39-9 #1

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REMINGTON ARMS COMPANY, Inc.

Research Department

BRIDGEPORT, CONNECTICUT

Subject: Progress Report on the Improvement of [illegible]

Prepared by: A. A. Schilling

Approved: A. E. Buchanan, Jr.

Copy No. 23 Date March 31, 1939

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DATE..................

REQUEST FOR PROCESS OR MATERIAL CHANGE

PROJECT. Progress Report on Improvement of Rim Oil

The attached Research Department report describes results obtained under the above project. It is now believed advisable to make the changes indicated therein and this sheet requests the approval of those department heads affected.

Please date your signature.

Research Director..............................

Process Engineer..............................

Standards Department..............................

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Bridgeport, Conn.
March 31, 1939

TO: H. A. BROWN
ILION

FROM: A. E. BUCHANAN, JR.

We are submitting herewith a report by A. A. Schilling on Project MCB-1577-R, describing the development of an improved REM oil. A modification has been developed which provides excellent protection against rusting and which has 50% greater film strength than the base oil.

For ease of evaluation, all tests were made on plain steel. The amount of the project was insufficient to permit supplementary tests on browned steel. You may wish to do this at Ilion, or the tests can be made here under a Part II. The program followed in this investigation was based on the objectives outlined by Mr. Chase prior to writing of the project.

We would like to point out that this development lends itself very well to advertising and demonstration. Photographs can be used in advertising which will show the improvement over the base oil in rust protection, and our salesmen can carry steel panels for demonstration. The latter will be particularly effective if the salesmen prepare the panels them-

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selves and expose them to rusting conditions on their cars.

The effectiveness and low cost of the rust inhibitor used in the oil suggest its use in oils used in plant processing and for the protection of component parts in stores.

A one-quart sample of oil prepared according to the recommended formula is being sent to Mr. Chase.

R. E. Buchanan, Jr.
Director of Research
WOS

WOS:MD

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Copy of Rem [illegible] sent to Brown at Ilion - A [illegible] copy given to him at Bpt

copy # 4 - sent to J J Callahan 12-21-43
Copy 24 - sent to J J Callahan 5-31-39

PROGRESS REPORT

IMPROVEMENT OF REM OIL

INTRODUCTION

In preliminary tests, REM oil was found to be inferior to several competitive oils in preventing rust. Project MCB-1577-R was written and approved in May, 1938 with the object of developing an oil composition that would have improved rust-proofing properties, without lessening the lubricating and non-gumming qualities of the present oil.

The projected work has now been completed. This report describes the evaluation of a number of experimental formulas in comparison with competitive oils, and recommends the adoption of one of these compositions as a new and improved REM oil.

SUMMARY

The poor rust-proofing qualities of the present REM oil have been confirmed in tests at Bridgeport and at the Experimental Station. From a considerable number of experimental formulations a composition has been selected which appears to be satisfactory as to rust-proofing, stability on storage, ease of preparation and cost. The film strength of this composition is 50% greater than that of the current product.

The present base oil is used in the new composition. The marked improvement in rust-proofing is obtained through the addition of a rust inhibitor (oleic acid) to the oil. The present oil does not contain this type of ingredient. An ex-

treme pressure lubricant which in itself has some rust-proofing qualities is included in the proposed formula. This ingredient replaces the tricresyl phosphate now used, which has been found to have poor rust resistance. A perfuming agent has been added to give the oil a distinctive odor.

The formula of the recommended composition is as follows:

	Addition to Base Oil % by Volume
Base Oil	-
Oleic Acid (Rust inhibitor)	2.00
GD-162* (EP lubricant)	0.25
Oil of Rosemary (Perfume)	0.40

PATENT SITUATION

Information concerning the successful compositions developed in this work has been given to the Remington Patent Division and the possibility of obtaining patent protection is being studied.

EXPERIMENTAL

Preliminary Results

Preliminary tests made at Bridgeport in 1937 indicated that the present REM oil offers little protection to steel against rusting, whereas certain competitive gun oils were found to be quite effective in this respect. These tests were made by

*Du Pont "Ortholeum" One-Sixty-Two

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applying the oils to steel panels which were stored in the humidifier oven to accelerate corrosion.

At this time the Experimental Station was making a study of corrosion resistant treatments for steel and comparative tests on the various gun oils were made there, using a somewhat different technique in which water was caused to condense on the steel test panels. The results of these tests paralleled ours and confirmed our conclusions as to the inferior rust-proofing properties of REM oil. The results also showed that tricresyl phosphate, the EP (extreme pressure) lubricant used in REM oil exhibited no rust-proofing properties. The results of the Experimental Station test are given in Table I (data taken from Experimental Station Report ESP-38-28, Corrosion Resistant Treatments for Steel, by Dr. I. F. Walker, page 18).

The water condensation test used in this work at the Station is as follows: Polished 2" x 4" 20 gage steel panels are used as the test metal. A thin layer of oil is applied to the panel by means of a piece of cheescloth, and the panels fastened with wax to the under side of a section of plate glass. The glass is placed over a brass pan containing water at room temperature. By keeping the top of the glass covered with water, it is kept at a temperature slightly lower than the atmosphere, and condensation of water on the surface of the panels is effected. Rapid corrosion of untreated panels is obtained in this test.

The work on corrosion resistant treatments at the

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TABLE I

Water Condensation Tests

Preliminary work--prior to Project

Gun Oil Applied	Rating* 4 Days	10 Days	16 Days	1 Month
CONTROL (untreated)	6	4	3	1
"3 in One" Oil	9	8	8	5
RANGOON Oil (2 parts mineral oil) (1 part animal oil)	10	9	9	6
FIENDOIL (Lard oil with phenol)	10	10	10	8
FILM Oil (Contains solvent)	10	8	8	8
REM Base Oil	7	5	4	3
Present REM Oil (Base oil with 2% tricresyl phosphate)	7	4	3	2
Tricresyl Phosphate	7	5	4	2
REM Base Oil plus 2% di Lorol phosphate	10	8	7	3
REM Base Oil plus 2% mono and di Lorol phosphate	9	7	6	3

*Rating
10 = no visible rust
7-9 = scattered rust spots
3-6 = large areas of rust
0 = heavy continuous rust

Station suggested a number of materials which might give rust-proofing properties to the REM oil base, and Dr. Walker, still working under the Station project, formulated and tested a number of compositions which consisted of the base oil to which the more promising agents were added to the extent of 2% and 10%.

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The standard of comparison was FIENDOIL, which although not a conventional lubricating oil, is sold as a gun oil and was found in preliminary tests at Bridgeport and the Station (see Table I) to have good rust-proofing qualities. The results showed that some of the experimental compositions gave very good protection against rusting and indicated that a greatly improved REM oil could be formulated along these lines. A few of the most significant results obtained in this early work are given in Table II. For a complete tabulation see ESP-38-28, pages 19-20).

TABLE II

Water Condensation Tests

Initial formulations--prior to Project

Sample	1 Day	3 Days	1 Week	2 Weeks	3 Weeks	4 Weeks
CONTROL (untreated)	5	2	1	1	0	0
REM Oil Base	6	4	3	3	2	0
FIENDOIL	9	8	8	8	7	6
REM Oil Base plus 2% Coconut oil acids	10	10	10	9.5	*	5
REM Oil Base plus 2% Oleic Acid	10	10	10	9	9	9
REM Oil Base plus 2% Sulfonated Red Oil (Unneutralized)	10	10	10	9	9	9
REM Oil Base plus 2% GD-162 (mono plus di Lorol phosphates)	10	10	9.5	8	*	6

Ratings: Same as Table I
* = No test made

Work Under Present Project

To take advantage of the promising results obtained

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in the preliminary work referred to above, Project MCB-1577-R, appropriating $375 for the improvement of REM oil, was written and approved. The first work under the project consisted in making storage tests to determine the stability of the most promising compositions formulated at the Station, containing 2% and 10% of rust inhibiting agent. The rust inhibitors contained in these samples were coconut oil acids, oleic acid and sulfonated Red Oil (unneutralized). (See Table II).

Storage of the 2% samples for a period of six months at room temperature resulted in only a very slight tendency for sediment formation, with the exception of sulfonated Red Oil, which gave considerable sediment. Storage at temperatures ranging from 30° to 50°F for two months did not cause separation of ingredients in any of these samples. The 10% samples in general showed more sediment formation and several of them separated into two phases. Since the 2% samples had shown good rust inhibition in the preliminary work, these were selected as a basis for further experimentation.

In formulating a new REM oil, the choice of rust inhibitor was obviously the most important problem. From a merchandizing point of view, it was desirable to continue the use of an EP lubricant so that the statement "Made with du Pont EP Lubricant that Stays Put" appearing on the can could be retained. Also, Ilion had requested that a perfuming agent be added to the oil to give it a distinctive odor. The problem therefore consisted in formulating and testing samples of the base oil to which had been added ingredients of each of these three classes.

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The rust inhibiting agents to be included in the final evaluation were dictated in part by the work already described. To the inhibitors suggested by the Station were added Belle Phenol, a material which had been found here to have some merit as a rust inhibitor, and phenol (ordinary), which is the inhibitor used in FIENDOIL.

The preliminary work had shown that tricresyl phosphate, the EP lubricant used in the present REM oil, had no rust-proofing properties but that GD-162 (mono plus di Lorol phosphate) did offer some rust protection. The latter material was therefore selected for use in place of the former.

To arrive at suitable materials which might be used to give the oil a distinctive odor a survey of available perfuming materials had to be made. Samples of odoriferous substances were obtained and tested by adding to 50 cc of the base oil whatever number of drops of each was required to produce a definite odor, and then classifying the odors as to suitability for this particular use. Table III gives a list of the materials tried, the relative amounts required and a description and classification of the odors. As a result of this study, oil of Rosemary and oil of Pine Needles were selected as the two most satisfactory odoriferous materials.

The materials to be tested having been determined, experimental formulas were made up for further evaluation. Each sample consisted of the base oil plus a rust inhibitor, a perfuming agent and EP lubricant GD-162. The amount of perfuming agent was adjusted to give a definite odor in the presence of

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TABLE III

Tests of Perfuming Materials

50 cc of REM base oil taken for each sample

Sample No.	Added Perfume	Quantity(Drops) (For definite odor)	Drops Per CC
1	Anise Seed	5	55
2	Cedar Wood	10	55
3	Cloves	5	57
4	Eucalyptus	5	62
5	Juniper Wood	10	62
6	Lemon	10	60
7	Pine Needle	6	63
8	Rosemary	5	61
9	Spruce	5	62
10	Thyme	5	60
11	Pine Oil	15	60
12	Sassafras	20	72

Least Satisfactory

2 - Cedar Wood - bitter odor
5 - Juniper Wood - pleasant odor, but weak
12 - Sassafras - somewhat sour
10 - Thyme - seedy, hayloft odor

More Satisfactory

11 - Pine Oil - public wash room association
4 - Eucalyptus - medicinal association
9 - Spruce - stronger than Juniper;weaker than Pine Needle
1 - Anise Seed - very sweet) odors too common
3 - Cloves - agreeable)
6 - Lemon - agreeable)

Most Satisfactory

7 - Pine Needles - clean, forest odor
8 - Rosemary - pleasant, slightly medicinal, but unusual and clean smelling.

the other ingredients. The formulations were made on a volume basis. The compositions are given in Table IV.

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TABLE IV

Sample No.	Inhibitor	Base Oil	EP GD-162	Oil Rosemary	Oil Pine Needles
1	2cc coconut oil acids 1*	100cc	0.25cc	0.3cc	-
2	"	"	"	-	0.3cc
3	2cc oleic acid 2*	"	"	0.3cc	-
4	"	"	"	-	0.3cc
5	0.5cc Belle Phenol (redis-tilled) 3*	"	"	0.4cc	-
6	"	"	"	-	0.4cc
7	1cc phenol 4*	"	"	0.4cc	-
8	"	"	"	-	0.4cc
9	2cc sulfonated Red oil (unneu-tralized) 1*	"	"	0.2cc	-
10	"	"	"	-	0.2cc

1* - From Experimental Station
2* - Technical grade, Eimer and Amend
3* - Used here in shot shell wax
4* - C.P. grade, National Aniline & Chemical Co.

The experimental compositions were evaluated at Bridgeport in the humidifier and salt spray cabinet in comparison with the base oil, the present REM oil and FIENDOIL, and at the Experimental Station in the water condensation test in comparison with the following oils: base oil, present REM oil, FIENDOIL, "3 in One" oil, WINCHESTER gun oil, STOEGEROL, Marble's NITRO SOLVENT oil and SEAL STEEL. The test used at the Station has already been described. For the tests here steel panels 3/4"x 4-1/2"x 0.019", having a hole near one end, were used. These

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were immersed in toluol to remove the protective grease film and wiped clean. The oils were applied by dipping the strips into the oils and removing, repeating the process several times, and finally allowing the excess to drain off. The coated strips were hung on racks and placed in the humidifier oven and salt spray cabinet.

The results obtained in the local tests are given in Tables V and VI, and those of the Station test in Table VII. The results of the three tests are in general agreement in showing that the experimental compositions are superior to the present REM oil and that those containing oleic acid as rust inhibitor are the best of the experimental compositions. The results of these tests are discussed more fully in another section of the report.

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TABLE V

Results of Humidifier Storage Test

(120°F and 90% R.H.)

The experimental compositions consisted of 100 cc of base oil with 0.25cc of GD-162, and with additions of inhibitors and perfumes as indicated.

	Sample No.	1 Day	4 Days	5 Days	6 Days	7 Days	8 Days	33 Days
1)	2cc COA-R	10	10	10	10	10	10	9
2)	2cc COA-PN	10	10	10	10	10	10	9
3)	2cc OA-R	10	10	10	10	10	10	9.5
4)	2cc OA-PN	10	10	10	10	10	10	9.5
5)	0.5cc BP-R	10	10	10	10	10	10	8.5
6)	0.5cc BP-PN	10	10	10	10	10	10	8.5
7)	1cc P-R	10	10	10	10	10	10	8
8)	1cc P-PN	10	10	10	10	10	10	7
9)	2cc SRO-R	10	10	10	9.5	9.5	9.5	4
10)	2cc SRO-PN	10	10	10	9	9	9	2
11)	Base oil alone	8	8	7	6	5	3	1.5
12)	REM oil	9	9	8	8	7	5	3.5
13)	FIENDOIL	10	10	10	10	10	10	9

Perfumes

R = Oil of Rosemary
PN = Oil of Pine Needles

Inhibitors

COA = Coconut oil acids
OA = Oleic acid
BP = Belle Phenol
P = Phenol (ordinary)
SRO = Sulfonated Red Oil (unneutralized)

Ratings: Same as previous tables

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TABLE VI

Results of Salt Spray Test

(3% Salt Solution used at room temperature)

The experimental compositions prepared as indicated in Table V.

	Sample No.	20 Hours	90 Hours
1)	2cc COA-R	9	5
2)	2cc COA-PN	8	5
3)	2cc OA-R	9	6
4)	2cc OA-PN	9	7
5)	0.5cc BP-R	8	1
6)	0.5cc BP-PN	8	1
7)	1cc P-R	7	1
8)	1cc P-PN	5	0
9)	2cc SRO-R	10	6
10)	2cc SRO-PN	-	-
11)	Base oil alone	5	0
12)	REM oil	5	0
13)	FIENDOIL	10	6

Perfumes

R = Oil of Rosemary
PN = Oil of Pine Needles

Inhibitors

COA = Coconut oil acid
OA = Oleic acid
BP = Belle Phenol
P = Phenol (ordinary)
SRO = Sulfonated Red Oil (unneutralized)

Ratings: Same as previous tables

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TABLE VII

Results of Water Condensation Test

Experimental compositions prepared as indicated in Table V.

Sample No.	Corrosion Rating Average of Duplicate Panels 3 Days	9 Days	17 Days	27 Days	38 Days	3 Months*	Rating in Bridgeport Humidifier Test after 33 Days
1) Control (no oil; av. of 4 panels)	3.8	3.8	2.5	2.5	2.3	2	-
2) Present REM oil	4.0	3.5	2.5	2.5	1.5	1.5	3.5
3) REM Base Oil alone	5.5	5	5	5	5	3	1.5
4) 2cc COA-R	10	10	9.8	9.5	9.5	5	9
5) 2cc COA-PN	10	10	10	10	10	5	9
6) 2cc OA-R	10	10	10	10	10	9	9.5
7) 2cc OA-PN	10	10	10	10	10	9	9.5
8) 0.5cc BP-R	10	9.8	9.5	9.5	9.5	6	8.5
9) 0.5cc BP-PN	10	9.8	9.5	9	9	3	8.5
10) 2cc BP-R	9.8	9.8	9.5	9	8.5	3	-
11) 4cc OA-R(1cc)	10	10	10	10	10	9	-
12) FIENDOIL	10	10	10	10	10	10	9
13) "3 in One" Oil	9.8	9	9	9	9	6	-
14) WINCHESTER Gun Oil	6.5	6	5.5	4.5	4.5	3	-
15) STOEGEROL	10	10	10	10	10	7	-
16) Marble's NITRO SOLVENT Oil	6	5	4.5	4	3.5	2	-
17) SEAL STEEL	10	5.5	3	2.5	2	0	-

Perfumes

R = Oil of Rosemary
PN = Oil of Pine Needles

*Test continued on one set after 38 days

Ratings: Same as previous tables

Inhibitors

COA = Coconut oil acids
OA = Oleic acid
BP = Belle Phenol
P = Phenol (ordinary)
SRO = Sulfonated Red oil (unneutralized)

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The accompanying photograph (Figure I) shows the condition of one of the two sets of Station test panels at the end of the 38-day test period. On these panels rust shows up as dark spots or as larger dark areas. The numbers on the panels correspond with the sample numbers in Table VII. Since the corrosion ratings are <u>average</u> values from duplicate sets of panels, there are slight discrepancies in some cases between the ratings given in the table and the appearance of the photographed panels. The marked superiority of the proposed composition over the present REM oil is strikingly shown in this photograph (compare sample 2 with 6 and 7).

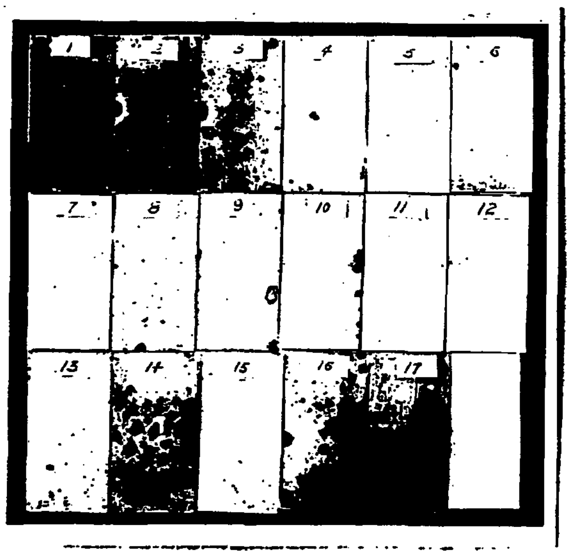

Figure I

Some of the experimental compositions were also subjected to film strength tests in the Cornell Friction Tester

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at the Station. In this test a bearing is lubricated with the oil and is loaded until seizure occurs; the load in pounds at seizure is a measure of the film strength of the oil. The proposed composition was found to have 50% greater film strength than the present oil:

	Load at Seizure (Lbs.)
Present REM oil (Sample 2)	1000
REM base oil (Sample 3)	1000
Proposed composition (Sample 6)	1500

In the course of this work it was noticed that the REM oil compositions wet polished steel surfaces with difficulty. This fault is also possessed by the competitive oils included in our tests, and appears, in fact, to be common to all lubricating oils. Attempts were made to improve this property in REM oil, but without success. Additions of large quantities of GD-162 and pine oil in ratios as high as 1:1 by volume, and of benzoic acid (0.25 grams/100 cc) did not improve the wetting characteristics appreciably. The addition of kerosene in the amount of 1:1 by volume improved the wetting properties, but such an addition could not be made to an oil marketed for general lubricating purposes.

Discussion of Results:

The results of the tests at Bridgeport and the Experimental Station are in general agreement in showing the poor rust-proofing properties of the present REM oil, and in the evalu-

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ation of the effectiveness of the experimental and competitive compositions. The present REM oil looked better in the Bridgeport humidifier test than in the Station water condensation test. The Station test showed that FIENDOIL is slightly superior to the best experimental compositions, while the humidifier test at Bridgeport showed that the experimental oleic acid compositions are slightly better than FIENDOIL. The salt spray showed no differences between these samples. STOEGEROL gave perfect protection during the early part of the Station test but allowed some rusting during the second two months. This oil was not included in the Bridgeport tests. "3 in One" was slightly inferior to STOEGEROL. Marbel's NITRO SOLVENT oil and SEAL STEEL gave relatively poor protection against rusting.

It should be pointed out the FIENDOIL, which is the one gun oil which appears to be substantially equal to the proposed REM oil, is a lard oil composition rather than a mineral lubricating oil and is not, therefore, streictly competitive with REM oil. SEAL STEEL is also not a lubricating oil but consists of a wax composition in a solvent. This product is one of the poorest tested and it might be desirable to develop a superior product of this type for the Remington line.

The relative merits of the various rust inhibitors used in the experimental compositions as indicated by the results of the three series of tests are as follows:

<u>Coconut Oil Acids:</u> The protection obtained by this inhibitor was of a high order but it proved to be inferior to oleic acid. The possible development of rancidity, which would

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result in an unpleasant odor, also makes the use of this material undesirable.

Sulfonated Red Oil (Unneutralized): This inhibitor showed considerable sludge formation during storage at room temperature and was less effective than the other materials.

Belle Phenol: This material gave good protection, although somewhat less than oleic acid. The characteristic strong odor of the phenol made it necessary to use larger amounts of perfuming agent to give the oil a pleasant odor.

Phenol (Ordinary): Phenol caused the oil to become very dark after storage at room temperature for one month. The rust inhibition was somewhat poorer than was expected in view of the results obtained with FIENDOIL, which contains this material.

Oleic acid: This is the best inhibitor tested and is the one recommended for use. It is readily available and low in cost. Increasing the quantity from 2% to 4% did not improve the results.

It will be noted that all the tests in this work have been conducted on bare steel, although gun oils are used very largely on browned steel. The tests were made on bare steel for the reason that the results are more quickly obtained, are more readily evaluated and can be recorded photographically. It would have been desirable to have followed the bare steel tests with other tests on browned steel but the amount of the project was insufficient for these additional tests. There is no reason to believe that the comparative results would be any

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different on browned steel.

The stability tests on the experimental compositions were made in glass bottles so that the results could be easily observed. Other tests are now under way in REM cans but some time will elapse before significant results are available.

The proportion of base oil in the proposed composition is so nearly the same as for the old oil (the added ingredients total less than 3%) that the effect on the functioning of guns under various temperature conditions should be unchanged.

The project under which this work was done proposed that the Research Department "develop and test formulations, the best of which will be recommended to Ilion for additional evaluation there". It will probably be desirable to include tests on browned steel and functioning tests in the evaluation which is to be made at Ilion. If Ilion prefers, this work can be done at Bridgeport under a Part II of the project.

Recommended Composition:

Consideration of all the test results shows that the composition containing oleic acid as the rust inhibitor is the best of the experimental formulas. The choice of GD-162 as EP lubricant has already been explained. Oil of Rosemary is favored as the perfuming agent because it costs less than oil of Pine Needles. The recommended composition is as follows:

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	Addition to Base Oil % by Volume	Laboratory Formula	Plant Formula
REM base oil	-	1000 cc	55 gal.
Oleic acid	2.0%	20	1 gal. 13 oz. (4165 cc)
GD-162*	0.25	2.5	17-1/2 oz. (520 cc)
Oil of Rosemary	0.4	4	28 oz. (835 cc)

*Marketed by the du Pont Organic Chemicals Dept. under the name "Ortholeum" One-Sixty-Two.

The preparation of the oil consists simply in mixing the ingredients at room temperature.

COSTS

The cost of the recommended composition is substantially the same as that of the present REM oil, the new cost being $0.2154 per gallon as compared with a current cost of $0.2159 per gallon.

The cost data are given in Table VIII.

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