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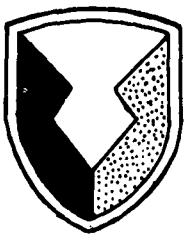

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MEDIUM ALKALINITY CLEANERS OF SUPERIOR DETERGENCY

BY

A. MANKOWICH

AMCMS CODE NO. 5026.11.84205
DA PROJECT 1-H-0-24401-A-110-05

16 APRIL 1963

ABERDEEN PROVING GROUND
MARYLAND

TABLE OF CONTENTS

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Coating and Chemical Laboratory
Aberdeen Proving Ground
Maryland

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ABSTRACT

The inefficiency of medium alkalinity (ca pH12), soak alkaline cleaners in deterging calcium soap-grease was overcome in two ways; viz; by incorporating a small amount of the organic chelating agent, tetrasodium salt of ethylenediaminetetraacetic acid and causing removal by an exchange mechanism, or by the addition of a small amount of specific high boiling - high flash point glycols which produce stable, single phase detergent solutions possessing synergistic power.

I. INTRODUCTION

Federal Specification P-C-436a, "Cleaning Compound, Alkali Type", covers a medium pH (12.1 maximum at use concentration) hot soak tank and hydrosteam cleaner suitable for use with nonferrous and ferrous materials. Sufficient silicate is specified to prevent galvanic corrosion of aluminum when coupled with magnesium. Because of its synergistic anionic-nonionic surfactant combination, this cleaner is superior to heavy duty formulations in the detergency of mineral and cutting oils, sodium soap-greases, and asphalt under specified conditions (2). The high alkalinity cleaners tend to be more efficient in the removal of metallic soap-grease, because high hydroxyl ion concentration has a mass action effect on the double decomposition reaction producing soluble sodium soap from insoluble metallic soap.

This report covers the experimentation involved in improving the calcium soap-grease removal efficiency of P-C-436a cleaners in either of two ways; viz; by incorporating a small amount of the organic chelating agent, tetrasodium salt of ethylenediaminetetraacetic acid (EDTA) and thus permitting removal by an ion exchange mechanism, or by the incorporation of a small amount of specific high boiling - high flash point glycols which produces synergistic, stable, single phase alkaline - organic detergent solutions.

II. DETAILS OF TEST

A. Experimental

Detergency was determined essentially as described in P-C-436a. Because of the extensive program, the steel test panels, after standardized polishing and prior to soil application and aging, were cleaned with c.p. acetone instead of with alkaline detergent. In addition to the specified non-detergent SAE 30 mineral oil and petroleum asphalt soils, calcium soap-grease conforming to Federal Specification VV-G-632, Type A, Grade 1 was used. The latter was applied to one face of the test panel by finger tips, and varied from 290 to 310 mg. The grease cleaning cycle consisted of 5 minutes immersion in 1,600 ml. of a boiling, 7.5% distilled water solution of the test compound, with standardized panel agitation at 2.5 and 5 minutes (as specified for asphalt soil tests in P-C-436a), followed by standardized rinsing (P-C-436a).

B. Surfactants

Three types of anionic syndets were used; namely, USP grade sodium dodecyl sulphate (SDS); 40% active, commercial sodium keryl (C_{10} - C_{20} straight chain alkyl) benzene sulphonate (SKBS); and purified grade sodium oleate soap. For some tests a 92% active form of SKBS was used, and designated SKKBS. The nonionic detergents included commercial ethylene oxide condensates of octylphenol, nonyl phenol, and 2,4,7,9-tetramethyl-5-decyne-4,7-diol, as follows:

polyoxyethylene (9-10) of octylphenol ---------- OPE9-10

ethylene oxide adducts of nonyl phenol:

15 mole ratio adduct ---------- NPPGE
30 mole ratio adduct ---------- NPTGE
100 mole ratio adduct ---------- NP100E

ethylene oxide adducts of decyne-4,7-diol:

10 mole ratio adduct	D-10
15 mole ratio adduct	D-15
30 mole ratio adduct	D-30

C. Other Additives

The organic chelating agent EDTA was a technical grade powder with a complexing value of 215 mg $CaCO_3$ per gram at pH 11. The glycols and triol were commercial grade solvents.

D. Cleaner Formulations

The standard comparison compound of P-C-436a has the following composition:

sodium metasilicate pentahydrate	34.5%
primary sodium phosphate monohydrate	12.0%
trisodium phosphate dodecahydrate	33.5%
OPE9-10	5.2%
SKBS	14.8%

Since SKBS is 40% active, it actually represents 5.9% anionic detergent plus 8.9% sodium sulphate builder. To simplify the evaluation of specific changes, several formulation parameters were kept constant. All compositions contained 5.2% nonionic and 5.9% anionic surfactants on a dry basis. Except in a few cases, the pH of the 7.5% solutions of the test compounds was ca 12 (at 25°C.).

III. RESULTS AND DISCUSSION

A. EDTA and Calcium Soap-Grease (CS-G) Detergency

Table II shows that CS-G detergency can be imparted to P-C-436a - type cleaners (Table I and section II-D) by the addition of 8.9 - 12% EDTA and the simultaneous elimination of trisodium phosphate from the formulation. Some surfactant specificity seeems to exist since these generalizations are not valid for cleaners containing the SDS-NPPGE, SDS-NPTGE, and SKKBS-NPTGE, anionic-nonionic mixtures. While in the latter three cases the combined physicochemical plus chemical (EDTA) actions are insufficient to deterge CS-G, in a few compositions (#89, #95, #91) the resultants of these actions are powerful enough to remove the soil in the presence of an added 10% or 15% trisodium phosphate. It is to be noted that at the average pH 12 level maintained in the developed detergent solutions, all formulated with primary sodium phosphate, that the latter is converted to a mixture of dibasic and tribasic sodium phosphates. Calculations based on the third ionization constant of phosphoric acid indicate that, at pH 12, the cleaners with no added trisodium phosphate (like # 83, for example) actually contain 10.7% trisodium phosphate dodecahydrate and 8.4% secondary sodium phosphate. Orthosphosphate ion above a certain concentration, and under the conditions present in these cleaners, seems to act as a precipitating agent tending to destroy the soluble calcium complex of EDTA as it forms and to re-precipitate insoluble calcium compounds on the basis metal.

The data of Table III show that a cleaner containing as much as 12% EDTA (#96) can meet the cleaning, stability, alkalinity, corrosion, and non-caking requirements of P-C-436a, as well as having the additional capability of efficiently deterging CS-G soil.

Table IV gives the results of exploratory CS-G soil removal tests in which EDTA is replaced by another organic chelating agent, the trisodium salt of nitrilotriacetic acid monohydrate (NTA) in cleaner # 85, a developed formulation possessing excellent mineral oil, asphalt, and CS-G detergent efficiency. It is indicated that in this application (solubilization and/or removal of calcium soap) NTA is not as effective as EDTA, since twice as much of the former is required.

B. Glycol-Triol Additives and CS-G Detergency

The addition of high boiling - high flash point glycols and triols to 7.5% solutions of the Table I formulations results in stable, single phase solutions. The cleaners selected for study (poor CS-G soil removers) contained no EDTA nor NTA, and included formulations with all three types of anionic surfactants. As measured by ability to deterge CS-G, Table V shows the synergistic effect, and the variation in this synergism, of small additions (as little as 3.75% by volume) of specific glycols. Hexylene glycol appears as a considerably more efficient additive than dipropylene glcyol. Hexanetriol is of little value in this application. The glycols alone are ineffective detergents of CS-G.

C. Conclusions

The addition of specific glycols to P-C-436a cleaner solutions not only greatly increases their detergent efficiency, but places them directly in competition with hydrosteam cleaners of the alkaline water-base and water-emulsion types intended for use on aircraft surfaces. Table VI gives typical formulations of these cleaners and their soil removal data. It is seen that the alkaline water-base type, which vigorously attacks aluminum (2S), can remove mineral oil and CS-G, but is practically without effect on asphalt soil. The water-emulsion type shows poor detergency on all three soils. The use of P-C-436a cleaners containing hexylene glycol in the hydrosteam cleaning of aircraft surfaces is strongly indicated. The use of hexylene glycol as a co-solvent additive to aqueous alkaline solutions was covered by this laboratory in CCL # 78 and patent application serial number 215,465.

IV. REFERENCES

1. Mankowich, A., "Coating & Chemical Laboratory Report, CCL # 108", 22 June 1961.
2. Federal Specification P-C-436a, "Cleaning Compound, Alkali Type".

APPENDIX

Tables

TABLE I

CLEANERS WITH Na_2SO_4 OR EDTA ADDITIVES

Cleaner	pH, 25°C. 7.5% sol'n.	Composition, % by weight, dry basis						
		nonionic	anionic	additive	Na_2CO_3	$NaH_2PO_4 \cdot H_2O$	$Na_2SiO_3 \cdot 5H_2O$	$Na_3PO_4 \cdot 12H_2O$
24	11.90	5.2% OPE9-10	5.9% SDS	8.9% Na_2SO_4	---	12.0	34.5	33.5
24-A	11.90	5.2% OPE9-10	5.9% SDS	8.9% EDTA	---	12.0	34.5	33.5
83	12.00	5.2% OPE9-10	5.9% SDS	8.9% EDTA	28.0	12.0	40.0	---
89	11.65	5.2% OPE9-10	5.9% SDS	8.9% EDTA	23.5	12.0	34.5	10.0
90	11.85	5.2% OPE9-10	5.9% SDS	8.9% EDTA	18.5	12.0	39.5	10.0
88	11.70	5.2% OPE9-10	5.9% SDS	8.9% EDTA	18.5	12.0	34.5	15.0
95	11.90	5.2% OPE9-10	5.9% SDS	12.0% EDTA	15.4	12.0	34.5	15.0
87	11.80	5.2% NPPGE	5.9% SDS	8.9% EDTA	28.0	12.0	40.0	---
39	11.90	5.2% NPTGE	5.9% SDS	8.9% Na_2SO_4	---	12.0	34.5	33.5
39-A		5.2% NPTGE	5.9% SDS	8.9% EDTA	---	12.0	34.5	33.5
86	12.00	5.2% NPTGE	5.9% SDS	8.9% EDTA	28.0	12.0	40.0	---
97		5.2% NPTGE	5.9% SDS	12.0% EDTA	26.5	12.0	38.4	---
36	12.00	5.2% NP100E	5.9% SDS	8.9% Na_2SO_4	---	12.0	34.5	33.5
36-A	11.90	5.2% NP100E	5.9% SDS	8.9% EDTA	---	12.0	34.5	33.5
74	12.00	5.2% NP100E	5.9% SDS	8.9% EDTA	28.0	12.0	40.0	---
94		5.2% NP100E	5.9% SDS	8.9% EDTA	18.5	12.0	34.5	15.0
36-B	11.10	5.2% NP100E	5.9% SDS	8.9% EDTA	---	12.0	---	68.0
36-C	10.70	5.2% NP100E	5.9% SDS	8.9% EDTA	34.5	12.0	---	33.5
73	10.70	5.2% NP100E	5.9% SDS	8.9% Na_2SO_4	34.5	12.0	---	33.5
28	11.80	5.2% D-10	5.9% SDS	8.9% Na_2SO_4	---	12.0	34.5	33.5
28-A	11.90	5.2% D-10	5.9% SDS	8.9% EDTA	---	12.0	34.5	33.5
84	12.00	5.2% D-10	5.9% SDS	8.9% EDTA	28.0	12.0	40.0	---
92	11.70	5.2% D-10	5.9% SDS	8.9% EDTA	18.5	12.0	34.5	15.0
91	11.90	5.2% D-10	5.9% SDS	8.9% EDTA	18.5	12.0	39.5	10.0
30	11.85	5.2% D-15	5.9% SDS	8.9% Na_2SO_4	---	12.0	34.5	33.5
30-A	11.90	5.2% D-15	5.9% SDS	8.9% EDTA	---	12.0	34.5	33.5
85	12.00	5.2% D-15	5.9% SDS	8.9% EDTA	28.0	12.0	40.0	---
93		5.2% D-15	5.9% SDS	8.9% EDTA	18.5	12.0	34.5	15.0

TABLE I (CONTINUED)

CLEANERS WITH Na_2SO_4 OR EDTA ADDITIVES

Cleaner	pH, 25°C. 7.5% sol'n.	Composition, % by weight, dry basis						
		nonionic	anionic	additive	Na_2CO_3	$NaH_2PO_4 \cdot H_2O$	$Na_2SiO_3 \cdot 5H_2O$	$Na_3PO_4 \cdot 12H_2O$
21	12.00	5.2% D-30	5.9% SDS	8.9% Na_2SO_4	--	12.0	34.5	33.5
21-A	11.90	5.2% D-30	5.9% SDS	8.9% EDTA	--	12.0	34.5	33.5
76	12.00	5.2% D-30	5.9% SDS	8.9% EDTA	28.0	12.0	40.0	--
96	12.00	5.2% D-30	5.9% SDS	12.0% EDTA	26.5	12.0	38.4	--
33	11.90	5.2% NPPGE	5.9% Na Oleate	8.9% Na_2SO_4	--	12.0	34.5	33.5
33-A	11.90	5.2% NPPGE	5.9% Na Oleate	8.9% EDTA	--	12.0	34.5	33.5
82	12.00	5.2% NPPGE	5.9% Na Oleate	8.9% EDTA	28.0	12.0	40.0	--
82-A	11.90	5.2% NPPGE	5.9% Na Oleate	12.0% EDTA	26.5	12.0	38.4	--
34	11.90	5.2% NPTGE	5.9% Na Oleate	8.9% Na_2SO_4	--	12.0	34.5	33.5
34-E	12.00	5.2% NPTGE	5.9% Na Oleate	8.9% EDTA	--	12.0	34.5	33.5
34-D	11.95	5.2% NPTGE	5.9% Na Oleate	8.9% EDTA	28.0	12.0	40.0	--
71	11.95	5.2% NPTGE	5.9% Na Oleate	8.9% Na_2SO_4	28.0	12.0	40.0	--
34-F	12.70	5.2% NPTGE	5.9% Na Oleate	20.9% EDTA	--	--	34.5	33.5
31	11.90	5.2% D-30	5.9% Na Oleate	8.9% Na_2SO_4	--	12.0	34.5	33.5
75	12.00	5.2% D-30	5.9% Na Oleate	8.9% EDTA	28.0	12.0	40.0	--
75-A		5.2% D-30	5.9% Na Oleate	8.9% EDTA	18.5	12.0	34.5	15.0
23	11.90	5.2% OPE9-10	5.9% SKKBS	8.9% Na_2SO_4	--	12.0	34.5	33.5
77	12.00	5.2% OPE9-10	5.9% SKKBS	8.9% EDTA	28.0	12.0	40.0	--
77-A	12.00	5.2% OPE9-10	5.9% SKKBS	12.0% EDTA	26.5	12.0	38.4	--
40	11.90	5.2% NPTGE	5.9% SKKBS	8.9% Na_2SO_4	--	12.0	34.5	33.5
80	12.00	5.2% NPTGE	5.9% SKKBS	8.9% EDTA	28.0	12.0	40.0	--
37	11.90	5.2% NP100E	5.9% SKKBS	8.9% Na_2SO_4	--	12.0	34.5	33.5
78	12.00	5.2% NP100E	5.9% SKKBS	8.9% EDTA	28.0	12.0	40.0	--
1	11.90	5.2% D-30	5.9% SKKBS	8.9% Na_2SO_4	--	12.0	34.5	33.5
79	12.00	5.2% D-30	5.9% SKKBS	8.9% EDTA	28.0	12.0	40.0	--
79-A	12.00	5.2% D-30	5.9% SKKBS	12.0% EDTA	26.5	12.0	38.4	--

TABLE II

DETERGENT EFFICIENCY - TABLE I CLEANERS (7.5% SOLUTIONS)

Cleaner	Mineral Oil 3 minute cleaning	Asphalt minutes for removal	Calcium Soap-Grease mg residue in 5 minute cleaning
24	Good	8	204
24-A	Good	12	131
83	Good	12	None
89	Good	10	None
90	Good	--	0.5 - 1.5 - 5
88	Good	12	1 - 2 - 8
95	----	11	None
87	Good	10	32 - 61
39	Good	19	218
39-A	Good	21	124
86	Good	13	33 - 44
97	Good	--	3 - 4
36	Good	21	248
36-A	Good	21	41
74	Good	19 - 19	None
94	----	--	36
36-B	Good - test panels corrode	> 21 - poor	2 - 15
36-C	Good - test panels corrode	19 - 20	None
73	Good	16	145
28	Good	5 - 6	69
28-A	Good	6	36 - 64
84	Good	6	None
92	----	--	0.3 - 0.6
91	Good	7	None
30	Good	7 - 9	96
30-A	Good	7	82
85	Good	6	None - in 3-4 min.
93	----	--	12
21	Good	6 - 7	228
21-A	Good	15	47
76	Good	11	1 - 4 - 17
96	Good	7	None - in 3 min.
33	Good	10 - 13	247
33-A	Good	15	235
82	Good	12	51 - 77
82-A	Good	13	None
34	Good	7 - 9	250
34-E	Good	7	184
34-D	Good	8 - 10	None
71	Good	8	269
34-F	Good	9	130
31	Good	10	253
75	Good	11 - 12	None
75-A	Good	--	4

TABLE II (CONTINUED)

DETERGENT EFFICIENCY - TABLE I CLEANERS (7.5% SOLUTIONS)

Cleaner	Mineral Oil 3 minute cleaning	Asphalt minutes for removal	Calcium Soap-Grease mg residue in 5 minute cleaning
23	Good	12	209
77	Good	18 - 21	0 - 3
77-A	Good	> 21	None in 4 min.
40	Good	16	166
80	Good	11	49 - 54
37	Unsatisfactory	21	244
78	Good	15 - 18	None
1	Good	9 - 9	107
79	Good	9	4 - 11
79-A	Good	8	None in 3½ - 4 min.

NOTE: "Good" mineral oil detergency denotes no water-breaks and no residue-pattern stains in P-C-436a test.

TABLE III

FED. SPEC. P-C-436a TESTS OF CLEANER # 96 (CONTAINING 12% EDTA)

Cleaning efficiency	-	Mineral oil and asphalt soils	-	Good - Table II
Stability	-	Cleaning efficiency after 40 hours boiling (7.5% solution)	-	Good
Corrosion	-	2S aluminum, 1 hour in boiling 7.5% solution	-	None, no weight change, no staining nor pitting.
pH	-	7.5% solution at 25°C.	-	Passes, 12.00
Penetration	-	Caking tendencies	-	Passes, 73 - 86

TABLE IV

NTA VS EDTA AS CALCIUM SOAP-GREASE SOLUBILIZERS

Cleaner	% Concentration of chelating agent in cleaner	Detergent Efficiency: Mineral Oil 3 minute cleaning	Asphalt minutes for removal	Calcium Soap-Grease mg residue in 5 minute cleaning
85	0.67% EDTA	Good	6	None in 3 - 4 min.
85-N	0.67% NTA	Good	--	136 to 201
85-N[1]	1.34% NTA	Good	7	None to 2.6

Cleaner concentrations = 7.5%

TABLE V

EFFECT OF GLYCOL AND TRIOL ADDITIVES ON DETERGENCY

Cleaner	Additive (solvent)	Additive (ml)	Detergency: Mineral Oil 3 minute cleaning	Asphalt minutes for removal	Calcium Soap-Grease mg residue in 5 minute cleaning
21	None	None	Good	6 - 7	228
21-X	Hexylene glycol	60	----	--	None
23	None	None	Good	12	209
23-A	Hexylene glycol	40	----	--	31
23-B	Hexylene glycol	60	----	--	None in 4 minutes
23-C	Hexylene glycol	80	Good	7	None in 3 minutes
34	None	None	Good	7 - 9	250
34-X	Hexylene glycol	60	Good	7	None in 3½ minutes
30	None	None	Good	7 - 9	96
30-F	Dipropylene glycol	120	Good	8	None in 3½ minutes
23-E	Dipropylene glycol	60	Good	--	139
23-F	Dipropylene glycol	100	----	--	14
23-G	Dipropylene glycol	140	Good	6	None in 4½ minutes
34-Y	Dipropylene glycol	140	----	--	0.4 - 2
34-Z	Dipropylene glycol	160	----	--	None - 0.4

TABLE V (CONTINUED)

EFFECT OF GLYCOL AND TRIOL ADDITIVES ON DETERGENCY

Cleaner	Additive (solvent)	Additive (ml)	Detergency: Mineral Oil 3 minute cleaning	Detergency: Asphalt minutes for removal	Detergency: Calcium Soap-Grease mg residue in 5 minute cleaning
23-I	1,2,6-hexane triol	120	----	--	177
23-J	1,2,6-hexane triol	160	----	--	119

NOTE: Cleaning solution in all cases = 1,600 ml of 7.5% aqueous solution of cleaner plus additive solvent.

TABLE VI

ALKALINE WATER-BASE AND WATER-EMULSION CLEANERS

Water-Emulsion Cleaner		Alkaline Water-Base Cleaner	
oleic acid	13 grams	$Na_3PO_4 \cdot 12H_2O$	120 grams
triethanolamine	4 grams	OPE9-10	24 grams
OPE9-10	4 grams	ethylene glycol monoethyl ether	72 grams
sodium toluene sulphonate	2 grams	water	to make 1,600 ml
ethylene glycol monobutyl ether	9 grams	Used at a boil	
petroleum solvent, 95% aromatic	37 grams		
water	20 grams		
hexylene glycol	11 grams		

Above diluted 1:4 by volume with water. used at 130°F.

Cleaner	Detergency: Mineral Oil 3 minute cleaning	Detergency: Asphalt mg residue at 21 minutes	Detergency: CS-G 5 minute cleaning; mg residue
Alkaline water-base	Satisfactory	297.	None to 3.6
Water-emulsion	Unsatisfactory 59.5 mg residue	193.	106.

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AD No_____Accession No_____ Coating & Chemical Laboratory, Aberdeen Proving Ground, Md., CCL # 140 - MEDIUM ALKALINITY CLEANERS OF SUPERIOR DETERGENCY - A. Mankowich, Rpt No. 140 - 16 April 1963, 15 pgs, AMCMS Code No. 5026.11.84205, DA Proj No. 1-H-0-24401-A-110-05 The inefficiency of medium alkalinity (ca pH12), soak alkaline cleaners in deterging calcium soap-grease was overcome in two ways; viz; by incorporating a small amount of the organic chelating agent, tetrasodium salt of ethylenediaminetetraacetic acid and causing removal by an exchange mechanism, or by	Unclassified	Unclassified	AD No_____Accession No_____ Coating & Chemical Laboratory, Aberdeen Proving Ground, Md., CCL # 140 - MEDIUM ALKALINITY CLEANERS OF SUPERIOR DETERGENCY - A. Mankowich, Rpt No. 140 - 16 April 1963, 15 pgs, AMCMS Code No. 5026.11.84205, DA Proj No. 1-H-0-24401-A-110-05 The inefficiency of medium alkalinity (ca pH12), soak alkaline cleaners in deterging calcium soap-grease was overcome in two ways; viz; by incorporating a small amount of the organic chelating agent, tetrasodium salt of ethylenediaminetetraacetic acid and causing removal by an exchange mechanism, or by
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Unclassified the addition of a small amount of specific high boiling - high flash point glycols which produce stable, single phase detergent solutions possessing synergistic power.	Unclassified the addition of a small amount of specific high boiling - high flash point glycols which produce stable, single phase detergent solutions possessing synergistic power.
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