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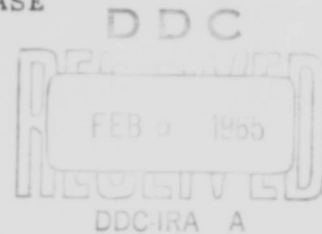
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## TECHNICAL REPORT

ALUMINUM COMPLEX SOAPS AS THICKENERS  
FOR MULTI-PURPOSE GREASE

By

Max T. Fisher



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FOR MULTI-PURPOSE GREASE**

By



Max T. Fisher

Approved by:



G. O. INMAN

Acting Laboratory Director

21 December 1964

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Rock Island, Illinois

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## ABSTRACT

Aluminum complex soaps were evaluated as potential thickeners for greases which would meet the low temperature requirements and exceed the upper temperature limitations of Military Specification MIL-G-10924B Grease, Automotive and Artillery. The reaction of aluminum isopropylate with mixtures of fatty acids and other organic acids in a mutual solvent is the preferred method of forming these complexes. Of the 17 different lower molecular weight organic acids complexed with an aluminum soap, an aluminum benzoate-soap complex showed the best thickening efficiency and highest dropping point.

The effects of benzoate to soap ratios, preformed complex vs in situ preparations, solvent effects on in situ preparations and additive, thickener concentration and dispersion effects were evaluated on aluminum benzoate-soap complex greases made with 74 SUS @ 100°F. oil. The better greases thickened with 5% aluminum benzoate-soap complex were close enough to Military Specification MIL-G-10924B to encourage further formulation adjustments to meet and exceed specification requirements.

## RECOMMENDATIONS

Aluminum benzoate-soap complex grease should be used as a base for formulating a grease that will surpass the requirements of Military Specification MIL-G-10924B. Its good properties included high dropping point, oxidation and copper corrosion resistance, shear stability and good apparent viscosity characteristics at  $-65^{\circ}\text{F}$ . It is believed that certain deficiencies can be overcome with additives. Homologues of benzoic acid or the use of different fatty acids might enhance certain basic properties of aluminum complex soap greases as could the use of oil bases other than the one investigated here.

Functional life tests, such as described in ASTM Method D1741-60T, should be run on these greases under a wide variety of conditions of temperature and load.

ALUMINUM COMPLEX SOAPS AS THICKENERS  
FOR MULTI-PURPOSE GREASE

CONTENTS

	<u>Page No.</u>
Object	1
Introduction	1
Procedure	2
Results and Discussion	3
Literature References	16
List of Prior Reports	17
Distribution	18

## ALUMINUM COMPLEX SOAPS AS THICKENERS FOR MULTI-PURPOSE GREASE

### OBJECT

The formulation and evaluation of aluminum complex soap greases having a broader usable temperature range than current Military Specification MIL-G-10924B greases.

### INTRODUCTION

Increased emphasis on military applications requiring grease lubrication at higher speeds, heavier loads and over wider temperature ranges for longer periods of time requires the surveillance of new lubricant developments and the improvement of lubricants meeting current military specifications.

The Petroleum Products Unit of Rock Island Arsenal Laboratory is custodian of Military Specification MIL-G-10924B, Grease, Automotive and Artillery and its Qualified Products List. In this capacity, the trend of automotive manufacturers to recommend longer periods of time between re-lubrication of points requiring grease in their passenger and truck vehicles is of particular interest. Their conviction of the inadequacy of current wheel bearing and chassis greases of the MIL-G-10924B type to perform well under these stiffer requirements is reflected in the recommendation of specially formulated greases for their particular application - sometimes two or three per vehicle. Multiplying this by the number of automobile manufacturers gives an idea of the large number of greases it is becoming necessary for a service station or the government to stock to lubricate according to the wishes of each company. The associated risk of misapplication is also multiplied many times.

Prior to this trend Military Specification MIL-G-10924B grease was used in practically all automotive applications by the Armed Forces. Complaints were rare and could usually be traced to improper lubricating techniques. This laboratory is unconvinced that this grease will not perform satisfactorily in the newer vehicles based on cursory tests in local equipment. Like individual owners, however, the government must follow the manufacturers' recommendations in order not to invalidate the guarantees.

If a reasonable cost grease of this type can be developed with extended temperature range, improved

extreme pressure characteristics and increased shear stability, perhaps the auto manufacturers' misgivings could be alleviated. Then the government, as well as service station proprietors, could return to a single specification grease satisfactory to all.

One of the best prospects for achieving these objectives is believed to be with the complex soap thickeners. Amott and McLennon<sup>(1)</sup> discussed barium and calcium acetate-soap complex greases with dropping points in excess of 400°F. in 1951. These greases contained about 20% thickener and about an SAE 30 oil and therefore would not satisfy military low temperature requirements. J. Panzer<sup>(2)</sup> discussed the nature of this type grease in 1961. Polishuk,<sup>(3)</sup> in 1962, discussed physical and chemical properties of complex soap greases. He states that complex barium soap greases have high dropping points, good water resistance and average mechanical stability but have poor temperature reversibility and high manufacturing costs. Calcium complex soap greases, while having high dropping points also have poor temperature reversibility and resistance to shear breakdown. Trivalent aluminum can form a complex soap with two dissimilar acids as well as retaining a hydroxyl group which has been shown to be desirable in a soap for thickening grease. Aluminum complex soap greases made by Polishuk were claimed to have good temperature reversibility, good water resistance, bleeding and shear stability characteristics in addition to requiring a lesser amount of thickener due to the smaller size of the individual fibers.

On the basis of this information the latter would seem to offer the best prospects of the soap complexes in producing an MIL-G-10924B type grease with a broader temperature range.

#### PROCEDURE

The oil used in all grease formulations in this report was one typical of that used in MIL-G-10924B greases. Following are the characteristics of this oil:

Viscosity @ 100°F., SUS	74
Viscosity @ 210°F., SUS	36
Viscosity Index	41
Pour Point, °F.	-60

The commercial fatty acid used throughout all the formulations in this report had the following composition:

Myristic Acid	0.4%
Palmitic Acid	29.2%
Stearic Acid	69.5%
Oleic Acid	0.9%
Saponification No.	204-207

## RESULTS AND DISCUSSION

Attempts to make aluminum soap greases directly with gelatinous aluminum hydroxide and fatty acids or by double decomposition of the sodium, potassium or ammonium soap with aluminum sulfate or hydroxide met with only partial success. In situ and preformed soap methods were tried. For example, preformed thickeners were prepared by first forming the ammonium soap or complex (1-1 weight ratio) of each. These were reacted with diluted gelatinous aluminum hydroxide, washed and dried. Attempts to make greases with each of these compounds resulted as shown in Table I.

Even at these high thickener concentrations, the consistencies or dropping points were unsatisfactory. Incomplete reactions may be a factor but there was no known information on which to base comparisons for citrate or tartrate complexes. The fast cooled aluminum acetate-soap complex grease showed excessive syneresis.

It was brought to our attention that aluminum alcoholates can be used to obtain aluminum soaps and complexes directly with the formation of side products easily removable by heating.<sup>(4)</sup> The following aluminum acetate-soap complexes were formed by dissolving the acids in cyclohexane and reacting with a cyclohexane solution of aluminum isopropylate either in the oil (in situ) or out of the oil (preformed). The latter could be added to the oil as a dispersion or with the cyclohexane evaporated. The greases were also formed under a variety of heating and cooling conditions as indicated in Table II.

Considerable dropping point improvement over greases thickened with straight aluminum soap is evident with those thickened with 1-1 weight ratio aluminum acetate-soap complex (3-1 molar ratio). Batch #7 is thickened with the 1-1 molar ratio complex which is a stiffer grease at the expense of a lower dropping point.

Hotten and Echols<sup>(5)</sup> name an aluminum benzoate-stearate complex as one of the preferred thickeners in their patent on "Complex Basic Aluminum Soap Greases." The example cited used 10% of the approximately 1-1 molar ratio complex thickener in 485 SUS @ 100°F. oil to get

ALUMINUM COMPOUNDS VIA ALUMINUM HYDROXIDE AS GREASE THICKENERS

TABLE I

Batch	Thickener	Thickener %	Method of Cooling	Penetration, ASTM		Dropping pt. of.
				Unworked	Worked	
1	Al Soap	20	Slow Fast	306 174	444 266	188 173
2	Al Acetate- Soap Complex	15	Slow Fast	215 171	322 197	206 -
3	Al Citrate- Soap Complex	20	Slow Fast			Too Fluid
4	Al Tartrate- Soap Complex	20	Slow Fast			Too Fluid

TABLE II

ALUMINUM ACETATE-SOAP VIA ALUMINUM ISOPROPYLATE AS GREASE THICKENERS

Batch	Acetic F.A. Ratio	Thickener %	Thickener Incorporation	Stirred to of.	Static Cooling From of.	Static Cooling Method	Penetration		Dropping pt. of.
							Unwkd.	Wkd	
5	1-1	20	Preformed w/cyclohexane	300	240	Slow Fast	190 190	286 309	351 -
6	1-1	10	"	300	240	Slow Fast	324 324	346 358	299 291
7	1-3	10	"	300	240	Slow Fast	286 286	292 281	205 184
8	1-1	15	"	300	240	Slow Fast	346	365	334
					Cooled quickly to 240°F.			Too Fluid	
11	1-1	15	Preformed - no cyclohexane	380	280	Slow Fast	268 249	336 283	262 218
12	1-1	15	In situ w/cyclohexane	380	300	Slow Fast	204 230	290 249	306 314

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"unctuous soft grease" with an ASTM dropping point of 400 + °F. The formulations shown in Table III were prepared with this type complex obtained by reacting the acids with aluminum isopropylate using cyclohexane as the solvent.

For the 1-1 weight ratio aluminum benzoate-soap complex grease (2-1 molar ratio), it was advantageous to process at the higher temperature while the opposite was true for the 1-3 weight ratio greases (2-3 molar ratio). The presence of cyclohexane during the formation of a grease reduced thickening efficiency.

As shown in Table IV, higher dropping points and stiffer consistencies were obtained with the 1-3 weight ratio complex (2-3 molar ratio) than with the 1-1 weight ratio complex (2-1 molar ratio) of aluminum benzoate-soap. Both were performed with aluminum isopropylate in cyclohexane and the solvent evaporated before incorporating into the oil. However, as the 1-3 ratio thickener concentration was reduced to 5%, where a more desirable consistency was obtained, the dropping point was considerably lower.

The additives used with the greases shown in Table V had little effect on their consistencies. There was some reduction in dropping points at the 5% thickener concentration. The oxidation stability and copper corrosion resistance of the uninhibited grease is not bad but is improved by the additives. Too much additive can cause etching of the copper as shown for Batch 30, however. These additives are inadequate for inhibition at 250°F. or rust prevention by ASTM Method D1743.

Batch 33 was mixed as a one kilogram batch in a Model "4" KitchenAid Mixer, the metal bowl of which was heated with a Glas Col mantle. This is in place of the beaker - variable speed stirrer - hot plate equipment used for the 100-200 gram batches prepared previously. Other MIL-G-10924B tests were run on this batch as shown in Table VI. These tests were made on the grease about 3-1/2 months after it was forced through a 200 mesh stainless steel screen.

The grease softened somewhat after storage but showed no oil bleeding. The water and work stability portions were very soft when removed from test but recovered sufficiently after the one hour temperature stabilization period to pass the specification requirements. The apparent viscosity was only a shade higher than that required by the specification.

**TABLE III**  
**ALUMINUM BENZOATE-SOAP COMPLEX GREASES-15% THICKENER**

Batch	Benzoic- F.A. Ratio	Thickener Incorporation	Stirred to °F.	Static Cooling From °F.	Static Cooling Method	Penetration, ASTM		Dropping Pt. °F.
						Unworked	Worked	
15	1-1	In situ with cyclohexane	300	240	Slow	Fluid		-
			Cooled quickly to 240°		Fast	Fluid		-
83	1-1	"	300	300	Slow	Fluid		-
					Fast	185	283	504
18	1-1	Preformed - no cyclohexane	430	370	Slow	140	185	455
					Fast	159	197	-
87	1-1	"	300	300	Slow		Semi-fluid	
					Fast		Semi-fluid	
					Slow	144	172	499
20	1-3	"	450	320	Fast	118	163	-
					Slow	110	137	570
86	1-3	"	300	300	Fast	159	152	516
					Slow		Semi-fluid	
85	1-3	In situ with cyclohexane	300	300	Fast		Semi-fluid	

**TABLE IV**  
**PREFORMED ALUMINUM BENZOATE-SOAP COMPLEX GREASES-**  
**AFFECT OF THICKENER CONTENT**

Batch	Benzoic- F.A. Ratio	Thickener %*	Stirred to °F.	Static Cooling From °F.	Static Cooling Method	Penetration, ASTM		Dropping Pt. °F.
						Unworked	Worked	
18	1-1	15	430	370	Slow	140	185	455
					Fast	159	197	-
19	1-1	10	450	300	Slow	204	238	400
					Fast	163	236	-
84	1-1	5	440	300	Slow		Fluid	
					Fast		Fluid	
					Slow	144	172	499
20	1-3	15	450	320	Fast	118	163	-
					Slow	157	200	510
22	1-3	10	420	300	Fast	153	195	-
					Slow	140	212	506
23	1-3	7.5	400	300	Fast	161	212	-
					Slow	213	272	406
24	1-3	5	400	320	Fast	200	266	406

\*Thickener content somewhat higher due to oil evaporation.

TABLE V  
 PERFORMED ALUMINUM BENZOATE-SOAP COMPLEX GREASE (1-3 RATIO) -  
 AFFECT OF ADDITIVES ON GREASE CHARACTERISTICS

Batch	Thickener %	Additives *	Penetration, ASTM		Dropping pt. of.	Oxidation, psi drop		(2) Cu Corrosion	(3) Rust Prevention
			Unworked	Worked		400 hrs @ 210°F.	200 hrs @ 250°F.		
24	5	None	213	272	406	20	56	Pass	-
29	10	0.5% PAN 0.5% NPA	159	199	530	-	-	-	-
31	7.5	"	161	223	504	-	-	-	-
30	5	"	200	272	373	6	20	Cu Etched	-
32	5	0.25% PAN 0.25% NPA	198	272	375	4	16	Pass	-
33	5	"	206	258	386	-	-	Pass	Fail

\* PAN - phenyl- $\alpha$ -naphthylamine  
 NPA - nonyl phenoxy acetic acid

- (1) ASTM Method D942
- (2) ASTM Method D1261
- (3) ASTM Method D1743

TABLE VI

COMPARISON OF BATCH 33 WITH MIL-G-10924B REQUIREMENTS

<u>Test</u>	<u>Spec.</u>	<u>Batch 33</u>
Penetration, ASTM (screened thru 200 mesh)		
Unworked, initial	265-295	264
Unworked after 3-1/2 mo.	-	277
Worked, initial	265-295	273
Worked, after 3-1/2 mo.	-	303
Work Stability, penetration change	-25 to +45	+27
Water Stability, penetration change	-10 to +45	+19
Apparent Viscosity @ -65°F.		
25 reciprocal seconds	11,500-17,500	17,800
100 reciprocal seconds	Max. 8,500	8,800
Separation	Max. 6.0	8.0
Evaporation	Max. 10.0	5.6

In order to determine if the 1-3 weight ratio of aluminum benzoate to aluminum soap was the optimum proportion for preparing this type aluminum complex soap thickened grease, the batches shown in Table VII were made with preformed, solvent-free thickeners as indicated.

It appears that some excess of the aluminum soap component in the complex is necessary for thickening and that the optimum proportions are near the 1-3 weight ratio (2-3 molar ratio) of aluminum benzoate to aluminum soap.

All the previous aluminum benzoate-soap thickeners have been based on the reaction of two acid radicals with each aluminum ion. Others<sup>(6)</sup> have found that only under special conditions is it possible to obtain the tri-acid aluminum compounds and that a hydroxyl radical is advantageous in the formation of greases. To check this, Batch 39 was made with the thickener based on the reaction of three acid radicals for each aluminum ion. No grease was formed with a 5% thickener concentration under the conditions used in formulating the other greases.

TABLE VII

**ALUMINUM BANZOATE-SOAP COMPLEX GREASES-  
EFFECT OF VARYING BENZOATE-SOAP RATIOS (BASED ON 2 ACIDS/Al)**

Batch	Thickener %	Al Benzoate-Al Soap Ratio		Penetration, ASTM		Dropping Pt. °F.
		Weight	Molar	Unworked	Worked	
34	5	1-1	2-1		Fluid	-
35	10	3-1	6-1		Fluid	-
36	5	1-6	1-3	268	309	278
37	5	1-2	1-1		Fluid	-
39	5	1-3	2-3		Fluid	-
		(3 Acids/Al)				
40	5	1-3	2-3	193	252	483
45	5	100%	Al Soap	343	372	143
*52	5	1-3	2-3	230	324	238

\* Al benzoate and Al soap preformed separately and added to oil.

Aluminum soap and aluminum benzoate were preformed separately for Batch 52 using ingredients identical to those of Batch 40. The thickener did not completely dissolve, even at 500°F., which enhances the theory that complexes are formed in the co-precipitation method with properties unlike either of the component compounds alone.

The effect of using solvents other than cyclohexane for in situ preparations of aluminum benzoate-soap complex greases was determined as shown in Table VIII.

The selection of a solvent is shown to be very important for in situ preparations and isopropanol or ethyl ether was found to work well with this particular complex.

An in situ preparation of a larger batch of this same formulation grease using isopropanol was made in a Model "4" KitchenAid Mixer which resulted in a much lower dropping point than that of Batch 49. The possibility of the formation of larger thickener particles during the reaction of Batch 54 because of the slower stirring speed was considered as suggested by Chessick and Zettlemyer's work on "Basic Factors in the Formation and Stability of Nonsoap Greases." (7) This was tested by reacting the thickener ingredients in the equipment used for Batch 49 before transferring this premix to the

TABLE VIII

IN SITU PREPARATION OF 5% ALUMINUM BENZOATE-SOAP  
COMPLEX GREASES (1-3 WEIGHT RATIO)-SOLVENT EFFECTS

Batch	Solvent Used	Static Cooling Method	Penetration, ASTM		Dropping Pt. °F.
			Unworked	Worked	
46	cyclohexane	Slow		Fluid	
		Fast		Fluid	
47	ethyl ether	Slow	182	275	484
		Fast	236	275	372
48	chloroform	Slow		Fluid	
		Fast		Fluid	
49	isopropanol	Slow	196	249	497
		Fast	208	262	499
50	toluene	Slow	222	309	363
		Fast:	226	298	319

KitchenAid Mixer for further processing. This explanation appeared to be valid until larger kilogram batches were made later as shown in Table IX.

The degree of agitation or concentration of reactants in the premix oil seemed to make little difference in the consistencies or dropping points of the larger batches. The high dropping points of Batches 49 and 55 are suspect. High evaporation loss of the light oil in these greases during the determination of dropping points could produce some erratic results. This is investigated later as shown in Table XII.

To determine the effect of other organic acids as complexing agents in the in situ preparation of 5% aluminum complex soap greases using isopropanol as the mutual solvent, the batches shown in Table X were made. All were cooled slowly in a preheated oven from 300°F.

The greatest improvement in dropping point was achieved with succinic, itaconic, suberic, azelaic and sebacic acids - all dibasic acids. None, in these ratios at least, had the thickening efficiency or high dropping point of the aluminum benzoate-soap complex grease of the same thickener concentration.

Three of these dibasic acids (succinic itaconic and azelaic), which produced the most improvement in dropping point over the straight aluminum soap grease, were each

TABLE IX

IN SITU PREPARATION OF 5% ALUMINUM BENZOATE-SOAP COMPLEX GREASES  
(1-3 WEIGHT RATIO W/ISOPROPANOL)-EFFECT OF REACTION CONDITIONS

Batch	Batch Size, gm	Premix Reaction Medium	Penetration, ASTM		Dropping pt. of.
			Unworked	Worked	
49	100	Beaker-stirrer in 2/3 of oil	196	249	497
54	200	KitchenAid Mixer in 2/3 of oil	225	298	375
55	200	Beaker-stirrer in 2/3 of oil -finished in KitchenAid	208	279	487
*88	1000	Small stirrer in KitchenAid bowl w/2/3 of oil-finished in KitchenAid	268	274	398
*89	1000	Waring Blender in 1/2 of oil -finished in KitchenAid	268	272	398
*90	1000	Waring Blender in 1/3 of oil -finished in KitchenAid	266	272	378
*91	500	Waring Blender in 2/3 of oil -finished in KitchenAid	252	275	398

\* These greases passed through 200 mesh screen.

TABLE X  
ALUMINUM ORGANIC ACID SALT-SOAP COMPLEXES  
(1-3 RATIO) AS GREASE THICKENERS

Batch	Complexing Acid	Formula	Penetration, ASTM		Dropping pt. of F.
			Unworked	Worked	
56	Acetic	$\text{CH}_3\text{COOH}$	334	418	177
57	Citric	$\text{C}_3\text{H}_6(\text{COOH})_3$		Fluid	-
58	Tartaric	$\text{C}_2(\text{OH})_2(\text{COOH})_2$		Fluid	-
59	Caproic	$\text{C}_5\text{H}_{11}\text{COOH}$	249	316	178
60	Malonic	$\text{CH}_2(\text{COOH})_2$		Fluid	-
61	Succinic	$\text{C}_2\text{H}_4(\text{COOH})_2$		Fluid	-
62	None	-	322	366	375
*63	Itmellic	$\text{C}_5\text{H}_9(\text{COOH})_2$	234	309	175
*64	Lactic	$\text{C}_2\text{H}_4(\text{OH})\text{COOH}$	246	292	173
*65	Adipic	$\text{C}_4\text{H}_8(\text{COOH})_2$		Fluid	-
73	"	"	221	264	187
66	Itaconic	$\text{C}_3\text{H}_4(\text{COOH})_2$	362	376	-
67	Ethylbuteric	$\text{C}_3\text{H}_6(\text{C}_2\text{H}_5)\text{COOH}$	309	339	378
68	Ethylhexoic	$\text{C}_5\text{H}_{10}(\text{C}_2\text{H}_5)\text{COOH}$		Fluid	-
69	Suberic	$\text{C}_6\text{H}_{12}(\text{COOH})_2$	196	294	274
70	Pelargonic	$\text{C}_8\text{H}_{17}\text{COOH}$	332	395	173
71	Azelalic	$\text{C}_7\text{H}_{14}(\text{COOH})_2$	215	264	346
72	Sebacic	$\text{C}_8\text{H}_{16}(\text{COOH})_2$	364	336	240

\* 64 hours @ 300°F. before slow cooling compared to 1-4 hours for others.

reacted with benzoic acid and aluminum isopropylate in situ with isopropanol to yield complexes which consisted of one part aluminum benzoate to three parts aluminum salt of the dibasic acid by weight. In a 5% thickener to oil concentration, none of the three formed a grease.

Since aluminum salts of benzoic and certain dibasic acids complexed with aluminum soap produced higher dropping point greases, thickeners were tried consisting of aluminum complexes of one part benzoic acid to three parts of a combination of dibasic and fatty acids. The latter was one part dibasic to three parts fatty acids. Table XI shows the properties of these formulations prepared in situ with isopropanol in the same manner as the preceding batches.

These formulations are certainly no improvement over the greases thickened with aluminum benzoate-soap complex although higher thickener concentrations might help.

The low dropping point of Batch 33 and, subsequently, Batches 53 and 54 compared to the near 500°F. dropping points of Batches 29, 31 and 49 was a matter for concern. It is believed that the relatively slow temperature rise of ASTM Method D566 is conducive to the evaporation of the light oil of greases of this type resulting in abnormally high dropping points. To check this contention some of the high dropping point batches were also run by a proposed ASTM method involving the insertion of the sample into a fixed temperature bath (aluminum block) which enables the grease to drop in approximately five minutes with a minimum of oil volatilization. These results are shown in Table XII.

The figures in parentheses in the fixed temperature column are probably truer indications of the temperature below which the grease would be serviceable. The dropping point in this column is the thimble temperature at the time of drop plus  $\frac{2}{3}$  of the difference between this temperature and the bath temperature as specified by the proposed method. A higher fixed bath temperature would be necessary to obtain dropping points on Batches 49 and 86.

Extensive working in a motorized grease worker (ASTM Method D217) is often used for comparing shear stabilities of greases. Batch 88 grease, a 5% aluminum benzoate-soap complex grease prepared as shown in Table IX, was worked in this manner with results as shown in Table XIII.

ALUMINUM COMPLEXES OF BENZOIC, FATTY AND DIBASIC ACIDS  
AS THICKENERS FOR GREASE

TABLE XI

Batch	Thickener %	Complex Composition	Static Cooling Method	Penetration, ASTM		Dropping Pt. Of.
				Unworked	Worked	
77	5	25% Al Benzoate	Slow	Semi-fluid		427
		56.2% Al Soap 18.8% Al Succinate	Fast	Fluid		-
78	5	25% Al Benzoate	Slow	Semi-fluid		348
		56.2% Al Soap 18.8% Al Itaconate	Fast	Semi-fluid		452
79	5	25% Al Benzoate	Slow	Fluid		-
		56.2% Al Soap 18.8% Al Azelate	Fast	Fluid		-

TABLE XII

**DROPPING POINTS OF ALUMINUM BENZOATE-SOAP (1-3 RATIO)  
THICKENED GREASES**

<u>Batch</u>	<u>Thickener %</u>	<u>ASTM D566 Dropping Pt. F.</u> <u>Previous</u>	<u>Current</u>	<u>450 F. Fixed Bath Temp.</u>
29	5	530	528 (520)	436 (360)
31	5	504	393 (364)	407 (360)
40	5	497	493 (474)	No Drop (394)
86	15	530	514 (490)	No Drop (None)

( ) Temperature at which drop first appeared at thimble orifice.

TABLE XIII

**SHEAR STABILITY OF A 5% ALUMINUM BENZOATE-SOAP  
COMPLEX GREASE**

<u>Batch #88</u>	<u>ASTM Penetration</u>	<u>Temperature, °F.</u>
Unworked	266	84
Worked, 60 cycles	296	84
1000 cycles	315	93
10,000 cycles	357	101
100,000 cycles	366	102
Recovery, after 1/2 hour	319	95
after 2 hours	295	77
after 24 hours	277	80

The stability of Batch 88, which is representative of the other greases of identical composition, is considered to be good under this type shear stress.

The good basic properties of these aluminum benzoate-soap complex greases offer a basis for the development of a grease which will surpass the requirements of Military Specification MIL-G-10924B. Further improvements can conceivably be achieved through the use of homologues of benzoic acid, the use of oils and fatty acids, other than those utilized in this work and the use of additives to overcome certain deficiencies.

## LITERATURE REFERENCES

1. Amott, Earl and McLennan, L. W., "Complexes in Lubricating Oil Greases," NLGI Spokesman 14, 12, March 1951.
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LIST OF PRIOR REPORTS

<u>RIA Lab. No.</u>	<u>Date Issued</u>	<u>Title</u>
59-1254	7 May 1959	Dimethyldioctadecyl Ammonium Bentonite as a Thickener for Automotive and Artillery Grease
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ALUMINUM COMPLEX SOAPS AS THICKENERS FOR MULTI-PURPOSE GREASE, By Max T. Fisher

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Aluminum complex soaps were evaluated as potential thickeners for greases which would meet the low temperature requirements and exceed the upper temperature limitations of Military Specification MIL-G-10924B Grease, Automotive and Artillery. The reaction of aluminum isopropylate with mixtures of fatty acids and other organic acids in a mutual solvent is the preferred method of forming these complexes. Of the 17 different lower molecular weight organic acids complexed with an aluminum soap, an aluminum benzoate-soap complex showed the best thickening efficiency and highest dropping

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2. Greases-Physical Properties
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