforesaid corrosion inhibitor. Figure 2 illustrates the effect of increasing chloride concentration upon the breakdown of passivity of type 7075-T6 aluminum. Figure 3 illustrates a similar type of behavior with type 4340 steel.

Figures 4 and 5 illustrate the effect of adding 125 ppm of sodium dodecylbenzene sulfonate (SAR) to solutions of increasing chloride concentration, each containing the basic inhibitor mentioned above. A comparison of Figure 4 with Figure 2, although not strictly comparable, clearly indicates the increased protection afforded by the addition of sodium dodecylbenzene sulfonate to the basic inhibitor formulation. A more direct correlation is seen by reference to Figures 5 and 3.

20 Figures 6 and 7 illustrate the anodic polarization behavior of various metals in a synthetic urine solution. The composition of the synthetic urine is given in Table III below.

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TABLE III

Ingredients of Synthetic Urine (Wt in gm/liter)

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1....

	urea	20.60
5	5-hydroxyindoleacetic acid	0.0045
	uric acid	0.052
	glucuronic acid	0.431
	oxalic acid	0.031
	citric acid	0.462
10	glycolic acid	0.042
	creatine	0.0721
	guanidinoacetic acid	0.027
	formic acid	0.013
	glucose	0.072
15	ammonium sulfate	4.00
	potassium phosphate	0.175
	potassium chloride	0.0100
	potassium bromide	0.00\$
	sodium chloride	10.00
20	p-cresol	0.087
	greatinine	1.500
	acetone	0.0001
	hydroxyquinoline-2 earboxylic acid	8.0028
	potassium sulfate	0.134

Figure 6 illustrates that the corrosive behavior of synthetic urine closely approximates that of natural urine. Figure 7 illustrates the anodic polarisation behavior of type 7075-T6 aluminum, type 4340 steel, copper and brass in synthetic urine and in synthetic urine inhibited by the multifunctional inhibitor formulation containing 125 ppm of sodium dodecylbensene sulfonate.

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More specific inhibitor formulations are given in Tables IV and V below. All amounts are given in weight percent (in aqueous solution).

Formulation Т Component T 2 Borate 0.35 0.35 0.35 0.35 Mitrite .20 .20 .20 .20 15 Mitrate .20 .20 .20 .20 Silicate .01 .01 .01 .01 Phosphate .0125 .005 .005 .005 MBT .0065 .005 .005 .005 BT .005 .005 .005 .005 20 SAR, SAE .0125 .0075 SAD .0165 SAT .01 SAB .025 ZnSO4 .004 . 11 .02-.04

TABLE IV

		Por		
	Component	5	88	7
	Borate	0.25	0.35	1.40
5	Nitrite	. 05	.05	.20
	Nitrate	. 05	.10	240
	Silicate	.002	.01	.04
	Phosphate	.003	.005	.02
	MBT	.001	.003	.012
10	BT		.003	******
	SAR	.0075	.0075	.0075

Formulation 1 is preferred for use where the concentration of chloride is very high, e.g., brine. Formulations 2 and 3 are recommended for use in aggressive solutions such as are found in the bilge areas of aircraft. Formulation 4 will provide protection in high chloride contaminated water, i.e., up to about 1 weight per-

TABLE V

cent NaCl.

Formulation 6 is a preferred formulation for general purpose use. It is effective where little or no dilution is expected during use and low concentrations of chloride and other aggressive reactants are present, i.e., up to about 100 ppm chloride ion. Formulation 5 is effective in situations where no dilution is expected and the concentration of chloride ion or other aggressive reactant is very low. Formulation 7 is for contact inhibitors to form a protective surface layer during immersion.

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The concentrations of the various components can be varied by about 20% for conditions where dilution in use is expected.

In Table VI, below, the representative results of tests with several experimental formulations are summarized. These immersion 5 tests were carried out on type 7075-T6 aluminum and type 4340 steel in LM NaCl solutions.

TABLE VI

Ismersion Test Results

No	Inhibitor wt% in 1M NaCl	pH Initial	Final	Specimen	Time of Exposure (weeks)	Surface Appearance (Visual Observation)	Nemarks
ī	0.35 Borate + 0.2 Nitrate +					AA	
	0.01 Silicate	/.90	7.90	Al	2	Several pits	Better Inhibitor
	+ 50 ppm Phos- phate + 30 ppm NBT + 100 ppm SAO			Steel	2	Clean & shiny; few pits	required
2	0.35 Borate + 0.6 Nitrate +						
	0.6 Nitrite + 0.01 Silicate + 50 ppm Phos-	8.30	8.20	X1	16	Clean Few pits	Better Inhibitor
	phate + 30 ppm MBT			Steel	1 6	Clean, Few fine pits Many pits at edge	required
<u>3</u>	0.35 Borate + 0.2 Mitrate + 0.05 Nitrite + 0.01 Silicate	8.20	8.15	X1	4 16	Clean & shiny Clean & shiny	Improve-
	+ 50 ppm Phos- phate + 50 ppm MBT + 100 ppm SAE			Steel	4 16	Clean, pits Clean, several pits	required

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4	0.35 Borate + 0.2 Nitrate + 0.05 Nitrite + 0.01 Silicate + 50 ppm Phos- phate + 50 ppm SAM	8.20	8.25	Al Steel	2 2	Dull, patches of Corrosion Several pits	Better Inhibitor required
5	0.35 Borate + 0.1 Nitrate + 0.05 Nitrite + 0.01 Silicate + 50 ppm Phos- phate + 50 ppm NBT + 50 ppm	8.80	8.70	Al Steel	2 10 2	Clean Clean, few corrosion streaks Clean	Fair
6	SAP + 100 ppm SAE 0.35 Borate + 0.2 Nitrate + 0.2 Nitrite + 0.01 Silicate + 125 ppm Phos- phate + 60 ppm MBT + 100 ppm SAP + 210 ppm	8.15	8.10	Al Steel	10 2 12 2 12	Clean & shiny Clean & shiny Clean & shiny Clean & shiny, two fine pits	Excellent inhibition
7	AAR + 210 ppm SAD + 40 ppm EnSO ₄ 0.35 Borate + 0.2 Nitrate + 0.2 Nitrate + 0.01 Silicate + 50 ppm Phos- phate + 75 ppm SAT + 500 ppm	8.15	8.20	Al Steel	2 8 2 8	Clean & shiny Clean & shiny Clean & shiny Clean & shiny	Excellent Inhibitor

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	0.2 Nitrate +						
	0.2 Nitrite +	9.35	9.20	A1	2	Clean & shiny	
	0.01 Silicate + 50 ppm Phos-				8	Clean & shiny	Excellent
	phate + 100 ppm			Steel	2	Clean & shiny	Indiditor
	MBT + 75 ppm SAR + 100 ppm ZnSo ₄				8	Clean & shiny, one fine pit	
9	1% SAB	6.25	6.25	<u>۸۱</u>	4	Clean & shiny	
.					24	Badly corroded	Better
				Steel	4	Clean & shiny	required
					24	Badly corroded	
10	0.35 Borate + 0.2 Nitrate +						
	0.2 Nitrite +	8.15	8.20	Al	2	Clean & shiny	
	0.01 Silicate				8	Clean & shiny	Excellent
	phate + 100 ppm			Steel	2	Clean, one pit	Inhibitor
	MBT + 250 ppm				8	Clean & shiny	

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In Table VI above and in Table VII, below, the term borate refers to sodium borate tetrahydrate, nitrate to sodium mitrate, nitrite to sodium nitrite, silicate to sodium metasilicate pentahydrate, and phosphate to sodium hexametaphosphate.

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In Table VII below, the representative results of tests with several experimental formulations are summarized. These tests were carried out on type 7075-T6 aluminum, type 4340 steel, and brass in synthetic urine solution, and in a mixture of synthetic urine and coffee.

Inhibitor Time of wt% in synthetic Exposure Surface Appearance pH Initial Final urine* Specimen (weeks) (Visual Observation) Remarks No 11 0.35 borate + 0.2 nitrite + Clean & shiny 0.2 nitrate + 9.35 9.25 A1 2 0.01 silicate + 8 Clean & shiny 100 ppm ZnSO + 50 ppm phosphate Excellent 2 Clean & shiny Steel Clean & shiny, one + 75 ppm SAR 8 fine pit 12 0.35 borate + 0.2 nitrite + 0.2 nitrate + 8.50 8.50 **A1** 4 Clean & shiny 0.01 silicate + 0.01 phos-Steel 4 Clean & shiny Excellent phate + 0.01 MBT + 125 ppm Brass 4 Clean & shiny SAR 13 0.35 borate + 0.2 nitrite + 0.2 mitrate + 8.15 8.15 A1 2 Clean & shiny Excellent 12 0.01 silicate + 125 ppm phos-2 Brass phate + 60 ppm 12 MBT + 40 ppm InSO₄ + 100 ppm SAR + 200 ppm Steel 2 12 n -, three SAD fine pits

TABLE VII

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14 0.35 borate + 8.15 8.15 **A1** 2 Clean & shiny Excellent 0.2 nitrite + 8 0.2 nitrate + 0.01 silicate 2 н 44 Brass n ۰, 18 + 50 ppm phos-phate + 100 ppm 8 MBT + 250 ppm Steel 2 Clean, one pit appear-SAB ing on one surface 8 Clean & shiny 15 0.35 borate + 8.15 8.00 A1 4 Clean & shiny Excellent 0.2 nitrite + 5 32 0.2 nitrate + 0.01 silicate .* -Brass 4 ** •• Ð + 50 ppm MBT + 32 j. 500 ppm ZnSO4 et 4 Steel 1. ю + 75 ppm SAT ١y м 32

* Except Run 15 which was 50% synthetic urine and 50% coffee.

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The corrosion inhibiting formulations of this invention may be used in aqueous solution as rinse-type inhibitors and as immersiontype inhibitors. The corrosion inhibitor may be compounded dry, and stored in bulk for later solution in water. In the dry form, the corrosion inhibitor may be incorporated from 20 to 50 weight percent, preferably about 30 weight percent, into a commercial soap formulation, e.g., a handsoap, for use in the lavatory of an aircraft or ship.

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The corrosion inhibitor may be incorporated into a coating composition, such as a paint primer by encapsulating the inhibitor formulation with a cellulosic or nylen or other suitable encapsulating material using conventional encapsulating techniques, and incorporating 20 to 50 weight percent, preferably about 30 weight percent, of the encapsulated inhibitor into a conventional coating 15 composition. The corrosion inhibiting components may be released if the coated surface is scratched or otherwise physically damaged.

Various modifications can be made to the above described invention.

ABSTRACT OF THE DISCLOSURE

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A multifunctional corrosion inhibitor consisting essentially of an alkali metal borate, an alkali metal nitrate, an alkali metal nitrite, an alkali metal metasilicate, an alkali metal phosphate, mercaptobenzothiazole and at least one selected surfactant.





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