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TECHNICAL REPORT

DEVELOPMENT OF A GEL-RESISTANT
PRESERVATIVE OIL

By

R. LeMar

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PEQUA Project Order No. 8030-4230.1-30-50801-01

OCO, R and D Branch Project No. -

Department of the Army Project No. -

Report Number 61-999

Copy Number -

CLS -

Date 10 March 1961

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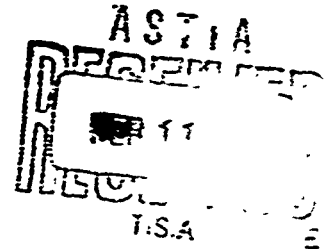
Rock Island Arsenal Laboratory Report No. 61-999 entitled,

"Development of a Gel-Resistant Preservative Oil,"

by R. LeMar, 10 March 1961

Page 9, paragraph 3; lines 3, 5, 6 and page 12, paragraph 3;
lines 1, 5, 6, 7: Substitute "barium dinonyl naphthalene
sulfonate" for "barium petroleum sulfonate."

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DEVELOPMENT OF A GEL-RESISTANT PRESERVATIVE OIL

By

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10 March 1961

PEQUA Project Order
No. 8030-4230.1. 21-5601-01

Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

A larger quantity of RIA Blend 42a as defined in RIA Report No. 59-3003 was needed. New, larger batches of additives were obtained which when used in this blend resulted in pronounced thickening of the oil.

Gel test methods as described in the above report were modified to more precisely discern thickening tendencies in these oils.

In order to determine the cause of the thickening of the 42a blend, studies were carried out on a series of blends, wherein systematic changes were made in concentrations of certain additives suspected of causing this thickening. It was found that three of the additives used in the above blend contributed to oil thickening. These were the fatty amine oleate, oleic acid and microcrystalline wax. A combination consisting of lanolin and alcohol improved the gel-resistance of the oil blend.

Corrosion resistance tests were carried out on these new blends to determine the effect of formulation changes upon this property. The Humidity Cabinet-Ultraviolet Light protection potential of this type of blend was lowered slightly by additive changes designed to improve the viscosity stability.

RECOMMENDATIONS

Gel tests for oils (hereafter called Accelerated Viscosity Stability tests) should include viscosity determinations in addition to visual evaluation of oil flowability. Furthermore, such tests for oils should include cyclic exposure at temperatures from -40°F to 77°F to determine the oil's ability to recover its fluidity at room temperature after hard freezing.

Lanolin-alcohol combinations can be used to improve the viscosity stability of highly inhibited formulations.

The improved formulation described in this report should be given pilot storage tests.

Further studies should be made to improve the stability against ultraviolet light protection of these blends, and to determine what characteristics of the microcrystalline wax additive are related to induction of thickening in these blends during storage.

DEVELOPMENT OF A GEL-RESISTANT PRESERVATIVE OIL

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DEVELOPMENT OF A GEL-RESISTANT PRESERVATIVE OIL

OBJECT

To determine what additives in RIA Blend 42a⁽¹⁾ were producing oil gelling and how this could be corrected.

To develop viscosity stability tests for oil formulations used for long-term storage of machine tools.

To determine the effect of increased viscosity stability upon protection test results.

INTRODUCTION

The Production Equipment Agency (hereinafter referred to as PEQUA) desired to evaluate the potential usefulness of RIA Blend 42a (RIA Report No. 59-3003⁽¹⁾) in pilot tests on machine tool storage. Since the estimated amount required was 25 gallons, larger, new samples of the additives used for this formulation were obtained. A small amount of the RIA blend was made from the new batches of additives in order to check the properties of the formulation using these new materials. The viscosity of this blend was checked after 2 gel tests (method as described in RIA Report 59-3003). It was found that in room temperature storage the material changed from a liquid to a nonflowing gel after four months. Similarly the viscosity @100°F showed a 119% increase after two accelerated viscosity stability tests.

A quantity of RIA Blend 42a material which had been retained from the original work, showed only a 3% increase in viscosity after 12 months of room temperature storage. Comparison of these two facts indicated that one or more of the additives ordered in large quantity for the PEQUA blending program, were at fault.

PEQUA's proposed use of this RIA formulation was primarily for internal machine tool preservation. Therefore, the possibility of gelling had to be eliminated. If such a preservative gelled during its presence in the machine tool, the machine might have to be disassembled for cleaning. In that case the use of this RIA blend would have no advantage in convenience of use as compared with hard-film preservatives.

In view of these facts, it was decided to determine which additives were producing the oil blend thickening, and possible methods of correcting this phenomena. In addition it appeared desirable to refine the gel test method originally

defined in RIA Report No. 59-3003 so as to evaluate more thoroughly long-term preservative oils for any latent gelling tendencies.

The effect of increased viscosity stability on protection tests also needed to be considered since superior corrosion-prevention properties were a key property for these oils.

PROCEDURE AND RESULTS

A. Test Methods Employed

The original 'gelling test' (hereafter referred to as the Accelerated Viscosity Stability Test), as described in RIA Report No. 59-3003, used the following time-temperature exposure sequence:

1. 2 hrs. @130°F
2. 2 hrs. @40°F
3. 2 hrs. @130°F
4. 2 hrs. @40°F
5. 8 hrs. @130°F
6. 16 hrs. @77°F
7. 64 hrs. @40°F
8. 4 hrs. @77°F

Thereafter the tube containing the oil sample was tilted and the oil observed to see whether it still flowed freely.

It appeared desirable to measure the oil viscosity in addition to merely observing the flowability. A viscosity determination @100°F was added to the gel sequence.

As the work of evaluating oil thickening continued, it appeared desirable to measure the oil's ability to recover after exposure to temperatures as low as -40°F.

Oil was cycled as follows for this test (hereafter called the Low Temperature Stability Test).

1. 24 hrs. @-40°F
2. 24 hrs. @77°F
3. 24 hrs. @0°F
4. 24 hrs. @77°F

The viscosity of the material @100°F was then determined.

In addition to these two tests, a room temperature storage test was conducted. The viscosity of the blends was determined during storage at intervals of 3-10 days.

B. Examination of RIA 42A Blend Components

Larger quantities of all the necessary additives and base oils were ordered for preparation of the 25 gallons of RIA Blend 42a (see Table I). When received, two of these additives differed in color and appearance from the small samples of the same additives used in the original experimental work. These were a 1,3 Propylenediamine diolate derivative of animal fat (hereafter referred to as POF) and the microcrystalline wax. In addition, to these two materials, oleic acid was also evaluated since it was suspected of undergoing oxidation, thereby contributing to oil blend thickening. These additives were evaluated by blending a series of formulations wherein the concentrations of the additives in question were varied. The thickening tendencies of these blends were tested to determine what effect, changes in certain additive concentrations had on this property.

1. Effect of POF

The POF contained a voluminous, white precipitate suspended in the yellow, oily liquid. None of the experimental sample used in the original compounding work remained, therefore, no direct comparison could be made, however, it was recalled that the original sample of POF, also a yellow, oily liquid, had contained no such suspension. In a comparison of POF effects, a sample of POF was centrifuged to remove the white colored contaminant. Two formulations, ND-1 and ND-2 were prepared, containing 3 and 1.5 parts of POF respectively, as shown in Table I. Comparison of accelerated viscosity stability test results of blends No. 1 and 2 in Table II show that the material containing 3 parts of POF demonstrated a viscosity increase of 357% as compared with 30% for the blend containing 1.5 parts, both compared after 3 test sequences. The influence of the POF is also shown in the viscosity increase during room temperature storage wherein blend ND-1 containing 3.0 parts gave a 0.4" greater rate increase than blend ND-2 containing 1.5 parts of POF.

2. Effect of Microcrystalline Wax

The microcrystalline wax used in the original work (hereafter called Wax A) reported in RIA Report No. 59-3903⁽¹⁾ was a dark brown, opaque material. Its dropping point and viscosity values were respectively, 183.5°F and 18.3 centistokes @210°F.

The new sample of wax (hereafter called Wax B) was a yellow, translucent material with dropping point and viscosity values of 164.3°F and 13.5 centistokes @210°F, respectively. This wax demonstrated thixotropic behavior during its viscosity determination.

TABLE I
COMPOSITION OF OIL BLENDS

ND Blends	Parts By Weight										RIA 420	
	1	2	3	4	5	6	7	8	9	10		
<u>*Components</u>												
Macro-Wax A	-	-	-	-	3	-	-	-	-	-	-	3
Macro-Wax B	3	3	2	3	-	3	3	3	3	3	3	-
POP	3	1.5	1.5	3	3	3	1	-	-	-	-	3
Olivic Acid	1	1	1	1	1	.5	1	1	.3	.3	1	1
Di-2-ethyl hexyl sulfide	4	4	4	4	4	4	4	4	1.5	1.5	4	4
Propyl oleate	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
Barium sulfonate dispersion	10	10	10	10	10	10	10	10	9	9	10	10
Lanolin	-	-	-	-	-	-	2	-	1.5	1.5	-	-
t-Butyl alcohol	-	-	-	-	-	-	-	2.3	1	1	-	-
n-Heptyl alcohol	-	-	-	-	-	-	-	3.6	2	2	-	-
Phenyl salicylate	-	-	-	-	-	-	-	-	-	3	-	-
Ex:30 Oil**	-	-	-	-	-	-	-	-	-	-	3	-
Oil A	45	38	27	37	37	40	32	27	39.9	33	40	40
Oil C	10	38	50	37	37	35	43	49	43	43	40	40

*See Table III for further description of components.

**Base oil ratio was varied as needed, so as to maintain the viscosity of the blends near to that within the range of 150-200 centistokes @100°F.

TABLE II

VISCOSITY INCREASES AFTER VISCOSITY STABILITY TESTS

ND Blends	<u>% Viscosity Increase Per Sequence</u>								
	1	2	3	4	5	6	7	8	9
<u>Tests</u>									
Accelerated Visc. Stab.									
Sequence 1	17	27	9	50	20	30	21	23	6
" 2	75	31	12	119	-	36	-	42	10
" 3	357	30	-	-	-	38	-	-	12
Low Temp. Visc. Stab.									
	-	-	-	-	-	65	-	-	2
<u>% Viscosity Increase Per Day</u>									
Room Temp. Storage	1.4*	1**	1.2**	Gel in 4 months	-	2*	3*	-	0.2**

*Viscosity rate-of-increase rose between successive measurement intervals

**Viscosity rate-of-increase decreased between successive measurement intervals.

Two sets of tests were used to determine the effect of Wax B on oil thickening. The first compared 2 formulations, ND-3 and ND-2, containing respectively, 2 and 3 parts of Wax B. Examination of viscosity stability test results (see Table II) after 2 test sequences, show that ND-3 gave less than half the increase displayed by ND-2. This indicated that decreasing the amount of Wax B decreased the thickening potential of the oil blend. The second comparison was between the ND-4 and ND-5 blends. ND-4 contained 3 parts of Wax B while ND-5 contained 3 parts of Wax A. Examination of viscosity stability test results (see Table II) after one test sequence, show that the Wax A blend displayed less than half the viscosity increase produced in the Wax B blend. This indicated that Wax B was far more active than Wax A in producing thickening in these oil blends. Since the nature of the micro wax was related to oil thickening and its presence was essential for imparting desired Salt Spray protection properties to the material, these two waxes were further evaluated in regard to their physical properties which are listed in Table III.

The test results in Table III show that Wax A was a harder, higher melting material. The acidity test on Wax B indicated this material to contain additives or contaminants which produce a pronounced change in the pH level of distilled water. The appearance was also very different. The viscosity stability tests indicate that Wax A was more desirable in maintaining the viscosity stability of the blend. In addition, the darker color may also be desirable in reducing light access to a metal surface coated with the oil blend, thus decreasing any corrosion-stimulating effects of light.

3. Effect of Oleic Acid

It was suspected that oleic acid because of its unsaturated character, was being oxidized slowly in the oil blend thereby tending to thicken the formula. The fact that those oil blends which thickened frequently formed a firm film at the oil-air interface where air was available for the oxidation, tended to confirm this possibility.

Blend ND-4 was compared with Blend ND-6 wherein the oleic acid content was reduced by half, to 0.5 part. The Accelerated Viscosity Stability test results (see Table II) show that the ND-4 viscosity increased by a factor approximately three times that of the ND-6, both considered after two test sequences. In addition, the progressive increase in viscosity from test to test was much lower in ND-6. Thus, in two consecutive tests, the viscosity of ND-6 increased by factors of 30% and 38% whereas for ND-4, the viscosity increased by factors of 50% and 119%. These test results

TABLE III

DESCRIPTION OF COMPONENTS

1. 'Practical Grade' Chemicals

- | | |
|--------------------|--|
| a. POF | f. Barium dinonyl Naphthalene Sulfonate - (50% dispersion of neutral salt in coastal ext. mineral oil) |
| b. Oleic acid | |
| c. Propyl oleate | |
| d. t-Butyl alcohol | |
| e. n-Decyl alcohol | g. Phenyl Salicylate |

2. 'USP' Grade Chemical

- a. Lanolin

3. Characteristics of microcrystalline Waxes, A and B

<u>Test & Method</u>	<u>Wax</u>	<u>Result</u>
a. Dropping Pt. Op, ASTM No. D127-49	A	183.5
	B	164.1
b. Viscosity @210°F, cs ASTM No. D445-53T	A	18.6
	B	13.5 (thixotropic)
c. Penetration, mm. ASTM No. D1321-55T	A	1.5
	B	1.9
d. Acidity, pH change in dis- tilled water when boiled with 3% wax for 1/2 hr.	A	-0.2
	B	+1.2
e. Appearance Visual description	A	Dark brown, opaque
	B	Yellow, translucent

4. Characteristics of Base Oils A and B

<u>Test & Method</u>	<u>Oil</u>	<u>Result</u>
a. Viscosity @100°F ASTM No. D445-53T	A	19.2
	C	1050.
b. Volatility, % ASTM No. D972-56	A	2
	C	0.1
c. Failure Time, hrs. MFT-792 Humidity Cabinet	A	1
	C	4

indicated that oleic acid in concentrations above 0.5 part could significantly increase the rate-of-increase of viscosity from test to test.

C. Development of Gel-Resistant Blends

It was apparent that the following factors were related to the degree of gelling in these oil blends:

1. Nature and quantity of the wax.
2. Quantity of PCF.
3. Quantity of oleic acid.

It was desirable to maintain micro wax concentration at a 3% level since this additive was essential for the Salt Spray Protection properties of these oil blends. The presence of the PCF was not so essential protection-wise so this material was eliminated from the blend. It was also apparent that the oleic acid content should be reduced to 0.5% or lower.

In order to further stabilize the oil blend viscosity, the use of certain additives was investigated.

Lanolin was investigated since it was known that this material, dissolved in oil at concentrations of 5-10%, did not produce thixotropic behavior in its oil solution. It was assumed that if lanolin were present in combination with the micro-wax that when the wax crystallized at low temperatures, lanolin would tend to include itself in the crystal structure, so that after the temperature was raised the contaminated wax crystals would tend to break down and redissolve in the oil.

Alcohols of from 4-16 carbon atom chain lengths were also evaluated on the assumption that they would increase the solvent power within the oil to a degree that would prevent agglutination of the additives therein.

Blend ND-7 illustrates the effect of including two parts of lanolin in this type of formulation. ND-7 can be most closely compared, formulation-wise, to ND-2. It is seen from Table II that one viscosity stability test produces a greater thickening than in ND-2 containing no lanolin.

Blend No. ND-8 illustrates the effect of inclusion of a mixture of alcohols, t-butyl and n-decyl alcohol. Comparison with ND-2 in Table II shows greater thickening for this blend than for the blend containing no alcohol after two viscosity stability tests. This occurred even though

no POF was present. A combination of lanolin plus alcohols was used in Blend ND-9. In this formulation, the concentration of amine was reduced to 1.5% since protection tests on ND-8 and similar blends had indicated that the amine-alcohol combination at higher concentrations tended to cause the bulk oil film to be washed from a metal surface under high humidity conditions. In addition, no POF is present and the oleic acid content has been reduced to 0.3%. Table II indicates that this blend showed superior viscosity stability, giving a viscosity increase of 10 and 12 percent respectively after 2 and 3 tests. Its room temperature stability was good showing a total daily average increase of 0.2%. As indicated, the rate of room temperature increase, decreased with time of storage tending to approach a constant value. The material also showed good stability under exposure to very low temperatures, demonstrating no residue formation and only slight viscosity increases after freezing when reliquified at a temperature of 77°F. The change in formulation acted to increase the pour point of this material from +16°F (No. #42A) to +30°F.

D. Protection Tests

Both the HCUV and 20% salt spray tests were applied to Blend ND-9 as these tests are specified in RIA Report No. 59-3003(1). This blend passed the 48 hour salt spray requirement and the HCUV test requirement of 15 cycles. With regard to the HCUV test panels, light staining was produced on the panel surface. This was removable with chloroform.

In an effort to improve the HCUV protection, phenyl salicylate, an UV light absorber and corrosion inhibitor was substituted for part of the barium petroleum sulfonate in the blend. Previous unreported work had indicated a ratio of 2 to 1 for phenyl salicylate to barium petroleum sulfonate to be desirable so blend ND-9 was changed to include 3 gms of the former and 1.5 gms of the latter. This blend, designated as ND-10, passed the HCUV test with very little overall staining which was removable with chloroform.

The accelerated viscosity stability and salt spray protection tests were not completed on this blend.

The formulation sequence for these types of oil blends is as follows:

1. Mix the measured amounts of micro wax, lanolin, di-2-ethylhexyl amine, n-decyl alcohol and oil. Heat to 200°F with agitation until the wax is dissolved.

2. Cool to 150°F, and add the remainder of the ingredients, heating with stirring at 150°F for five minutes. Allow to cool to below 100°F, and then reheat to 150°F with stirring. Allow to cool to room temperature with stirring.

DISCUSSION

It was found that certain micro waxes, POF and oleic acid, all could contribute to oil blend thickening. Combinations of lanolin and alcohols were found to reduce this thickening characteristic. So as to ascertain such tendencies in accelerated tests, the accelerated viscosity stability and a low temperature recovery test were developed and used in the work covered by this report. The oil blends of increased viscosity stability were found to show slightly less protection potential in the HCUV test, but performed satisfactorily in the 20% salt spray test.

These factors are more fully discussed in the sections which follow below:

A. Viscosity Stability

The pronounced thickening that occurred with RIA Blend 42A (RIA Report No. 59-3003) when a second batch of additives was used, indicated the accelerated 'gel' test as described in the above report needed modification to more accurately discern thickening potential in oils. The modifications and additions in the method are included in detail in the Appendix to this report, paragraphs 1.5 and 2.2.

It can be noted therefrom, when comparison is made with the original method, that two major changes have been made.

1. The viscosity @100°F is determined after each test sequence and provision is made for determining whether an oil with thickening tendencies is likely to continue thickening until a gel state is reached.

2. A low temperature recovery test is used to reduce the possibility that crystallization or coagulation of thickening agent or other additive would not be reversible at ambient storage temperatures.

These oil blends were generally sufficiently thixotropic so as to require exposure at 100°F for one hour before constant viscosity values could be obtained.

B. Evaluation of Oil Blend Thickening

The second batch of additives that produced the pronounced gelling in RIA Blend 42A (RIA Report No. 59-3003) was evaluated in relation to the formulation with the following conclusions as indicated by the experimental work.

1. Comparative studies on waxes A and B indicate that a hard wax with an ASTM dropping point of 180°F or higher is desirable for maximum viscosity stability. However, further studies are needed to confirm this hypothesis. It would appear desirable to obtain a synthetic wax of known constitution in order to eliminate the variations that can occur in microcrystalline wax both in its nature and processing (bleaching, filtering, etc.).

Microcrystalline wax imparts good salt spray protection properties to blend ND-9 for which its required concentration is 3%. The desired properties for such a wax are presently defined as follows:

- a. ASTM Dropping Point (°F) - 160 to 190.
- b. Viscosity at 210°F (centistokes) - 14 to 20 with no thixotropic behavior.
- c. Additives or bleaching residues - none shall be present.
- d. Appearance and quality - the wax shall be filtered but not bleached and be opaque or dark colored.

2. The POF was a complex oleate resulting from the processing of animal fats. Besides the probability of variations in nature of the 'pure' product, it was apparent that storage could affect its composition. It was not used in the improved blend, ND-9.

3. It was found that practical grade oleic acid in excess of 0.5% could produce thickening in the presence of wax B. The recommended concentration in the improved blend was reduced to 10% of the wax content or 0.3%.

4. In order to impart viscosity stability to the blend after both low and high temperature exposure, it was indicated that a lanolin-alcohol combination could be used as follows:

- a. USP Grade Lanolin - at least 1/2 of the wax concentration.
- b. Practical Grade Alcohols - equal to or greater than the wax concentration.

In the modified blend, lanolin is used at 1.5% and a mixture of tert-butyl and n-decyl alcohol is used at 1 and 2% respectively.

5. It was observed that the alcohols interacted with the di-2-ethyl hexylamine to produce a washing away of the bulk oil film under high humidity conditions. Therefore, the amine concentration should be half of or less than the wax concentration. In the modified blend it was reduced to 1.5% for the practical grade amine.

6. The propyl oleate and barium petroleum sulfonate (practical grades) were both placed at 1.5 times the wax concentration or 4.5% (9% of 50% suspension). In order to improve HCUV test results, phenyl salicylate may be partially substituted for the barium petroleum sulfonate. Blends of the latter type using 3% phenyl salicylate and 1.5% barium petroleum sulfonate have shown improved results in the HCUV test, but other basic test data were not obtained (salt spray protection, viscosity stability, etc.).

LITERATURE REFERENCES

- i. LeMar, R., "Development of An Internal Preservative Oil For Machine Tools", Rock Island Arsenal Report No. 59-3003, 24 November 1959.

APPENDIX

PROPOSED QUALITY CONTROL TESTS AND REQUIREMENTS FOR AN INTERNAL PRESERVATIVE FOR MACHINE TOOLS

In order to facilitate the development and recognition of the proposed new long-term internal preservative oil for machine tools, a list of test methods and their requirements are listed below.

The tests and requirements are based on existing oil specifications and the properties of the best blends that have been prepared at this Laboratory.

This list can serve as a basis for a proposed Grade 2 oil to be part of Specification MIL-L-3150 in the event the oil is accepted as useful in Ordnance preservation. The list can be used by PRQUA to obtain the type of oil it desires for long-term internal preservation of machine tools.

The oil that meets the requirements of the following test list will be a somewhat heavier version of the present MIL-L-3150 oil with approximately two to three times the protection potential of that oil (as indicated by accelerated corrosion tests). It will be resistant to temperature and humidity cycling effects and moisture condensation.

In the event that it is developed and added to the present MIL-L-3150 specification as a "Grade 2" oil therein, it will be the first Ordnance oil specification to include a cyclic, accelerated corrosion test.

Section I - Test Types and Requirements

1. Requirements

1.1 Material - The lubricating oil, Long-Term Preservative, Heavy, covered by these tests shall be an oil or oil-like material, free from injurious ingredients affecting serviceability, with sufficient inhibitor added to meet the requirements of the following tests.

1.2 Viscosity - The Kinematic Viscosity at 100°F shall be 150 cs. minimum and 200 cs. maximum.

1.3 Pour Point - The pour point shall be +30°F maximum.

1.4 Volatile Matter - The percentage of volatile matter shall be 5% maximum.

1.5 Stability -

1.5.1 Bulk Oil - The oil shall show no visible separation or residue formation after the test specified in 2.2.1. The oil viscosity change shall not be greater than $\pm 20\%$.

1.5.2 Oil Film - The oil film shall show no visible separation or breakage and shall appear homogenous after the test specified in 2.2.2.

1.5.3 Accelerated Viscosity Stability - The oil viscosity determined at 100°F shall not change more than $\pm 5\%$ after one test sequence. Greater percentage increases in viscosity are permissible only, if during not more than 4 consecutive test sequences, the viscosity value shows a decreasing rate-of-increase from sequence to sequence. In all cases, the oil shall also flow readily after each test sequence when tests are carried out as specified in 2.2.3.

1.5.4 Low Temperature Viscosity Stability - The oil viscosity determined at 100°F shall not change more than $+5\%$ after one test sequence. Greater percentage increases in viscosity are permissible only, if during not more than four consecutive test sequences, the viscosity value shows a decreasing rate-of-increase from sequence to sequence. In all cases, the oil shall also flow readily after each test sequence when tests are carried out as specified in 2.2.4.

1.6 Corrosion - The copper strip shall show no evidence of green or black discoloration or exhibit any evidence of etching or pitting after the test specified in 2.1 at 212°F.

1.7 Corrosion Protection - The oil shall afford protection to steel panels when tested for a minimum of 15 cycles in the humidity cabinet-ultraviolet light test specified in 2.3. Two of three panels tested shall show no more than a trace of corrosion and any staining present must be removable with chloroform.

1.8 Removal - The oil used to coat the test panels shall be removable in 3 cycles at the conclusion of the test in 1.7, according to the test specified in 2.4.

1.9 Salt Spray Resistance - The oil shall afford protection to steel panels when tested for a minimum of 48 hours in the 20% Salt Spray (Fog) Cabinet as specified in 2.5. Two of three panels tested shall show no more than a trace of corrosion.

Section II - Test Methods

2. Test Methods

2.1 Physical and Chemical Tests - The following tests shall be made according to the method specified below.

<u>Tests</u>	<u>ASTM Method No.</u>
Viscosity*	D445-53T
Pour Point	D97-57
Volatile Matter	D972-56
Corrosion	D130-56

*Note: The oil is allowed to stand in the viscosity tube at 100°F for one hour. Consecutive determinations are then made until three such determinations yield values within 0.2 centistokes of one another. The average of these three values shall be considered the oil viscosity.

2.2 Oil Stability

2.2.1 Bulk Oil Stability

2.2.1.1 Procedure - A tall form, screw-top, quart jar shall be three-fourths filled with the oil and the lid tightly affixed. The jar of oil shall then be stored at 77°F for one year without agitation. It shall then be observed under a bright light for layer formation. Thereafter it shall be inverted 4 times at 5 second intervals and the lid removed and the oil poured from the jar. The jar is observed for signs of an insoluble residue. The viscosity @ 100°F is then determined on the sample if it is homogeneous.

2.2.2 Oil Film Stability

2.2.2.1 Procedure - A 2" x 3" x 1/8" polished steel panel prepared according to 2.4.1 shall be immersed in the oil one minute. Thereafter it is withdrawn and allowed to drain, without movement at 77°F for 24 hours. It is then observed under a bright light. If the oil on the panel surface shows signs of separation or the oil film is broken or very uneven, the oil shall be considered as failing this test. Mild rippling will be permitted.

2.2.3 Accelerated Viscosity Stability

2.2.3.1 Procedure - Place the oil sample into a standard pour point tube as specified in ASTM Method No. D97-57. Stopper the tube and expose to the following temperature cycle sequence:

<u>Test Sequence</u>	
<u>Temp. °F</u>	<u>Time (Hours)</u>
130	2
40	2
130	2
40	2
77	16
130	8
40	6
77	4

The pour point tube shall then be turned to a horizontal position. The oil shall flow freely to its new level within the tube within five seconds. The oil is then placed in the viscosity tube and allowed to stand therein for one hour at 100°F. Consecutive viscosity determinations are then made until three such determinations yield values within 0.2 centistokes of one another, which shall be averaged to give the oil viscosity.

2.2.4 Low Temperature Viscosity Stability

2.2.4.1 Procedure - Place the oil sample into a standard pour point tube as specified in ASTM Method No. D97-57. Stopper the tube and expose to the following temperature cycle sequence:

<u>Test Sequence</u>	
<u>Temp. °F</u>	<u>Time (Hours)</u>
-10	24
77	24
0	24
77	24

The four points shall then be turned to a horizontal position. The oil shall flow freely to its new level within five seconds. The oil is then placed in the viscosity tube and allowed to stand therein for one hour at 100°F. Consecutive viscosity determinations are then made until three such determinations yield values within 0.2 centistokes of one another, which shall be averaged to give the oil viscosity.

2.3 Corrosion Protection

2.3.1 Preparation of Test Panels - Three test panels, 2" x 4" x 1/8" with rounded edges, made from cold rolled steel (FS 1020) shall be cleaned with hot naphtha and rinsed in hot anhydrous methanol. The clean panels are then polished with 240 grit aluminum oxide or silicon carbide abrasive and immediately cleaned by the following method.

- a. Wipe face of the panels with clean surgical gauze.
- b. Spray the panels at an angle of 25° from the vertical with clean naphtha, using a paint spray gun.
- c. Immerse the panels in boiling naphtha for 5 to 10 minutes.
- d. Rinse in hot, anhydrous methanol.
- e. Desiccate the panels for a period of 1 to 2 hours at 77°F before use.

2.3.2 Procedure - Three test panels, prepared as specified in 2.3.1, shall be immersed in the oil for one minute and thereafter suspended on test racks and allowed to drain 24 hours at 77°F and 35% RH. The oil-coated panels are alternately exposed in a humidity cabinet (see 2.3.4) for 17 to 18 hours, then to ultraviolet light (see 2.3.4) for 5 hours, then to an environment at 77°F and 35% RH for 1 to 2 hours for a total of 15 test cycles. During non-work days the specimens shall remain in the humidity cabinet. Non-work days shall not be counted as part of the 15 test cycles. The work shall be scheduled so that at least 1 cycle is completed before the occurrence of a non-work day.

2.3.3 Panel Racks - These shall be constructed so as to be transferable between the humidity cabinet and ultraviolet light cabinet. In addition, shields shall be placed thereon to be directly above each test panel so as to protect the panels from drippage, etc. During the initial 24 hour drainage and during the test exposure, the oil-coated panels

are left mounted on these racks so as to disturb the panels as little as possible during the transfer from one condition to the other.

2.3.4 Ultraviolet Light Cabinet - An Atlas Twin Arc Cabinet or similar apparatus shall be operated under the following conditions.

- a. An alternating current of 125-145 volts at 15-18 amperes is applied to the arc during the burning period.
- b. No water spray is applied to the test surfaces of the coated panels.
- c. The test panels are located 12 to 15 inches from the arc and are rotated about the arc.
- d. The black panel temperature is $135 \pm 2^{\circ}\text{F}$.
- e. The apparatus is operated five days a week, five hours a day.

Note: The carbon assembly should not be cleaned when the oil-coated panels are suspended therein. Large particles of carbon, grit, etc., may be thrown onto the oil film resulting in penetration of the film and rapid corrosion of the metal surface thereabout.

2.3.5 Humidity Cabinet - The humidity cabinet shall be maintained at a relative humidity of 95-100% and at a temperature of $100 \pm 2^{\circ}\text{F}$. These conditions shall be maintained by directing a fresh, water vapor-saturated stream of air against a baffle plate in the cabinet, at a rate to give 1 to 1.5 changes of air per hour. Auxiliary heat is applied through a water layer at the bottom of the cabinet. The panels shall be suspended so that the plane of each panel is parallel to the direction of air flow.

2.3.6 Cleaning and Rating the Panels - The oil film is removed from the panels by rubbing with solvent-soaked tissues or cloths. The oil shall be considered as passing this test, if at the end of 15 cycles, 2 of 3 panels show no more than a trace of corrosion and no trace of erosion. A trace of corrosion is defined as not more than 3 rust spots, none larger than 1 mm in diameter. Erosion is defined as visible spots or areas where the polished surface has been dissolved away without rust formation. Rusting of the outer 1/4" of the panels or 1/2" around the holes used for suspending the panel during test shall not be cause for rejection. Light stains removable with chloroform shall be permitted.

2.4 Removal - After completion of the HCUV test described in 2.3, the oil film shall be removable by not more than 3 wiping cycles when tested according to paragraph 4.5.12 of Military Specification MIL-C-14201A, Grade 1.

2.5 Salt Spray Resistance

2.5.1 Preparation of Test Panels - Three test panels 2" x 3" x 1/16" with well rounded edges made from cold rolled steel (AISI 1020) shall be cleaned with hot naphtha and rinsed in hot anhydrous methanol. The panel surfaces are then abraded by sandblasting with sharp, dry, white sand, free from organic matter. The sand shall meet the following requirements, using test sieves conforming to specification RR-S-365.

Through No. 10 sieve, min. %	100
" " 20 sieve, max. %	10
" " 50 sieve, max. %	10

Following sandblasting, the panels are freed from dust by tapping the edges against a clean object. The panels are then rinsed with warm methanol, allowed to flash dry and further cleaned by the following method.

- a. Spray the panels at an angle at 25° from the vertical with clean naphtha, using a paint spray gun.
- b. Immerse the panels in boiling naphtha for 5-10 minutes.
- c. Rinse in hot, anhydrous methanol.
- d. Desiccate the panels for a period of 1-2 hours at 77°F before use.

2.5.2 Procedure Three test panels, prepared as specified in 2.5.1 shall be immersed in the oil for one minute. Immediately thereafter they are mounted on the salt spray test rack with a square section of filter paper (saturated with the test oil) between the oil-coated panel and the wooden rack back. The panels shall be allowed to drain 24 hours on the rack at 77°F and 35% R.H. Thereafter the panels (on the rack) are placed in the 20% salt spray (fog) cabinet for a period of 48 hours.

2.5.3 Panel Racks - These shall be wooden racks designed to expose one test surface of the oil-coated panel at an angle of 15° from the vertical. They shall be mounted in the salt spray cabinet so that the plane of the exposed surface is parallel to the principal direction of horizontal flow of fog through the cabinet.

2.5.4 Salt Spray Cabinet Operation - The 20% salt spray cabinet shall be operated in accordance with Federal Test Method Standard 791 - Method Number 4001.1.

2.5.5 Cleaning and Rating the Panels - The panels shall be rinsed successively in distilled water, acetone, and V&M naphtha. The oil shall be considered as passing this test if at the end of 48 hours, 2 of 3 panels show no more than a trace of corrosion. A trace of corrosion is defined as not more than 3 rust spots, none larger than 1 mm in diameter. Rusting of the outer 1/4" of the panels (1/2 inch from the top edge) shall not be cause for rejection.

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A larger quantity of RIA Blend 42a as defined in
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batches of additives were obtained which when
used in this blend resulted in pronounced
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Gel test methods as described in the above report
were modified to more precisely discern thickening
tendencies in these oils.

In order to determine the cause of the thickening
of the 42a blend, studies were carried out on a
series of blends, wherein systematic changes
were made in concentrations of certain additives
suspected of causing this thickening. It was
found that three of the additives used in the
above blend contributed to oil thickening.
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Corrosion resistance tests were carried out on these new blends to determine the effect of formulation changes upon this property. The Humidity Cabinet-Ultraviolet Light protection potential of this type of blend was lowered slightly by additive changes designed to improve the viscosity stability.

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FOR ERRATA

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THE FOLLOWING PAGES ARE CHANGES

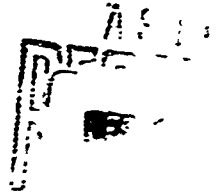
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AD 257552

Erratum Sheet for
Rock Island Arsenal Laboratory Report No. 61-939 entitled
"Development of a Gel-Resistant Preservative Oil,"
by R. LeMar, 10 March 1961

Page 9, paragraph 3; lines 3, 5, 6 and page 12, paragraph
lines 1, 5, 6, 7: Substitute "barium dinonyl naphthalene
sulfonate" for "barium petroleum sulfonate."

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Erratum Sheet for

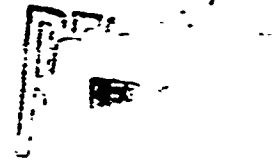
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END CHANGE PAGES