A Study of Certain Physical and Chemical Effects of Aromatic Halides, Unsaturated Carboxylic Acids, Saturated Ketohydroxy Acids, and Saturated Ketone Alcohols Upon Hydrocarbon Oils

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By

Clyde Aubrey Crowley

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Loyola University

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SECTION I

STATEMENT OF PROBLEM

The physical and chemical factors responsible for the maintenance of tenacious lubricating films between metal surfaces are not well understood.

From time to time, numerous chemical components have been added to commercial lubricating oils for the purpose of increasing their lubricating ability, (reducing frictional losses), improving their film strength characteristics, reducing the metal attack by components present in or formed during lubrication with hydrocarbon lubricants, and for other stated purposes of lesser moment. That certain of these addition products have met with wide acceptance and are considered as valuable adjutants to lubrication is further proof of the deficiencies of highly refined hydrocarbon oils.

It has been the purpose of the studies reported here to determine certain of the chemical and physical changes produced by the addition of selected chemical components to a reference hydrocarbon oil of known low viscosity and to further study the influence of dual additions of corrosion inhibiting and 'corrosive' types of additions.

As a supplement to these studies, corresponding tests have been conducted upon a number of motor oil addition pro-
ducts offered in the American and foreign markets.

The data reported here should be considered as exploratory in every sense. The conclusions drawn in the summary (Section VIII) may be accepted as indicative of the performance of the limited number of molecular components investigated and as guidance for further serious investigation.
SECTION II

HISTORICAL SURVEY

Since the dawn of civilization, mankind has continually directed its efforts toward the development of machines and appliances of innumerable types whose purpose it has been and is to minimize the physical labors required for unit operations; and to thus elongate the minute, the hour, the day, the year, and a lifetime by making possible greater accomplishments. Early scientific developments were invariably dependent upon elementary but fundamental concepts of mechanism. The application of these concepts and the urge for mechanical efficiency has, since the first effort in this direction, required lubricants whose office is and always has been to reduce the energy wasted by friction, and to thus make possible greater productiveness with less and less effort. These efforts have resulted in the machine becoming increasingly efficient by virtue of improved design, construction, and lubrication.

Design and construction are outside the domain of the chemist, except as his efforts are related to the perfection of more suitable materials for fabrication. The third requisite of mechanical progress, lubrication, is distinctly a
chemical problem. As one studies the history of lubrication it becomes immediately apparent that this adjutant of mechanical perfection has been most neglected and continues to receive sparse attention.

During modern times, improved design and a greater choice of raw materials from which to construct mechanical devices and appliances, have made it more and more apparent to the observant worker that the application of proper lubricants is the difference between success and failure in many instances. Previous to the discovery and application of petroleum as a lubricant, animal fats, vegetable fats, shale oils and other easily available naturally occurring oily substances were used as lubricants. For many purposes animal fats and vegetable fats still enjoy wide usage and appear to give excellent service.

The ever-increasing popularity of mechanical devices and the far flung applications of machines of innumerable types has created a demand for lubricants which, in all probability, could not have been made except for the development of the petroleum resources of the world. It has long been recognized that petroleum lubricants, as used today, are for many purposes not the equal of naturally occurring fats and oils. The observation of this fact has led scientists to give spasmodic attention to the actual problems involved in lubrication and has resulted in the formulation of numerous theoretical and practical hypotheses upon
which to base further study and from which to gain further practical experience.

It was the purposes of the studies reported in this paper to determine the effects produced when certain chemical bodies of known constitution are added to hydrocarbon oils. A survey of the literature revealed a vast amount of qualitative and descriptive data relating to the action of a limited number of chemical entities. The data reported on those compounds which have been studied was found to be, in most cases, incomplete and of such a nature as to be of little use for the purpose of predicting the relative effects of the chemical components involved.

The technological importance of lubrication is so great that it will sooner or later demand the serious attention of an increasing number of investigators. The work reported in this paper may be considered as wholly exploratory.
SECTION III

THEORIES OF LUBRICATION AND THEIR WEAKNESS

The necessity for lubrication was early attributed to imperfections in rubbing surfaces. The frictional power consumption was assumed to have been caused by the interlocking of projections on bearing surfaces. This physical inter-locking of surface irregularities was assumed to be the prime cause for the restriction of motion and was held responsible for the losses which occur during the transmission of power through the medium of bearing surfaces. It was, however, soon observed that surfaces which had been lapped to such an extent that physical imperfections were negligible, still offered large resistance to motion. This resistance to motion is so great that surfaces lapped as plain as is possible wear rapidly when moved together without the application of a lubricant, thus pointing to the now accepted view that other factors enter into the development of friction.

Corse (1) very clearly reviews the expositions of Cuypers (15) and points out that the physical inter-locking of surface irregularities is unnecessary for the development of large values of friction. He further puts forth the possibility that an inter-locking of the heterogeneous electromag-
Electromagnetic fields of force radiating from surface molecules may, and probably are, responsible for high values of friction under conditions where metal to metal contact exists within the bearing unit. It would further appear that some slight linkage of electromagnetic fields might also exist under conditions of abnormally thin film separation (lubrication). The magnitude of the forces involved in such thin film linkages would, however, appear to be of small moment if it may be assumed that the minimum film thickness is proportional to the thickness of a hydrocarbon molecule. Harkin's (2-A) work indicates that this value should be in the neighborhood of 4.70A. If the length of an average carboxyllic acid molecule approximating the equivalent of an 18 carbon atom chain is taken as 250A it becomes apparent that the substitution of upstanding molecules of this type would increase the minimum metal to metal separation under conditions of thin film lubrication from 9.40A to 500A. This increase in minimum film thickness would in any event reduce the effectiveness of electromagnetic linkages as the square of the separation distances, or as 88 to 2500. This would reduce the electromagnetic field intensity to 0.035 of its original value. The increase in coefficient of friction with increased bearing pressure may be at least partially dependent upon this consideration.
Engineers classify lubrication films according to their thickness. So-called 'thick film lubrication' prevails under those conditions where bearing tolerances are sufficiently large to permit a thick coating (multi-molecular) of lubricant on both journal and bearing surfaces. With this type of lubrication, it would appear that the constitution of the lubricant has little to do with the efficiency of lubrication and that the only factor of importance is the viscosity of the lubricant. Accordingly, bearings operated under conditions of large clearance are lubricated with oils of high viscosity. So long as unit pressures remain sufficiently low to enable the film of lubricant to be maintained, metal to metal contact cannot exist and it would be surmised that the electrostatic linkages considered in connection with thin film lubrication would be a factor of small moment.

The second type of lubrication is known as 'thin film lubrication'. It is of this state that the present studies relate. Thin film lubrication is considered of great importance because it may be encountered under emergency conditions even in equipment designed for lubrication with high viscosity oils. It is known that metal contact actually exists under certain conditions even on machines lubricated with very high viscosity oils.

Thin film lubrication prevails when sudden changes in
motion occur, when sudden pressures are applied to the bearing-supported mechanisms, when gear teeth mesh under load, at the point of contact of cutting tools, and between parallel surfaces rubbing under such conditions as exist between pistons and cylinder walls in engines of all types, compressors, and other mechanically similar units.

At the moment of transition from thick film lubrication to thin film lubrication, it has been observed that the tangential friction drag per unit area increases rapidly. Under the conditions of thin film lubrication, it has been known for many years that a factor commonly termed the 'oiliness' of the lubricant becomes important. In order to be effective, under the conditions of thin film lubrication, a lubricant must possess the property of completely wetting the metal surfaces, spreading into a thin film, and maintaining itself in this condition even though great strains are applied.

Cuypers (15) defines 'oiliness' as the sum of the phenomena which tends to maintain a lubricating film under the influences of the solid surfaces, and decreases friction without generating fluid friction, within the lubricant itself, in excess of the reduction effected between the metals. He further states:

"While with complete film lubrication the viscosity is the factor which causes the decrease in friction, the cause of reduction in friction in the case of complete
film lubrication, is not the viscosity but the 'oiliness' of the lubricant."

As reported by Corse (1), Woog (28,29,30), has investigated the factors upon which good oiliness relies and has suggested a method for accomplishing this. The following four paragraphs are quoted direct from Corse's summary of Woog's papers:

1. "The mutual keying together of the molecules of the lubricant, by which we understand the resistance they oppose to pulling apart, insofar as it depends on a single molecule's having a hold on its neighboring molecules. For this hold, the size of the molecular volume of a lubricant, so much firmer does it hold even the thinnest film together."

2. "The form and dimensions of the lubricant molecule: we should not imagine the molecules as symmetrical, spherical shapes, but as more complicated forms showing strong irregularities. An oil film which consists of long molecules which stand close together like the bristles of a brush is much more resistant to the tendency of rotation than is an oil film consisting of molecules more nearly spherical."

3. "The presence of specially active centers of energy in certain molecules, which forces them under certain conditions to take on quite different positions: especially effective are such centers of energy when they are located at the end of the molecule. It appears that the durability of the oil film standing in direct contact with the solid surfaces will be so much the greater the firmer the lubricant molecules anchor themselves to the surfaces. And this anchoring will always be especially intensified if the force of attraction due to the metal surfaces themselves is strengthened by a special affinity of the lubricant molecules in which even such active centers of energy orient themselves toward the surfaces."

4. "The fourth factor of oiliness of a lubricant is the elastic stiffness of its molecules."
In view of the data reported in this paper, together with other data which the writer has had an opportunity to study, it appears that the following comments to the above four points as put forth by Woog are subject to criticism as follows:

1. Woog's theory would indicate that the film of lubricant is to be considered as an extremely thin sheet or film between the bearing and shaft and that it is in no wise influenced by the metals from which the bearings and shaft are constructed. Accordingly, he holds that the molecular volume is the important consideration.

The writer favors the theory that the dimensions of the molecules composing the lubricating medium are of relatively no consequence and that instead some factor equivalent to inter-molecular bonding is of greater importance. By this is meant, that assuming a continuity of the moving film, the factors which would make a film difficult to rupture would require strong bonding between consecutive molecules making up the mobile film.

Woog's theory does not sufficiently emphasize the importance of oriented molecular entities on the faces of the bearing and shaft. This appears to be a fatal weakness to the theory which could only be surmounted by assuming the presence, in the lubricant, of certain molecules which tend to orientate themselves on the metal.
surface to be lubricated and thus exert a smoothing out action on the surface itself. The smoothing out effect is suggested in view of the fact that the best lapped surface may still contain crevices, ridges, depressions and other irregularities whose amplitude can be as much as $70^\circ$ to $80^\circ$ A. from the crest to valley. In practice the amplitudes may be many times greater. Carboxylic acid molecules containing 18 carbon atoms per chain are considered to be in the neighborhood of $20^\circ$ to $25^\circ$ A. in length. If we assume that molecules of carboxylic acids orient themselves on the surface of the metal with the carboxyl group at the metal interface, it is apparent that under ordinary conditions we should expect that the maxima and minima of surface variation would become much less than $80^\circ$ A. and would more nearly approach the length of the carboxylic acid molecule. If this is the case, then the presence over the entire bearing surface and shaft surface of oriented carboxylic acid molecules would tend to limit the surface variations to substantially $20^\circ$ or $25^\circ$ A. units in the case of carboxylic acids containing substantially 18 carbon atoms per molecule. The case of substituted and unsaturated carboxylic acids merits further consideration and its effects will be discussed at length with the data pertaining to tests on their performance.

2. In Woog's second requirement as detailed above, it
is indicated that the shape of the molecule is of considerable importance in determining its fitness as a lubricant. The suggestions put forth appear logical if we interpret his statements as referring to those molecules which form the thick film layer between the hydrocarbon tails of oriented carboxylic acids. On the basis of his statements, we would be led to the conclusion that open chain aliphatic hydrocarbons would in all cases be superior as thick film lubricants to cyclic hydrocarbon molecules. This appears to be borne out in practice, inasmuch as paraffinic oils are generally considered superior to naphthenic lubricants.

3. In Woog's third qualifying paragraph, he brings out the importance of certain types of Polar groups which show a tendency to 'anchor' themselves to the metal surfaces. In this connection, the findings reported here tend to emphasize the importance of Polar groups and to further indicate the nature and limit the location of those Polar groups which are most effective. At this point, it should be mentioned that it does not appear desirable that lubricants contain unstable Polar groups which upon decomposition, addition to or substitution into, can give rise to the formation of groups which would attack the bearing metals. Furthermore, it is indicated that the presence of chemical groups in the lubricant which exert a corrosive action upon bearing or shaft is of considerable importance in selecting the type of carboxylic acid molecule which will be most
effective. It will later be shown that the presence of oleic acid in the proportions of not to exceed 1% and probably in lesser percentages, inhibits the corrosive action of certain phosphoric acid esters to the extent of 80% while it is not only ineffectual but actually accelerates the corrosive action of phenyl chloride. In other words, an oil containing a trace of phenyl chloride is made more corrosive and more destructive to bearing surfaces and shafts by virtue of the presence of oleic acid. This would suggest the possibility of substitution at the double bond in oleic acid by the degradation products of the phenyl chloride molecule.

4. The statement above that the fourth factor of oiliness depends upon the elastic stiffness of the molecules present is not entirely clear. This statement may be interpreted as referring to the presence or absence of double bonds in the lubricant molecule. If we assume this interpretation, then this qualification becomes entirely in accord with present findings, provided we further restrict this 'molecular flexibility' to the molecules which orient themselves upon the surface of the metals being lubricated. By the methods used in this investigation, it has not been possible to determine the comparative effectiveness of olefines and saturated hydrocarbons as filler lubricants which operate between the oriented boundaries.
Wilson and Bernard (27) have proposed a definition for 'oiliness'. Their definition follows: 'Oiliness is defined as that property of lubricants by virtue of which one fluid gives lower coefficients of friction (generally at slow speeds or high loads) than another fluid of the same viscosity'. With deference to the incompleteness of the Bernard definition, particularly with respect to its omitting the problem of film rupture, strength, Burwell and Camelford (13) have proposed that the Wilson and Bernard definition should be revised to read: 'Oiliness is defined as that property of lubricants by virtue of which one fluid gives lower coefficients of friction (generally at slow speeds or high loads) than another fluid of the same viscosity and at the same time increases the film strength of such lubricants.'

The data reported here would indicate that if the definition for oiliness as suggested by Bernard and Wilson is to be modified along the lines suggested by Burwell and Camelford, that it should be further modified in such a manner as to definitely eliminate, limit, or specify the classification of film strength desired. As a result of the work reported here, it has been found advisable to classify film strength enhancing addition agents according to the mechanism by which the increase in film strength is produced.
Unsubstituted carboxylic acids, when added to hydrocarbon oils relatively free from olefines and unstable chemical molecules have been shown to definitely increase the film strength of the lubricant without effecting the corrosion of the metals making up the bearing and shaft. Furthermore, it has been found that the addition of certain phosphoric acid esters, halogenated compounds, sulphur compounds and certain other molecular components to be discussed in detail later, increase the film strength of the lubricant in many cases far above the values which have been realized to date from carboxylic acid additions; however, this increase in film strength has been attended with rapid corrosion and erosion of bearing metal and shaft. It has been further observed that the addition of certain types of carboxylic acids to oil samples carrying corrosive film strength improving molecules either completely inhibits the corrosion or only partially inhibits the corrosion. When corrosion is completely inhibited, the film strength of the complex is reduced to the value originally produced by the particular carboxylic acid alone. In those cases where corrosion has been only partially inhibited, the film strength of the lubricant was found to be reduced to a value intermediate between that produced by the carboxylic acid alone and the corrosive agent alone. This points to the conclusion that molecular components which improve film strength by virtue
of their corrosive action can only be inhibited from corro-
roding by the addition of molecules which have the property
of orienting themselves on the surface of the metals to
such an extent as to preclude contact of the corroding ad-
dition. In these cases, the film strength is, as would be
expected, reduced to the value produced by the inhibitor
alone. This points to the fallacy in attempting to use
corrosive additions for the purpose of promoting film stren-
gth and adding with them inhibitors to overcome their de-
leterious effects.

Persuant to these views, it would appear that a further
revision of the definition of oiliness should be made as
follows: Oiliness is defined as that property of a lubri-
cant by virtue of which one fluid gives lower coefficients
of friction (generally at low speeds or high loads) than
another fluid of the same viscosity and at the same time
increases the film strength of such lubricants without
causing corrosion to bearing surfaces and without promoting
corrosion by any components which may be present under the
conditions imposed upon the lubricant in use.
SECTION IV
LABORATORY PROCEDURE

For the purpose of this investigation a quantity of highly refined mineral oil of paraffinic origin was procured. The lubricant used was found to have a viscosity of 85 sec. at 100° F. (Saybolt Universal). This oil was tested and found to have a comparatively low lubricating value, a quality considered desirable for the purposes, inasmuch as it was desired to determine the effects of certain molecules and combinations of molecules upon the lubricating quality and film strength characteristics of hydrocarbons.

Samples of the above oil were made up, each containing 3% by volume of the following:

Table A

| Phenyl Chloride          | Tricresyl Phosphate |
| Alpha Chloronaphthalein  | Butyl "Carbitol"   |
| Triamylamine             | Lauric Acid        |
| "Alketoacid" C-18        | Butyl Lactate      |
| Ricinoleic Acid          | Isopropyl Ether    |
| Triethanolamine          | Trioxymethylene    |
| Linoleic Acid            | Dibutyl Phthalate  |
| Palmitic Acid            | Pine Needle Oil    |
| "Alketone" C-32          | Thymol            |
Table A (continued)

"Alketone" C-20  Benzaldehyde
Stearic Acid  Oleic Acid
Undecylenic Acid

Samples were also prepared as follows:

Table B

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
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<tbody>
<tr>
<td>White Oil</td>
<td>99%</td>
<td>98%</td>
<td>96%</td>
<td>94%</td>
</tr>
<tr>
<td>Oleic</td>
<td>1</td>
<td>2</td>
<td>4</td>
<td>6</td>
</tr>
</tbody>
</table>

| E  |    | White Oil         | 94% |
|    |    | Oleic Acid        | 3   |
|    |    | Phenyl Chloride   | 3   |

Samples containing mineral oil to which was added tricresyl phosphate, 3%, and the following components were also prepared for the determinations discussed in Section VII.

Table C

<table>
<thead>
<tr>
<th></th>
<th></th>
<th>&quot;Alketone&quot; C-18</th>
<th>3%</th>
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<tbody>
<tr>
<td>Oleic Acid</td>
<td>1%</td>
<td>Palmitic Acid</td>
<td>3</td>
</tr>
<tr>
<td>Oleic Acid</td>
<td>2</td>
<td>Ricinoleic Acid</td>
<td>3</td>
</tr>
<tr>
<td>Oleic Acid</td>
<td>3</td>
<td>&quot;Alketone&quot; C-20</td>
<td>3</td>
</tr>
<tr>
<td>Oleic Acid</td>
<td>4</td>
<td>&quot;Alketone&quot; C-32</td>
<td>3</td>
</tr>
<tr>
<td>Oleic Acid</td>
<td>6</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

TEST METHODS

An adsorption dynamometer of the Faville LeVally type was used in accordance with the procedures herein described.
for the conduct of the tests reported. It was found necessary to make certain modifications in the standard Faville LeVally machine in order to render test runs reproducible. Fig. 1 which follows shows the assembly arrangement of the Faville LeVally machine. It will be noted that a pair of pincher arms press the pair of bearings, of types detailed in Fig. 3 against a 1/4 inch drilled rod shaft under spring tension. The draw-up wheel shown in Fig. 1 and 4 is automatically rotated to bring about a continuous and regular application of pressure increments. The automatic draw-up arrangement was actuated by an eccentric driven by the upper portion of the main bearing shaft. The rate of pressure increments was, therefore, 1 notch per shaft revolution, or 290 notches per minute. Each notch on the take-up wheel corresponded with a bearing squeeze on the shaft produced by 0.000067 inches of spring compression. The dynamometer unit was driven by a synchronous motor to maintain constant speed throughout all tests.

When the test operations were initiated, it was hoped that the construction of the machine would permit the measurement of oil film thickness by comparing the calibrated take-up wheel indications with a previously run pressure curve. Accordingly, a calibration run was made with hardened steel bearings of the type shown in Fig. 3A pressed against a drill rod shaft without lubrication. A large number of tests were made to determine the relationship between take-up wheel calibration and
Fig. 1, Absorption Dynamometer Assembly

Take Up Wheel
(see Fig 4 for details)

See Fig 2

A - Bottom View

B - Right hand Side View
Fig. 2 Details of Shaft and Spindle Assembly
Fig. 3  Types of bearings used

A

B

C

90°

0.0875"

0.75"

Driven by Eccentric

Fig. 4  Automatic pressure increase arrangement
applied pressure as indicated on the pressure gauge. Curve 1 attached indicates the variation which was found to occur in repeat runs. The heavy line is plotted to the average of all calibration tests conducted. In view of the cyclic variations in the notch-up to produce a given pressure, all hope of measuring film thickness by this method was abandoned. The limit curve showing the relation between notch-up and pressure, however, has been retained in curves, in order to give some idea of the probable change in oil film thickness under conditions of increasing pressure.

INTERPRETATION OF DYNOMOMETER DATA

In computing comparative values discussed, the following symbols have been used uniformly. Consideration of these values together with the diagrams appended is included to make clear the performance of the test equipment.

\[ r_k = \text{radius of shaft} = .125 \text{ inches.} \]
\[ d_k = \text{distance between center of shaft and point of contact between torque arm and sylphon tip.} \]
\[ f_1 = \text{force tangential to shaft.} \]
\[ f_2 = \text{force applied to sylphon gauge by torque arm.} \]
\[ t = \text{moment of torque developed by friction in pound inches.} \]
\[ d_1 = \text{length of pressure application arm (total).} \]
\[ d_2 = \text{distance between pressure arm fulcrum and shaft center.} \]
\[ P = \text{gross pressure on bearing faces.} \]
CURVE I
CALIBRATION OF TAKEUP WHEEL

PRESSURE (lb's)
The bearing faces pressing against the surface of the shaft (Fig. 1 and 2) produce a frictional force which tends to cause the bearings to rotate with the shaft. These forces may be resolved into a single force acting tangential to the surface of the shaft. The product of this resolved frictional force times the radius of the shaft represents the moment of torque developed by friction.

\[(1) \, t = r_k \times f_1\]

This torque, which tends to produce rotation of the bearing unit, is counter-balanced by the moment of the torque arm pressing against the tip of the pressure gauge sylphon.

The moment of this torque arm is equal to the product of the distance between the center of the rotating shaft and the point of support of the torque arm on the sylphon tip, and the force acting on the fluid filled sylphon as indicated by the Bourdon type of gauge actuated by the sylphon or:

\[(2) \, t = d_k \times f_2\]

Equating 1 and 2 gives an expression for the force tangential to the bearing shaft.

\[(3) \, \frac{r_k \times f_1}{r_k} = d_k \times f_2 \quad \text{or} \quad f_1 = d_k \times \frac{f_2}{r_k}\]

Frictional power consumption is most readily calculated in horsepower from the English system calibration of the machine, by multiplying frictional torque times radians per
minute and dividing by 33,000 or:

\[ \text{(4) Frictional H. P.} = \frac{2\pi t \times \text{R.P.M. of shaft}}{33,000} \]

\[ \text{(5) Watts consumed by friction} = \text{H. P.} \times 746 \]

In order to compare the friction rates at the various test pressure applied, the following coefficient was used:

\[ \text{(6) Torque coefficient} = \frac{t}{f} \times 100 \]

This coefficient was selected because torque is proportional to frictional power consumption and it was found helpful to evaluate repeat runs on the basis of this coefficient in determining the validity of the tests in question.

OPERATING PROCEDURE

The film strength tests reported in Sections V and VI were conducted on hardened steel bearings of the types shown in Fig. 3A and 3B as indicated. The shaft consisted of a hardened section of polished drill rod. In all cases, both shafts and bearings were lapped to a good polish.

Previous to each test, the machine was thoroughly washed, first with naphtha and then with trichlorethylene to remove the last trace of impurity from previous tests. The clean bearing halves and shaft were then installed in the machine and the pressure jaws applied loosely. The oil container was filled with its standard (70 cc.) charge of oil, the machine started, and the pressure wheel slowly drawn up to a gross pressure of 250 pounds. At this instant, the time clock was started and
the machine allowed to operate for three minutes. This initial three minutes was considered as a break-in period, whose function it was to allow the bearing faces to properly align and to remove any slight surface irregularities which might exist. In all cases, torque and pressure were stabilized within the first few seconds of operation and remained constant throughout the three minute break-in period. At the conclusion of the break-in period, the automatic draw-up mechanism driven by the eccentric was thrown into communication with the notched draw-up wheel and allowed to apply pressure automatically until the pressure as indicated by the gauge had reached 500 pounds. At this point the automatic notch-up mechanism was removed for 5 seconds while notch-up values and torque readings were taken. The notch-up arrangement was immediately replaced and allowed to continue until the pressure had been increased by another increment of 250 pounds. This procedure was repeated uniformly until bearing seizure or excessive wear occurred. In all cases, the final torque value was recorded regardless of whether the test was run to seizure or to excess wear.

The tabular data reported in Section IX reports all values taken in the following units:

\[ t = \text{Torque in Pound Inches.} \]

\[ P = \text{Pressure in gross pounds of pressure applied to bearing face.} \]

\[ n = \text{Notch-up or take-up in number of notches actually taken up during pressure interval.} \]
The constant pressure wear tests reported in Section VII in connection with commercial products were made according to the following procedure:

Bearings of the type shown in Fig. 3C were accurately machined to a depth of .075 inches and to a curvature corresponding with a 1/4 inch diameter shaft. The shaping of these bearing faces was effected in a specially constructed fixture to insure uniformity. These bearings were then run in to produce a satisfactory test surface.

The above described bearings were drilled at a uniform distance below the bearing surface (in a fixture) and an iron constantine thermo-couple was installed for the purpose of temperature recording.

The above bearings, equipped with thermo-couples were installed in the test dynamometer. After installing the bearings, the pressure was brought to 10 lbs. and the machine started. The pressure was immediately increased to a gross pressure of 50 lbs., and a running 5 minute break-in period allowed to insure suitable bearing linement and normal machine operation. At the end of the break-in period, readings of notch-up wheel, temperature, torque, and pressure were taken. For the remaining period of the test, the pressure arrangement was adjusted at one minute intervals to maintain a uniform pressure of 50 lbs. gross. Readings of temperature, torque, and wear were then taken at 5 minute intervals throughout the
duration of the test.

FILM STRENGTH TESTS ON COMMERCIAL PRODUCTS

The film strength tests reported in Section VII were conducted on hardened steel bearings of the type shown in Fig. 3B. These bearings were carefully lapped to a highly polished surface and the tests conducted in a manner similar to the V-bearing seizure tests detailed above.

After a 5 minute break-in period, the automatic ratchet take-up mechanism was allowed to operate until the pressure had increased to 250 lbs. The machine was allowed to operate for 5 minutes at this pressure, after which readings of temperature, and torque were taken. The machine was immediately notched up by automatic feed for an additional increment of 250 lbs. and operation allowed to continue for 5 minutes, after which readings were taken. This procedure was continued until seizure occurred.

ADDITIONAL LABORATORY PROCEDURES

Additional laboratory procedures resorted to in the course of gathering the data reported herein will be discussed in the various sections involved.
SECTION V

CARBOXYLIC ACIDS, NEUTRAL SUBSTITUTION PRODUCTS, ALIPHATIC HYDROCARBONS ESTERS

A total of 12 derivatives consisting of saturated and unsaturated carboxylic acids, hydroxy carboxylic acids, ketone alcohols and keto hydroxy carboxylic acids were examined with the adsorption dynamometer described in Section IV. The following is a summary of the seizure pressures and corrosion observations in connection with these tests:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Detailed Test Data in Table</th>
<th>Seizure Pressure</th>
<th>Corrosion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference Hydrocarbon</td>
<td>I</td>
<td>750 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>'Alketoacid' C-18</td>
<td>VII</td>
<td>1500 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Oleic Acid</td>
<td>II</td>
<td>1250 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>'Alketone' C-32</td>
<td>XIII</td>
<td>1250 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Palmitic Acid</td>
<td>XII</td>
<td>1250 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Linoleic Acid</td>
<td>XI</td>
<td>1250 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Butyl Lactate</td>
<td>XXI</td>
<td>1250 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Undecylenic Acid</td>
<td>XVI</td>
<td>1200 lbs.</td>
<td>Slight corrosion</td>
</tr>
<tr>
<td>Stearic Acid</td>
<td>XV</td>
<td>1200 lbs.</td>
<td>Solubility low—no evidence of corrosion</td>
</tr>
</tbody>
</table>


In reviewing these data, the following characteristics are significant.

(I) The carboxylic acids and the ester tested were found to be non-corrosive to the steel bearings under the test conditions prevailing. The one exception is in the case of the undecylenic acid. The sample of undecylenic acid used was made by the destructive distillation of castor oil and accordingly contained impurities difficult to remove and which may possibly have been responsible for the slight corrosion which was observed. This opinion is borne out by the fact that other carboxylic acids containing a double bond did not produce corrosion.

(2) The carboxylic acids having long carbon chains group themselves close together with respect to seizure pressure.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Detailed Test Data in Table</th>
<th>Seizure Pressure</th>
<th>Corrosion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ricinoleic Acid</td>
<td>VIII</td>
<td>1175 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Pine Needle Oil</td>
<td>XXIII</td>
<td>1100 lbs.</td>
<td>Slight corrosion</td>
</tr>
<tr>
<td>'Alketone' C-20</td>
<td>XIV</td>
<td>1000 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Lauric Acid</td>
<td>XIX</td>
<td>900 lbs.</td>
<td>None</td>
</tr>
</tbody>
</table>
(3) The carboxylic acid which required the greatest pressure before seizure occurred contains alpha, beta and gamma hydroxyl and carbonyl groups. In view of the fact that this particular carboxylic acid is made by the oxidation of petroleum fractions, it was not found practical to separate it into fractions of known molecular constitution, however, it is definitely known that two or more hydroxyl and carbonyl groups are substituted in the positions aforementioned. In view of this information and the seizure performance of the oil sample containing this material, it would appear that the orientation of the molecule at the metal interface involves a stronger bond.

To gain further information relative to the nature of the above indicated increase in seizure pressure, a series of tests were run with oleic acid additions in increasing quantities to the reference hydrocarbon oil used throughout these tests.

The following table records the significant values observed in connection with these tests:

| TABLE E |
|-----------------|--------|--------|--------|--------|--------|
| Oleic Acid Concentration Tests | 1%    | 2%    | 3%    | 4%    | 6%    |
| Seizure Pressure | 1250  | 1250  | 1250  | 1250  | 1500  |
| Seizure Torque  | 80    | 80    | 85    | 90    | 90    |
| $\frac{t \times 100}{P}$ | 6.4   | 6.4   | 6.8   | 7.2   | 6.0   |
In view of the fact that the seizure pressure was practically constant regardless of oleic acid concentration, it would appear that within the limits required to produce complete molecular orientation over the entire surface to be lubricated additional quantities of oleic acid have no function. The seizure torque values show an upward trend; however these values are well within the limits of experimental error. This fact is occasioned on account of the rapidity with which seizure occurs and the attendant difficulty in taking a maximum reading. The variation in torque coefficient values reflect this torque uncertainty.

The data shown in Table D relative to the performance of carboxylic acids and that shown in Table E relative to the concentration performance of a typical carboxylic acid points definitely to the conclusion that the improvement in film strength produced by carboxylic acids is due to the formation of an oriented film of carboxylic acid on the metal surface.

A further consideration of this view suggested the possibility of studying the heats of wetting of carboxylic acids in contact with metals. Due probably to the low values of heats of wetting, only a few investigators have studied this value.

The work of Breiger (12) was found to contain data on the heats of wetting of castor oil, linseed oil, paraffin
oil, refined machine oil, and oleic acid 1%/ in petroleum oil on copper.

Previous work by Crowley (14) indicates that tri-glycerides of fatty acids are ineffectual in reducing seizure values and torque values unless free carboxylic acid is present. Accordingly, the work of Breiger (12) was interpreted as indicative of the heat of wetting for the carboxylic acids corresponding to the tri-glycerides reported. This assumption is considered reasonable in view of the known fact that unless special precautions are taken, a sufficient quantity of free acid always exists in tri-glycerides to give concentrations well within the limits of that required to orient themselves on the metal surfaces under the test conditions reported by Breiger.

Assuming that Breiger's values apply to the carboxylic acids which would be present in castor oil and linseed oil, the following corrected values for heats of wetting in degrees Centigrade per 100 grams of copper adsorbent were tabulated.

<table>
<thead>
<tr>
<th>oleic Acid 1%/ in unknown mixture</th>
<th>Heat of Wetting</th>
<th>Seizure Values found here</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linseed Oil (Linoleic Acid)</td>
<td>13.85</td>
<td>1250</td>
</tr>
<tr>
<td>Castor Oil (Ricinoleic Acid)</td>
<td>12.07</td>
<td>1175</td>
</tr>
<tr>
<td>Paraffin Oil</td>
<td>3.85</td>
<td>750</td>
</tr>
</tbody>
</table>
It is significant to note that these values for the heats of wetting and seizure values as determined by the methods used in this study, agree in order; and further that the ratio of the heat of wetting of linseed oil (13.85) to that of castor oil (12.07) equals 1.09, while the ratio of the seizure pressure of Linoleic acid (1350 lbs.) to that of Ricinoleic acid (1175 lbs.) equals 1.065. The agreement in this one particular case is considered extremely close in view of the difference in the methods used.

Breiger's value for the heat of wetting of oleic acid was determined in a petroleum solution of undefined composition and therefore it would hardly be expected that his value for oleic acid would be comparable to the other values reported.

The property known as lubrication has been defined by Dallwitz (16) as being proportional to the ease with which the oil is adsorbed by the metal. He states that the tenacity of the oil film would appear to depend upon the capillary properties of the oil and the metals to be lubricated. Dallwitz–Wegener (17) have outlined a series of methods whose purpose it is to make possible the determination of these values. Pursuing the above citations indicates that these investigators believe that the lubricating power of an oil increases and decreases with changes in its angle of contact with the metal. Their conclusions indicate that the lower the surface tension of a lubricant,
the better will it be expected to perform its function.

In view of these findings, it would appear that the metal surface being lubricated acts as an adsorber and therefore the oil constitutes the adsorbent. Breiger suggests that this adsorbing function of metallic surfaces may be the result of residual valencies of the atoms on the surface. He further adds that adsorption therefore consists in saturating these valencies with the secondary valencies of the adsorbed molecules. He then goes on to define the heat of wetting as the heat liberated during adsorption.

The work of Southcomb and Wells (24) indicates that exceedingly small quantities of unsaturated groups produce extraordinary increases in the lubricating ability of poor oils.

Wells and Southcomb (24) have also found that the glycerides of fatty acids possess lower surface tensions than do hydrocarbon oils. They further show that the reduced surface tension of triglycerides is attendant with the presence of free fatty acids. They further show that the surface tension of natural glycerides, free from fatty acids, are of the same magnitude as the surface tensions of mineral oils. They further give evidence to the effect that the lubricating power of either natural glycerides, free from fatty acid, or a hydrocarbon oil is greatly enhanced by addition of a small percentage of fatty acids. Wells (24A)
shows that oleic acid forms an oriented film on metal surfaces which has a thickness of $4 \times 10^{-7}$m.

In view of the findings reported here and additional data recorded in the literature, it would appear that increase in film strength of the lubricant and reduction in friction is effected to the greatest extent when molecules are present which have the property of forming an oriented layer on the surface of the metals to be lubricated. It would further appear that oxygen-containing groups, hydroxyl and carbonyl, are most effective. It cannot be inferred that the presence or absence of double bonds in the added molecules are of any more than pure physical significance. The effectiveness of oxygen in this respect is attested to by the findings of Gilson (18). In his investigations, a machine was arranged so that the bearing being lubricated could be operated in controlled gaseous atmospheres. This procedure was followed pursuant to results from certain previous tests, which had appeared to have been affected by the presence of gaseous components present in the atmosphere surrounding the test equipment. The following quotation is taken from Gilson's work:

'... A small bearing was mounted so that tests could be made on it while running in any atmosphere. The friction in a vacuum was much higher than in air. Oxygen gave lower friction than air; if the oxygen was exhausted the friction increased but not to the original value for a vacuum. The bearing was run continuously in a vacuum and readings taken at 8 hour intervals.'
The friction became progressively higher, and had not reached a maximum at the end of test. If the bearing was then run in air for 8 hours the friction dropped almost to its original value in oxygen. Moisture was also found to have an effect, the friction being much less in moist than in dry hydrogen."

"The experiments indicate that for efficient lubrication some kind of reaction is necessary which is dependent upon the presence of moisture and of oxygen. The fact seems to be well established that the surrounding atmosphere has a very decided effect upon the friction obtained. The differences in friction cannot be accounted for by differences in viscosity of the oil. All of the friction curves tend to come together in the region around 100°C., but it is not yet known whether or not this has any significance."

Further conclusions indicate, from the tests conducted will be discussed in Section VIII in deference to further data reported in the following section.

The attached Curve II presents in curve form the comparative performance of the group members reported in this section together with a diagramatic illustration of the suggested bonding at the metal interface and the writers interpretation of the influence of double bonds.
SECTION VI

TESTS WITH AROMATIC HALIDES AND OTHER MOLECULAR COMPONENTS

A total of 15 chemical compounds were added to the reference hydrocarbon oil in the proportions of 3% by volume and tested under conditions identical to those discussed in Section IV. The following table summarizes these findings with respect to seizure pressure, and corrosion.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Detailed Test Data In Table</th>
<th>Seizure Pressure</th>
<th>Corrosion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference Hydrocarbon</td>
<td>I</td>
<td>750 lbs.</td>
<td>None</td>
</tr>
<tr>
<td>Tricresyl Phosphate</td>
<td>XVII</td>
<td>2000</td>
<td>Corroded</td>
</tr>
<tr>
<td>Phenyl Chloride</td>
<td>III</td>
<td>1500</td>
<td>Corroded</td>
</tr>
<tr>
<td>Benzaldehyde</td>
<td>XXV</td>
<td>1300</td>
<td>Corroded</td>
</tr>
<tr>
<td>Alpha Chloronaphthalein</td>
<td>V</td>
<td>1250</td>
<td>Corroded</td>
</tr>
<tr>
<td>Dibutyl Phthalate</td>
<td>XXII</td>
<td>1100</td>
<td>Slight Corrosion</td>
</tr>
<tr>
<td>Triamylamine</td>
<td>VI</td>
<td>1000</td>
<td>Slight Corrosion</td>
</tr>
<tr>
<td>Triethanolamine</td>
<td>IX</td>
<td>1000</td>
<td>Slight Corrosion</td>
</tr>
<tr>
<td>*Butyl Carbitol</td>
<td>XVIII</td>
<td>1000</td>
<td>None</td>
</tr>
<tr>
<td>Thymol</td>
<td>XXIV</td>
<td>950</td>
<td>Very Slight Corrosion</td>
</tr>
<tr>
<td>Isopropyl Ether</td>
<td>X</td>
<td>750</td>
<td>None</td>
</tr>
<tr>
<td>*Trioxymethylene</td>
<td>XX</td>
<td>550</td>
<td>Corrosion</td>
</tr>
</tbody>
</table>

* Not completely soluble.
With the exception of sulphur compounds, the above series of tests included all of the commonly used extreme pressure addition products. It is to be noted that in all cases the chemical additions which produced notable improvement in the film strength of the lubricant also corroded both bearing and shaft.

In the case of the first four items listed above, the corrosion was severe. Phosphoric acid esters and chlorides are recognized as important components in extreme pressure lubricants. That these components are corrosive is also well understood.

These tests were conducted largely for the purpose of determining the relative corrosiveness and effectiveness as extreme pressure lubricant addition materials with a view of gaining information which could be used for purposes of comparison when non-corroding seizure pressure increasing components were added together with them.

Curve III attached indicates the general performance of these addition products with respect to their effects in seizure pressure.

Further discussions of these data will be contained in Section VII which follows.

TESTS ON PRODUCTS SOLD IN AMERICAN AND FOREIGN MARKETS

Samples of products sold in the American and foreign
markets were procured and tested to determine their comparative actions when added to motor oils of standard quality.

These samples are identified by letters as follows:

<table>
<thead>
<tr>
<th>Reference Oil</th>
<th>Source</th>
<th>Class</th>
<th>Data In Table</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>American</td>
<td>A</td>
<td>BB</td>
<td>XLII</td>
</tr>
<tr>
<td>American</td>
<td>B</td>
<td>AA</td>
<td>XLIII</td>
</tr>
<tr>
<td>Foreign</td>
<td>C</td>
<td>BB</td>
<td>XLIV</td>
</tr>
<tr>
<td>American</td>
<td>D</td>
<td>AA</td>
<td>XLV</td>
</tr>
<tr>
<td>American</td>
<td>E</td>
<td>BB</td>
<td>XLVI</td>
</tr>
<tr>
<td>American</td>
<td>F</td>
<td>EE</td>
<td>XLVII</td>
</tr>
<tr>
<td>American</td>
<td>G</td>
<td>EE</td>
<td>XLVIII</td>
</tr>
<tr>
<td>American</td>
<td>H</td>
<td>DD</td>
<td>XLIX</td>
</tr>
<tr>
<td>American</td>
<td>I</td>
<td>FF</td>
<td>L</td>
</tr>
<tr>
<td>Foreign</td>
<td>J</td>
<td>CC</td>
<td>L</td>
</tr>
</tbody>
</table>

A summary of the test values from this series of tests is shown in the following table.

<table>
<thead>
<tr>
<th>Product</th>
<th>Wear</th>
<th>Torque</th>
<th>Condition of Bearing</th>
<th>Temperature Rise</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plain Oil Only</td>
<td>100%</td>
<td>100%</td>
<td>Corroded</td>
<td>100%</td>
</tr>
<tr>
<td>A</td>
<td>Reduced</td>
<td>Reduced</td>
<td>Badly Corroded</td>
<td>Reduced</td>
</tr>
<tr>
<td></td>
<td>20.5%</td>
<td>4.4%</td>
<td></td>
<td>4.8%</td>
</tr>
</tbody>
</table>
To throw light on the components present in these products, to which may be attributed their characteristic actions, the following table has been prepared. The class letters correspond with the classifications noted in Table H.
Table I

<table>
<thead>
<tr>
<th>Class of Product</th>
<th>Composition</th>
<th>Notes</th>
<th>Samples Tested and Identification</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA</td>
<td>Graphite only - High Concentration</td>
<td>Graphite settled rapidly</td>
<td>D and B</td>
</tr>
<tr>
<td>BB</td>
<td>Graphite only - Lower Concentration</td>
<td>Slight settling of graphite</td>
<td>A, C and E</td>
</tr>
<tr>
<td>CC</td>
<td>Graphite and Animal Oil</td>
<td>Graphite settled due to acidity of fat</td>
<td>J</td>
</tr>
<tr>
<td>DD</td>
<td>Graphite and Chemicals</td>
<td>No settling of graphite</td>
<td>H</td>
</tr>
<tr>
<td>EE</td>
<td>Chemical additions - No graphite</td>
<td>Acid after test</td>
<td>G and F</td>
</tr>
<tr>
<td>FF</td>
<td>Graphite and Hydrocarbon Derivative</td>
<td>No settling of graphite. No acidity after test.</td>
<td>I</td>
</tr>
</tbody>
</table>

At first glance, the greatest improvement in lubricating conditions would appear to rest between sample F and I. In view of the fact that under the test conditions prevailing an increase in wear rate would bring about greater reductions in pressure between the one minute take-up periods, it would be expected that F, which permitted higher wear than I would show a lower torque value and a lower temperature rise than I. Consideration of this factor, together with the entire absence of corrosion on the bearings and shafts lubricated with I, as shown by the photomicrographs removes all doubt as to preference.
Unused sample of Product I (unconfined). Magnification - 125 X.

Unused sample of Product I (confined under cover glass). Magnification 125 X.
Photomicrograph of Copper Lead type bearing used in Test No. LV. Evidence of chemical attack is apparent. Magnification - 125 X.

Photomicrograph of Copper Lead type bearing used in Test No. LVI. Chemical attack is not indicated. Magnification - 125 X.
Photomicrograph of Cadmium Silver type bearing used in Test No. LII. Chemical and mechanical attack of the bearing surface is indicated. Magnification - 125 X.

Photomicrograph of Cadmium Silver type bearing used in Test LII-A. The practical absence of chemical attack is indicated, showing effectiveness of inhibitors present. Magnification - 125 X.
Photomicrograph of steel shaft from Test LII. Corroded bearing alloy has been imbedded in the shaft. Magnification - 125 X.

Photomicrograph of steel shaft from Test LII-A. The surface is practically free from bearing metal and corrosion inclusions, indicating that inhibitors present have prevented both contact and corrosion. Magnification - 125 X.
Photomicrograph of oil from Test No. LII. Large particles of metal as shown were found to be present in considerable quantity. Magnification 125 X.

Photomicrograph of oil from Test No. VII-A. The low concentration of metal particles and their small size indicates that tearing has been minimized. Magnification - 125 X.
A study of these data indicates that the presence of colloidal graphite in lubricating addition products is desirable if it is present in suitable form and concentration.

To gain further information on the function of colloidal graphite, a series of tests were conducted with lubricants containing product I. The base lubricants to which this product was added ranged in viscosity from 10-W to S.A.E.-50. The oils selected for these tests consisted of standard grades of lubricating oils of paraffinic, mixed, and naphthenic origin.

For one of these tests, the colloidal graphite was removed from sample I by repeated filtration through an ultrafilter unit under vacuum.

After test, the above oil sample was subjected to analysis for metallic sludge. The following table reports this value together with corresponding metallic sludge analysis value found in the case of the untreated reference oil only, and a value for the product previous to the removal of its normal graphite content.

<table>
<thead>
<tr>
<th></th>
<th>Metallic Sludge % by Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plain oil (without additions)</td>
<td>.270</td>
</tr>
<tr>
<td>With Product I</td>
<td>.160</td>
</tr>
<tr>
<td>With Product I (Graphite removed)</td>
<td>.210</td>
</tr>
</tbody>
</table>

Assuming that 0.270% of metallic sludge is proportional to the wear produced when unmodified oil only is used as a
lubricant, it is apparent that the presence of the normal colloidal graphite content in this product is responsible for 45.5% of the total wear reduction which the product effected in these tests. This value is arrived at as follows:

\[
\text{Wear Reduction for Product I} = \frac{(0.27 - 0.16) \times 100}{0.27} = 40.7%
\]

\[
\text{Wear Reduction with Graphite Removed} = \frac{(0.27 - 0.21) \times 100}{0.27} = 22.22\%
\]

Reduction in Wear due to Graphite = 18.5%

\%

% of Total Wear Reduction due to Graphite = \[
\frac{18.5 \times 100}{40.7} = 45.5\%
\]

This product has been further studied in connection with oils of various viscosities and origins as follows.

Table J

Numbers in parentheses refer to tests of a series. The first number in all cases being the control or reference test without additions. Complete tabular data on these tests will be found in Section IX.

<table>
<thead>
<tr>
<th>Source of Oil</th>
<th>Bearings Used</th>
<th>Viscosity of Lubricant</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>10-W</td>
</tr>
<tr>
<td>Pennsylvania</td>
<td>Ag/Cd</td>
<td>(LII, LII-A, LIII, LIV)</td>
</tr>
<tr>
<td></td>
<td>Cu/Pb</td>
<td>(LVII, LVIII) (LV, LVI)</td>
</tr>
</tbody>
</table>
An inspection of the data outlined in Table K indicates that wear, temperature rise, torque, and seizure pressure, are uniformly improved by the addition of this product.
SECTION VII

THE COMBINED EFFECT OF CORROSIVE AND NON CORROSIVE FILM STRENGTH IMPROVING AGENTS

For the purpose of determining the effect of adding non corrosive film strength improving agents to corrosive film strength improving agents, a series of tests was conducted in which the test sample contained 3% each of tricresyl phosphate and the compound listed in column 1 of Table K which follows.

Table K

<table>
<thead>
<tr>
<th>Acid or Ester</th>
<th>Seizure Value Without Phosphate</th>
<th>Seizure Value With Phosphate</th>
<th>Detailed Test Data in Table</th>
<th>% of Corrosion Inhibited</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) &quot;Alketone&quot; C-32</td>
<td>1250</td>
<td>1650</td>
<td>XXXIX</td>
<td>68%</td>
</tr>
<tr>
<td>2) Oleic Acid</td>
<td>1250</td>
<td>1500</td>
<td>XXXII</td>
<td>80%</td>
</tr>
<tr>
<td>3) &quot;Alketoacid&quot; C-18</td>
<td>1500</td>
<td>1500</td>
<td>XXXV</td>
<td>100%</td>
</tr>
<tr>
<td>4) Palmitic Acid</td>
<td>1250</td>
<td>1425</td>
<td>XXXVI</td>
<td>86%</td>
</tr>
<tr>
<td>5) Ricinoleic Acid</td>
<td>1175</td>
<td>1250</td>
<td>XXXVII</td>
<td>94%</td>
</tr>
<tr>
<td>6) &quot;Alketone&quot; C-20</td>
<td>1000</td>
<td>1250</td>
<td>XXXVIII</td>
<td>80%</td>
</tr>
<tr>
<td>7) Reference oil only</td>
<td>750</td>
<td>----</td>
<td>I</td>
<td></td>
</tr>
</tbody>
</table>

The significance of the above tabular data is better understood by reference to the attached graphic presentation, Curve IV. The base line D has been drawn to correspond with the seizure pressure of the reference hydrocarbon oil when tested without additions. Another line has been drawn at 2000 lbs. to correspond with the seizure pressure when tricresyl
phosphate, 3% only, was added to the oil. Curve C was drawn connecting the seizure pressures for the acids and esters when added to oil but without the addition of tricresyl phosphate. Curve B connects the pressure values found when tricresyl phosphate was present in the lubricant along with the acid or ester indicated by the number at the bottom of the curve. These numbers correspond with the inhibitor values assigned in Curve II.

In view of the data summarized in a previous table, which indicated that increases in the concentration of oleic acid only in the test sample had no added effect on film strength, a similar series of samples was prepared and tested, containing fine concentrations of oleic acid and a constant quantity of tricresyl phosphate as shown in Table L. These results further tend to confirm the previously expressed view, and I believe quite generally accepted view, that carboxylic acid additions in quantity greater than is required to form an oriented layer over the surfaces offer no advantage.

If we interpret the distance X on Curve IV as a measure of the corrosive effect of tricresyl phosphate, and Z as proportional to the ineffectiveness of the inhibitor added, we may develop the following relations which makes possible the calculation of the per cent. efficiency of the various inhibitors tested.

Corrosion without inhibitor = X = A - D
Corrosion with inhibitor \( Z = (A - D) - (B - C) \)
\[ = A - B + C - D \]

Per cent. of Corrosion inhibited \( = \frac{100(A - B + C - D)}{A - D} \)

The values for the per cent. of corrosion inhibited column of Table K were calculated by the above formula.

<table>
<thead>
<tr>
<th>Table L: Oleic Acid</th>
<th>Tricresyl Phosphate</th>
</tr>
</thead>
<tbody>
<tr>
<td>1%</td>
<td>2%</td>
</tr>
<tr>
<td>3%</td>
<td>3%</td>
</tr>
</tbody>
</table>

| Reference Oil       | 96                  | 95                  | 94                  | 93                  | 91                  |
| 1%                  | 2%                  | 3%                  | 4%                  |

| Seizure Pressure    | 1500 lbs.          | 1500 lbs.          | 1500 lbs.          | 1500 lbs.          | 1500 lbs.          |
| 6%                  | 1575 lbs.          |

The limited amount of data reported here would indicate that any non corrosive film strength improving agent will, when added to a corrosive film strength improver, bring about certain changes in the performance of the combined sample which are complex functions, and which in any case are not additive as far as film strength or corrosion is concerned. It is to be observed that an addition agent which inhibits the corrosion of tricresyl phosphate completely also entirely overcomes its film strength improving properties; thus producing a net effect which is equivalent to the use of the inhibiting agent alone.

With oil samples containing phenyl chloride, the addition
of oleic acid increased the extent of corrosion as indicated by the following data:

<table>
<thead>
<tr>
<th>Table M</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>I. Oleic Acid</strong></td>
</tr>
<tr>
<td>Phenyl Chloride</td>
</tr>
<tr>
<td>Reference Oil</td>
</tr>
<tr>
<td>Seizure Torque</td>
</tr>
</tbody>
</table>

It is thought that the double bond in the oleic acid is involved in these results. The addition of one or more chlorine groups would be expected to produce such results as were observed.

Time did not permit the testing of more strongly Polar saturated carboxylic acids in connection with phenyl chloride.

The results with respect to tricresyl phosphate, and perhaps with respect to other corrosive agents which will not add to or form degradation products with carboxylic acids, may be summarized as follows.

<table>
<thead>
<tr>
<th>Table N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Classification of inhibitors when used in connection with corrosive products which will not form addition or substitution products with the inhibitor, and which have heats of wetting less than the inhibitor.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Molecule</th>
<th>Inhibitor Number of Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Alpha, beta and Gamma</td>
<td>10.0 - 9.5</td>
</tr>
<tr>
<td>-C = O and -OH substituted Carboxylic acids</td>
<td>10.0 - 9.5</td>
</tr>
<tr>
<td>Molecule</td>
<td>Inhibitor Number or Classification</td>
</tr>
<tr>
<td>-------------------------------------------------------------------------</td>
<td>-----------------------------------</td>
</tr>
<tr>
<td>2) Hydroxyl Substituted</td>
<td>9.5 - 9.0</td>
</tr>
<tr>
<td>Carboxylic acids (substitution beyond gamma position)</td>
<td></td>
</tr>
<tr>
<td>3) Saturated Carboxylic Acids</td>
<td>9.0 - 8.5</td>
</tr>
<tr>
<td>4) Unsaturated Carboxylic Acids and Hydroxy Ketones of low molecular weight.</td>
<td>8.5 - 7.5</td>
</tr>
<tr>
<td>5) Alcohol Ketones of High Molecular Weight</td>
<td>7.5 - 6.0</td>
</tr>
</tbody>
</table>

The method of tentatively assigning the inhibitor numbers will become apparent after inspecting Curve II.
SECTION VIII
CONCLUSIONS AND RECOMMENDATIONS FOR ADDITIONAL STUDY

The findings of the investigation reported here may be summarized under the heads which follow:

a) Unsaturated carboxylic acids do not corrode bearing metals but may accelerate the corrosive action of impurities which are present in lubricating oils. Acceleration in this case being closely related to the ease of formation of addition products by addition at the double bond.

b) The presence of chlorinated residues in petroleum oils would render the use of unsaturated carboxylic acids to lubricating oils hazardous to bearings.

c) Extreme pressure lubricants whose extreme pressure properties depend upon the action of chlorinated molecules can not be inhibited from attacking metal surfaces without loss of their extreme pressure properties.

d) It is proposed that the effectiveness of corrosion inhibitors can probably be classified according to the Polar groups present and the types of and locations of double bonds in their molecules, if due consideration is given to the structure, stability, and properties of the corrosive agent whose action is to
inhibited.

e) The reduction in friction losses attendant with the addition of orienting molecules to lubricating oils is thought to be the result of the attendant reduction in linkage of the electromagnetic fields of the frictional surfaces, as discussed in Section III.

f) The unit pressure at which rubbing surfaces will seize depends upon the bonding energy of the orienting molecules to the metal surfaces. It is thought that this value is closely related to the heat of wetting.

g) The ideal lubricant should contain orienting bodies of high bonding power with respect to all metals which it is expected to lubricate. The principle of selective adsorption should, therefore, be considered in formulating a lubricant which is to operate in zones bounded by dissimilar surfaces.

RECOMMENDATIONS FOR FURTHER STUDY

a) The effectiveness of saturated carboxylic acids and alpha, beta, and gamma carboxyl and hydroxyl substituted carboxylic acids as corrosion inhibitors should be investigated. These should be investigated in connection with aromatic halogenated compounds, sulphur derivatives, including sulphur chloride and nitrogen bases.
b) The general application of the corrosion inhibiting equation developed in Section VII should be thoroughly investigated. It should be studied with gas saturated media and through a range of temperatures adequate to prove or disprove or limit the practicability of the method. If the method should prove to be generally applicable it would offer a rapid concise method for gaining data with respect to corrosion, which by other methods requires months and years to gain imperfect data.

c) An adsorption dynomometer should be developed with a pressure applying mechanism actuated by weights and levers to eliminate errors and corrections of uncertain magnitude encountered with the spring unit used in the work reported here. Such a unit should be available at reasonable cost. It should preferably be equipped with an electrical arrangement for determining losses in order to eliminate mechanical difficulties.

d) The possibility of developing a procedure and equations for measuring the heat of wetting of metals with fluids by adsorption dynomometer methods should be thoroughly investigated. This is an important constant in lubrication and in other industrial activities and no means is at present at hand for its rapid and accurate measurement. The correlation between dynomometer seizure values and heat of wetting was pointed out in Section V of this report.
E) The comparative 'adsorption' values for colloidal graphite carboxylic acids and alpha, beta, and gamma substituted carboxylic acids should be thoroughly investigated.
SECTION IX - PART A

TABULAR DATA FROM MACHINE CALIBRATION AND TESTS WITH CHEMICAL ADDITIONS TO HIGHLY REFINED HYDROCARBON OIL
### Calibration of Absorption Dynamometer

(Pressure Draw Up Wheel)

<table>
<thead>
<tr>
<th>Gross Pressure in Pounds</th>
<th>RUN 1</th>
<th>RUN 2</th>
<th>RUN 3</th>
<th>RUN 4</th>
<th>Average</th>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<td>57</td>
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<tr>
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<td>56</td>
<td>58</td>
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<tr>
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<td>57</td>
<td>59</td>
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</tr>
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</table>

### Absorption Dynamometer Tests

With White Oil A as Lubricant

**I. Additions - None**

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<tr>
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<th>Notch Up (n)</th>
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<tbody>
<tr>
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<td>Notch Up (n)</td>
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<tr>
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<td>------------------</td>
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<td>1250</td>
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<td>48</td>
<td>2.08-6.8</td>
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</table>

<table>
<thead>
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<th>Notch Up (n)</th>
<th>t x 100 P</th>
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<td>54</td>
<td>1.6</td>
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</tr>
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</tr>
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<td>80(wear)</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Pressure (P)</th>
<th>Gross Torque (t)</th>
<th>Notch Up (n)</th>
<th>t x 100 P</th>
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<td>80</td>
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</table>
### V. ADDITIONS - HALOWAX 2025 3% (Chlorinated Naphthalene)

<table>
<thead>
<tr>
<th>Time</th>
<th>Gross Pressure (P)</th>
<th>Torque (t)</th>
<th>Notch Up (n)</th>
<th>$t \times 100$</th>
</tr>
</thead>
<tbody>
<tr>
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<td>-</td>
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<td>-</td>
</tr>
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<td>3</td>
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<td>7.5</td>
<td>48</td>
<td>1.5</td>
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<td></td>
<td>750</td>
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<td>58</td>
<td>1.7</td>
</tr>
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<td></td>
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<td>1250</td>
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### VI. ADDITIONS - TRIAMYLAMINE 3%

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<th>Torque (t)</th>
<th>Notch Up (n)</th>
<th>$t \times 100$</th>
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<td>7</td>
<td>45</td>
<td>1.4</td>
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<td></td>
<td>750</td>
<td>11</td>
<td>57</td>
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<td>4</td>
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<td>56</td>
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### VII. ADDITIONS - ALKETOACID C-18

<table>
<thead>
<tr>
<th>Time</th>
<th>Gross Pressure (P)</th>
<th>Torque (t)</th>
<th>Notch Up (n)</th>
<th>$t \times 100$</th>
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<td></td>
<td>1250</td>
<td>22</td>
<td>55</td>
<td>1.75</td>
</tr>
<tr>
<td>5</td>
<td>1500</td>
<td>27 (S-103)</td>
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### VIII. ADDITIONS - RICINOLEIC ACID 3%

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<th>Pressure (P)</th>
<th>Gross Pressure (P)</th>
<th>Torque (t)</th>
<th>Notch Up (n)</th>
<th>t x 100</th>
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<td>250</td>
<td>3</td>
<td>0</td>
<td>1.2</td>
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<tr>
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<td>7.5</td>
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### IX. ADDITIONS - TRIETHANOLAMINE 3%

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<td>7.5</td>
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<tr>
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### X. ADDITIONS - ISOPROPYL ETHER 3%

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<th>Gross Pressure (P)</th>
<th>Torque (t)</th>
<th>Notch Up (n)</th>
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</tr>
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</tr>
<tr>
<td>500</td>
<td>6.0</td>
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<td></td>
</tr>
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<td>8.8</td>
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### XI. ADDITIONS - LINOLEIC ACID 3%

<table>
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<tr>
<th>Time</th>
<th>Pressure (P)</th>
<th>Gross Pressure (P)</th>
<th>Torque (t)</th>
<th>Notch Up (n)</th>
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<tr>
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<td>Gross(P)</td>
<td>Torque (t)</td>
<td>Notch Up (n)</td>
<td>$\frac{t \times 100}{P}$</td>
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<td>--------------</td>
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### XII. ADDITIONS - PALMITIC ACID 3%

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<th>$\frac{t \times 100}{P}$</th>
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<td>1.25</td>
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<td>22.0(S-95)</td>
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### XIII. ADDITIONS - ALKETONE C-32 - 3%

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<th>$\frac{t \times 100}{P}$</th>
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<td>5.0</td>
<td>53</td>
<td>1.0</td>
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### XIV. ADDITIONS - ALKETONE C-20 - 3%

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<th>$\frac{t \times 100}{P}$</th>
</tr>
</thead>
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<td>47</td>
<td>1.3</td>
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### XIV. continued:

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<th>Notch Up (n)</th>
<th>( \frac{t \times 100}{P} )</th>
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</thead>
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<td>750</td>
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</tr>
<tr>
<td>4.25</td>
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### XV. ADDITIONS - STEARIC 3%
(Note: Part of Stearic Acid crystallized out during test.)

<table>
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<th>Gross Pressure (P)</th>
<th>Torque (t)</th>
<th>Notch Up (n)</th>
<th>( \frac{t \times 100}{P} )</th>
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<td>500</td>
<td>6.5</td>
<td>55</td>
<td>1.3</td>
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<tr>
<td></td>
<td>750</td>
<td>10.0</td>
<td>54</td>
<td>1.33</td>
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<td>54</td>
<td>1.50</td>
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<td>4.75</td>
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### XVI. ADDITIONS - UNDECYCLINIC ACID 3%

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<th>Notch Up (n)</th>
<th>( \frac{t \times 100}{P} )</th>
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<td>3</td>
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<tr>
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<tr>
<td></td>
<td>750</td>
<td>10.5</td>
<td>55</td>
<td>1.4</td>
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### XVII. ADDITIONS - TRICRESYL PHOSPHATE - 3%

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<th>( \frac{t \times 100}{P} )</th>
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</thead>
<tbody>
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<tr>
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<td>52</td>
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</table>
### XVII. continued:

<table>
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<th>Notch Up (n)</th>
<th>( \frac{t \times 100}{P} )</th>
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<tbody>
<tr>
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<td>1.47</td>
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### XVIII. ADDITIONS - DIETHYLENE GLYCOL MONO BUTYL ETHER 3%  
(Note: Not completely miscible with oil in above proportions.)

<table>
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<th>Notch Up (n)</th>
<th>( \frac{t \times 100}{P} )</th>
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<td>1.2</td>
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<td>51</td>
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<td>1.3</td>
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<td>58</td>
<td></td>
<td>1.6</td>
</tr>
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### XIX. ADDITIONS - LAURIC ACID 3%  

<table>
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<th>Torque (t)</th>
<th>Notch Up (n)</th>
<th>( \frac{t \times 100}{P} )</th>
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<tr>
<td>3</td>
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<td>0</td>
<td>1.2</td>
</tr>
<tr>
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<td>59</td>
<td></td>
<td>1.6</td>
</tr>
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### XX. ADDITIONS - TRIOXYMETHYLENE (1.7 g. in 65 cc. of oil)

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<th>Torque (t)</th>
<th>Notch Up (n)</th>
<th>t x 100 P</th>
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</thead>
<tbody>
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<td>0</td>
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</tr>
<tr>
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<td>54</td>
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</tr>
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<td>57</td>
<td>7.27</td>
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### XXI. ADDITIONS - BUTYL LACTATE - 3%

<table>
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<td>0.4</td>
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<td>1.1</td>
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### XXII. ADDITIONS - DIBUTYL PHTHALATE - 3%

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### ADDITIONS - PINE NEEDLE OIL 3%

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### ADDITIONS - THYMOL 3%

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<th>t x 100 P</th>
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<td>1.0</td>
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<td>1.3</td>
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### ADDITIONS - BENZALDEHYDE 3%

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<th>Notch (n)</th>
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<tr>
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<tr>
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<td>9</td>
<td>52</td>
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<tr>
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<td><strong>Gross Torque (t)</strong></td>
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<td><strong>t x 100 P</strong></td>
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<td>250</td>
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</tr>
<tr>
<td></td>
<td>500</td>
<td>7.0</td>
<td>56</td>
<td>1.4</td>
</tr>
<tr>
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<td>750</td>
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<td>1.6</td>
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<tr>
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<tr>
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<td>1250</td>
<td>80.0</td>
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<table>
<thead>
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<td><strong>Gross Torque (t)</strong></td>
<td><strong>Notch Up (n)</strong></td>
<td><strong>t x 100 P</strong></td>
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<tr>
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### XXXIII. ADDITIONS - OLEIC ACID 4% & TRICRESYL PHOSPHATE 3%

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### XXXIV. ADDITIONS - OLEIC ACID 6% & TRICRESYL PHOSPHATE 3%

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### XXXV. ADDITIONS - ALKETOACID C-18 3% & TRICRESYL PHOSPHATE 3%

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### XXXVI. ADDITIONS - PALMITIC ACID 3% & TRICRESYL PHOSPHATE 3%

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### XXXIX. ADDITIONS - ALKETONE C-32 & TRICRESYL PHOSPHATE 3%

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SECTION IX - PART B

TABULAR DATA FROM TESTS CONDUCTED ON CERTAIN MOTOR OIL ADDITION PRODUCTS AS SOLD IN AMERICAN AND FOREIGN MARKETS
**TESTS CONDUCTED ON CERTAIN MOTOR OIL
ADDITION PRODUCTS AS SOLD IN AMERICAN AND FOREIGN MARKETS**

REFERENCE OIL - Selective Solvent Refined Motor Oil
(Purchased in sealed container)

VISCOSITY - 140 sec., at 130°F. (Saybolt Universal)

BEARINGS USED - Inset construction, full faced Cadmium Silver bearing alloy.

PRESSURE - Held constant at 50 pounds gross.

In all cases the products subjected to test were added to the reference oil in the proportions recommended by the manufacturers.

<table>
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<th>Additions</th>
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<th>Cumulative Wear</th>
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SECTION IX - PART C

TESTS CONDUCTED WITH PRODUCT I IN CONNECTION WITH OILS OF VISCOSITY RANGING FROM 10-W TO S.A.E. 50 AS CONDUCTED ON FOUR TYPES OF BEARING SURFACES. THE OILS SELECTED FOR THESE TESTS INCLUDE REPRESENTATIVE SAMPLES OF PENNSYLVANIA (PARAFFINIC), MID CONTINENT (MIXED), AND CALIFORNIA (NAPHTHENIC) ORIGIN.
Oils of various origin were selected for these tests to gain information relative to the comparative performance of this particular product. The origin of oils used is as follows:

Pennsylvania (Paraffinic) -- LII to LXIV, LXVII to LXXIV
Mid-Continent (Mixed) ------- LXV, LXVI, LXXVII, LXXVIII
California (Naphthenic) ----- LXXV, LXXVI

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### LXI. ADDITIONS I VISCOSITY S.A.E.-20 BEARINGS-Lapped Steel

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SECTION X

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A. BOOKS


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DATE:

April 8, 1936