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PROPERTIES OF THE CARBIDE & CARBON SYNTHETIC POLYMER B-O-100 FLUIDS OF INTEREST IN LUBRICATION AND HYDRAULICS

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ABSTRACT

The chemical and physical properties of the Carbide & Carbon Chemical Corporation's B-O-100 synthetic polymer fluids essential in determining possible uses as lubricants and hydraulic fluids have been investigated as submitted by that Company and are presented herein. The fluids examined were designated by the producer as the 135B-O-100, 250B-O-100, and 400B-O-100 grades. Since these fluids were still in the development stage, it was necessary to gather independently and collate a large amount and variety of data concerning the properties and relative merits and faults of these fluids.

The B-O-100 fluids were found to have superior viscosity-temperature characteristics to conventionally refined petroleum oils. They are not as satisfactory in this respect as the "flat viscosity" petroleum oils made by adding synthetic polymers to a low flash petroleum oil, and are even less satisfactory than the methyl silicone polymer fluids. The evaporation rates are comparable to those of petroleum oils of equal viscosities. Pour points of between -50 and -60°F were found and in no case were precipitates or crystals observed even at -80°F.

The uninhibited fluids were found to be seriously susceptible to oxidative breakdown. However, the addition of the anti-oxidant recommended by the manufacturer was found to be remarkably effective, and accelerated oxidation tests in the presence of a variety of the common metals revealed these inhibited fluids to be of satisfactory stability for continued use at 250°F. When oxidation occurred under conditions of good aeration no precipitates or deposits were observed, while among the volatile products low molecular weight acids and aldehydes were identified.

The surface tensions of these fluids were not much different from those of petroleum lubricants. Their densities were very nearly equal to that of water at room temperature and decreased a few percent as the temperature approached the freezing point of water. They were found capable of dissolving from 1 to 2% of water at 140°F and from 3 to 5% at 32°F. Although they emulsified readily with water, the emulsions were not stable and were not much more viscous than the original fluid. These fluids were found to have adequate stabilities toward hydrolysis even at 212°F. The need for a rust inhibitor in these fluids for many conditions of use was revealed and a number of satisfactory inhibitors have been discovered. As the B-O-100 fluids are powerful solvents, it is important in using them to avoid prolonged contact with certain types of lacquered or painted surfaces.

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Although these fluids do not foam more than most of the commonly used petroleum lubricants and hydraulic oils, foaming difficulties may be encountered in some circumstances. This Laboratory found that the addition of approximately 0.01% of a silicone polymer fluid served as an effective anti-foam agent.

The inhibited fluids manifested excellent resistance to inflammability hazards such as would be indicated by measurements of the spontaneous ignition temperature, the spray inflammability, and incendiary fire tests using 50 caliber M-1 incendiary bullets.

Corrosion tests demonstrated that in ventilated systems inhibited B-O-100 fluids should be satisfactory as regards freedom from bearing corrosion.

These properties indicate that the inhibited B-O-100 fluids could be advantageously substituted for conventionally refined petroleum oils for use in internal combustion engines where unusually low temperatures or cold motor starting difficulties are encountered. They also have much to recommend them for use in internal combustion engines such as aviation engines where sludging and lacquer deposits can cause serious trouble. Another advantage of using them in aviation engines is due to the elimination of the combustion products of the lubricant by way of the exhaust system. These fluids are especially promising also for use where resistance to incendiaries or explosion damages is important. However, due to the greater change in viscosity with temperature than present polymer thickened petroleum hydraulic oils, they cannot be considered as non-inflammable substitutes for hydraulic oils now procured under specifications O.S.-2943 and AN-VV0-366b.



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INTRODUCTION

(a) Authorization

1. This investigation was authorized as part of Bureau of Ships project order No. 1121/42 and Bureau of Ordnance project order No. 40227 both of which concerned the development of non-inflammable hydraulic fluids and lubricants.

(b) Statement of the Problem

2. During the past several years the Carbide & Carbon Chemicals Corporation developed two new classes of synthetic polymer fluids in connection with their fellowships for research under the direction of Dr. Harvey R. Fife of the Mellon Institute. These fluids are designated by the producer as belonging to the B-50-50 and the B-0-100 series, the former being water-soluble and the latter only slightly water-soluble. The producer informed the Research Division of the Bureau of Ships of the existence and properties of these fluids in the summer of 1943. After a conference at the Navy Department on August 19, 1943 this Laboratory was requested to investigate the possibilities of these fluids for use as hydraulic and lubricating fluids.

3. The Carbide & Carbon Chemicals Corporation informed this Laboratory the B-50-50 fluids were in an advanced state of development, and a considerable volume was already being produced for the Army and several automobile producers for use as hydraulic brake fluids in automobiles and tanks. Patent applications had been filed by the inventors, W. J. Toussaint, F. J. Roberts, and Harvey R. Fife in patent applications serial No. 351,152 of August 3, 1942; serial No. 399,948 of June 26, 1941; and serial No. 437,722 of April 4, 1942. The B-0-100 fluids were a more recent development not in commercial or military use, and patent applications had not been filed by the inventors. An extensive series of tests on their properties as automotive and diesel engine lubricants had been carried on at the laboratory in Tonawanda, N. Y. of the Linde Air Products Division of the Union Carbide and Carbon Chemicals Corporation and were summarized in their Confidential Research Memorandum No. 37 by J. C. Kratzer dated August 6, 1943 and made available to this Laboratory.

4. After many of the tests described here had been completed the promising results obtained with the B-0-100 fluids led this Laboratory on January 29, 1944 to communicate with the Carbide & Carbon Chemicals Corporation urging that the inventors file applications for patents as early as possible

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in order to protect the interests of the company and to permit the Commissioner of Patents to impose a secrecy order on the entire development. The producer and the inventors were also urged in the meantime to adopt every reasonable precaution to keep the development confidential.

5. This progress report presents the results obtained through February 1944 of the investigation of the properties and possible uses of the B-0-100 synthetic polymer fluids. The results of the investigation of the B-50-50 fluids will be reported separately to prevent confusion between the two types of fluids many of whose properties differ greatly.

6. A careful examination of the properties of these new fluids was considered necessary not only because the producers had not had time to complete all the necessary investigations but also because such a pure polymer fluid had neither been used long enough nor in a wide enough variety of lubricating systems. It was also thought possible that the proper application, testing, and specification of these fluids could not be safely and entirely based on the principles and methods developed from past experience with petroleum or hydrocarbon oils.

(c) Known Facts Bearing on the Problem

7. From the patent applications on the B-50-50 fluids, from the information on the B-0-100 fluids supplied by Dr. Harvey Fife of the Mellon Institute Fellowship, the staff of the Linde Air Products Division at Tonawanda, New York, and Mr. D. B. Williams, Manager of the Process Division of the Carbide & Carbon Chemicals Corporation, and from recent research by this Laboratory, enough is known of the chemical constitution of these fluids and the anti-oxidant added to them to facilitate greatly the planning and execution of the tests described herein. However, nothing will be stated in this report concerning the mode of manufacture, the ingredients used, or the chemical composition of the final product. It is believed that such revelations would not materially assist in presenting the test data, the conclusions, or the suitability for applied uses of the product, while they would jeopardize the proper preservation of secrecy and the interests of the cooperating concern and laboratories.

(d) Commercially Available Materials

8. All samples tested were made in pilot plant lots and were obtained directly through the Carbide & Carbon Chemicals Corporation, New York City. The viscosity grades examined



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were those most readily made available by the producer. It is essential t emphasize that these fluids can be produced to have either lower or higher viscosities than the fluids described here. According to the producer these fluids are not blended from a few base stocks but are synthesized to the proper molecular weight and molecular weight distribution to obtain the desired viscosity, viscosity-temperature slope, and freedom from volatiles.

9. All of the B-O-100 series fluids have the same general chemical constitution, and that fact is indicated by the manufacturer's use of a common series designation "B-O-100". The manufacturer designates the approximate viscosity of any sample by a number preceding the series designation. Thus 250B-O-100 denotes a B-O-100 series fluid having a viscosity of approximately 250 Saybolt seconds at 100°F. The actual viscosities of the fluids submitted for these tests varied from 130 to 135 Saybolt seconds, from 250 to 260 and approximately 400 Saybolt seconds at 100°F.

10. At the request of this Laboratory samples were made available by the producer both with and without the anti-oxidant selected for use by the Carbide & Carbon Chemicals Corporation. Wherever in this report reference is made either to the "inhibited" B-O-100 fluid or to "the anti-oxidant", it will be meant that this particular anti-oxidant has been added to inhibit the oxidation of the polymer fluid. The inhibited B-O-100 fluids as normally supplied by the producer contain 2% by weight of the anti-oxidant. The samples tested and the manufacturer's designations are listed for reference in Table I.

RESULTS OBTAINED BY THIS LABORATORY

(a) Viscosity Temperature Characteristics by Helen F. Fobes and Charles M. Murphy

11. It has become increasingly important for the lubricants used in naval equipment to operate efficiently over wide extremes of temperature. As the viscosity is probably the most important fluid property of a liquid lubricant when operating under hydrodynamic conditions, the viscosity-temperature characteristics are basic data.

12. The kinematic viscosities of the B-O-100 fluids, both with and without the anti-oxidant, were determined in calibrated Cannon-Fenske type viscometers according to A.S.T.M. method (designation D445-42T). The baths were of the conventional type thermostatically controlled to within ± 0.03°F of the desired temperature for the determinations at 77°,

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100°, 130° and 210°F. A.S.T.M. kinematic viscosity thermometers calibrated by the National Bureau of Standards were used to measure the temperatures at the above mentioned points.

13. For the low temperature determinations a well insulated constant temperature bath with double windows was used. Temperature control was maintained by balancing a low wattage immersion heater against the cooling effect resulting from the subliming of dry ice contained in a compartment surrounding the liquid bath. Methyl cellosolve was used as the bath liquid. The temperature was measured by the E.M.F. from a 6 junction copper-cupron thermopile (calibrated at the freezing points of water and mercury and the sublimation point of solid carbon dioxide) in conjunction with a type B Rubicon potentiometer and a sensitive external galvanometer. The E.M.F. from the thermopile was 120 to 140 microvolts per degree F. Mercury-in-glass thermoregulators were used for all temperatures from 32° to -20°F. A bimetal regulator was used at -40°F and the temperature was controlled to ± 0.1°F.

14. The Cannon-Fenske viscometers were calibrated at 100°F. At all other temperatures a correction factor must be applied to compensate for changes in the dimensions of the viscometer and in the volume of the liquid. This factor has been determined over the range of 60° to 210°F, and the values for the lower temperatures were obtained by extrapolating the curve through the known points to the desired temperature.

15. The kinematic viscosities of the uninhibited and the inhibited B-0-100 fluids are given in Table II. The values for the inhibited fluids are plotted in Plate 1 on A.S.T.M. viscosity-temperature chart paper. For comparative purposes the characteristics of typical petroleum oils having corresponding viscosities are also shown. The Navy Symbols of the particular oils used were 1047 (SAE 10 and SAE 20W); 1080 (SAE 50); 2110 (SAE 10); and 2135 (SAE 20). The above fluids are forced-feed type lubricants. The 1000 series are 100% mineral oils having high viscosity indices. The 2000 series are 100% mineral oils having low viscosity indices. The 2000 series oils may contain additive agents as approved by the Navy.

16. Of the several methods of rating fluids according to the viscosity-temperature characteristics the most common are the "A.S.T.M. slope" and the "Viscosity Index." The A.S.T.M. slope is the tangent of the angle made by the viscosity versus temperature curve on the A.S.T.M. chart and is measured linearly. The lower the value of the slope the less

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is the change in viscosity with temperature. The slope method is quite useful in comparing fluids of the same viscosity grade. However, it is not applicable for comparing fluids of widely different viscosities since the A.S.T.M. chart is a log log versus log chart and therefore becomes more condensed at higher viscosities.

17. The viscosity index or V.I. is a figure calculated from the known viscosities at 100° and 210°F according to the following equation: V.I. = $\frac{L-U}{L-H}$ x 100. "U" is the viscosity of the oil at 100°F and "L" and "H" are the viscosities of hypothetical oils having the same viscosities at 210°F as the oil in question. Hypothetical oil L has a viscosity index of 0, and oil H a viscosity index of 100. The greater the numerical value of the viscosity index the better the viscosity temperature relationship. A viscosity index of 100 is considered good for a petroleum oil not containing polymer thickeners and is about as high a rating as can be obtained by present industrial refining methods. As the reference temperatures are 100° and 210°F this method gives no information as to the low temperature characteristics of the oil in question.

18. As would be expected, the addition of 2% of the Carbide & Carbon anti-oxidant affected the viscosity of the B-0-100 fluids. The viscosities of the fluids were increased, the increases becoming more pronounced at low temperatures. Thus the addition of the anti-oxidant affects the viscosity-temperature characteristics of the fluids adversely. This is apparent from a comparison of the A.S.T.M. slope and the viscosity indices of the uninhibited and inhibited fluids. The detailed results are shown in Table II.

19. The viscosity versus temperature graphs of the B-0-100 fluids when plotted on A.S.T.M. viscosity-temperature chart paper (D341-39) deviate from linearity at the extremes of temperatures. The deviations are upward at both ends of the curves. Petroleum oils generally show deviations from linearity at low temperatures over the temperature range investigated, while at higher temperatures they tend to curve downward. Compensation has been made for this downward curvature in the A.S.T.M. chart which is a plot of log log (η + 0.8) against log T, where η is the viscosity in centistokes and T is the temperature in degrees Rankine. Thus the constant (0.8) in the equation is of the wrong magnitude for the B-0-100 fluids. Plots of log log η versus log T result in straight line graphs at the high temperatures for all but the 400 fluid which shows a slight curvature upward at 210°F. As might be expected, there is an upward curvature at the low temperature end for all three fluids.

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20. The viscosity-temperature characteristics of the B-0-100 fluids are superior to those of conventionally refined petroleum oils not containing polymer viscosity modifiers. This superiority is particularly evident at low temperatures as shown in Plate 1. Navy Symbol 2110 and 1047 oils correspond in viscosity at 130°F to the 135B-0-100 fluid. Viscosity indices of 89 and 113 respectively of the petroleum oils as compared to 144 for the inhibited 135B-0-100 fluid reflect its superior viscosity-temperature characteristics over the range 100° to 210°F. At 0°F the 2110 oil did not flow through the viscometer capillary, and the 1047 oil behaved like a non-Newtonian fluid, i.e., the times of flow through the viscometer decreased for each successive viscosity determination and on long standing the time of flow increased. The A.S.T.M. slopes were calculated for the interval, 0° to 210°F. (Where the viscosity could not be determined at 0°F the slope was found over the interval 32° to 210°F.) The slopes of the 2110 and 1047 petroleum oils are 0.765 and 0.730 respectively as compared to 0.676 for the 135B-0-100 fluid.

21. Navy Symbol 2135 oil corresponds in viscosity at 130°F to the inhibited 250B-0-100 fluid. The superior viscosity-temperature characteristics of the B-0-100 fluid is shown by the V.I. of 143 and viscosity slope of 0.645 as compared to a V.I. of 40 and a slope of 0.825 for the 2135 petroleum oil. The 2135 oil was non-Newtonian in behavior at 0°F.

22. The 400B-0-100 fluid and Navy Symbol 1080 oil are of comparable viscosities at 210°F. A viscosity index of 141 and a viscosity slope of 0.591 as compared to a V.I. of 102 and a slope of 0.664 for the 1080 oil reflect the better viscosity-temperature characteristics of the 400 fluid. The 1080 oil exhibited non-Newtonian characteristics at 32°F and did not flow at 0°F.

23. The viscosities of the inhibited B-0-100 fluids were determined at -20°F, and no evidence of their being non-Newtonian at this temperature was observed. No attempt was made to determine their viscosities at -40°F as the times of flow in the available capillary type viscometers would have been excessively long.

24. The B-0-100 fluids unlike the conventional petroleum oils are not conveniently classifiable according to the SAE viscosity grading system. This can be seen from a consideration of the viscosity requirements of the various SAE grades.

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Viscosity in Centistokes

SAE Grade	At 0°F	At 130°F	At 210°F
10W	1089-2179		
10		18.2-25.3	
20W	2179-8716		
20		25.3-39.9	
30		39.9-55.2	
40		55.2-	-15.6
50			15.6-21.6
60			21.6-26.2

The inhibited 135B-0-100 fluid is in the SAE 10 and SAE 10W viscosity classifications. The 250B-0-100 fluid is in the SAE 20 and SAE 20W classifications. The 400B-0-100 fluid is classified as SAE 50 at 210°F, as SAE 30 at 130°F, and as SAE 20W at 0°F. It is possible for petroleum oils to fall in a certain SAE grade and also conform to the corresponding W grade requirement as do the 135B-0-100 and 250B-0-100 fluids. Since the 400B-0-100 fluid may be classed as SAE 30 and SAE 50, it is evident that this system of grading leads to absurd and confusing results when applied to the B-0-100 fluids.



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Conclusions

25. The B-O-100 fluids are superior as regards viscosity-temperature characteristics to conventionally refined petroleum oils not containing a polymer thickening agent. This is evidenced by their higher viscosity indices and lower A.S.T.M. viscosity slopes. They are also liquid at much lower temperatures. The uninhibited B-O-100 fluids have better characteristics than the inhibited fluids. The more favorable viscosity-temperature characteristics and lower pour points indicate that these fluids would be advantageously substituted for conventionally refined petroleum oils where extreme cold weather makes starting a cold motor difficult. The SAE system of classifying oils cannot be applied to the B-O-100 fluids. More nearly linear curves are obtained if log log η is plotted against log T rather than when plotted on A.S.T.M. viscosity-temperature chart paper.



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(b) Volatility Effects by Helen F. Fobes and Charles M. Murphy

26. The evaporation rates of the three viscosity grades (135, 250, and 400 of the B-0-100 fluids), both inhibited and uninhibited, were investigated because of their relation to oil consumption and constancy of viscosity. Fluids containing large amounts of volatile constituents may have excessive evaporation losses in storage and also under service conditions where elevated temperatures may be expected. Not only would high evaporation losses necessitate frequent additions of the lubricant, but the loss of volatile components could result in increasing the viscosity and in raising the pour point.

27. The rate of evaporation was determined by measuring the change in weight with time of a standard sample in a standard container under controlled conditions of heating and circulation of air. Crystallizing dishes 45 mm. inside diameter and 35 mm. deep containing 10 g. samples of each of the fluids to be tested were maintained at a temperature of 150°F ± 0.5°F in a convection type drying oven. The liquid surface exposed was 16 sq. cm. in area. Each sample was collected and weighed at intervals of approximately 24 hours until 210 hours of exposure had elapsed. At the end of the test the viscosity of the non-volatile fraction was determined at 100°F and the change in viscosity calculated.

28. The evaporation curves of the three inhibited fluids (Plate 2) were quite similar, having the same general shape but differing in the magnitudes of the evaporation losses. During the first 24 hours there was a rapid evaporation of the volatile components followed by a period of relative constancy in weight. Another period of evaporation was observed between 72 and 96 hours, as evidenced by a further loss in weight. After that only slight changes in weight with time were observed. As would be expected the least viscous fluid, 135B-0-100, had the greatest evaporation loss (0.7%). The 400B-0-100 fluid lost 0.4% in weight and the 250B-0-100 fluid 0.3% in weight. Such losses are approximately of the same magnitude as those usually encountered with petroleum oils of comparable viscosities.

29. The viscosities of these fluids and of Naval Specification petroleum oils of comparable viscosities were determined before and after evaporation and are given in Table III. The viscosities of all of the inhibited fluids increased during the tests, the viscosity increases being 1.30%, 0.94%, and 1.25% respectively for the 135, 250, and 400B-0-100 fluids.

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Such small rates of increase in viscosity would be considered of little consequence for most applications, and are only slightly greater than those of petroleum oils of comparable viscosity.

30. The evaporation rates of the uninhibited fluids were much greater than those of the corresponding inhibited fluids. The evaporation rates varied considerably during the first 96 hours for each of the uninhibited fluids. After that the evaporation rates were relatively constant as evidenced by the approximate linearity of the curves. However, the rates were different for the various fluids. The total losses in weight in 210 hours were 3.7%, 2.1%, and 3.2% respectively for the 135, 250, and 400B-0-100. Thus the weight losses were from 5 to 10 times greater than the values obtained for the inhibited fluids for for petroleum oils.

31. The viscosities were determined before and after the evaporation tests and are given in Table III. The viscosities of all the uninhibited fluids decreased during the tests, the decrease being 5.06%, 4.37%, and 19.3% respectively for the 135, 250, and 400B-0-100 fluids. The larger decrease in viscosity of the 400 fluid is due to the well-known rapid rise in the viscosity of a solution with the increase in the molecular weight of the polymer added.

32. The evaporation curves of the inhibited fluids were typical of the effects to be expected from non-volatile fluids containing small amounts of volatile constituents (see Plate 2). They differed, however, in that a second period of evaporation was observed between 72 and 96 hours. This period was preceded by a slight gain in weight. This could be due to oxidation of the less stable constituents. On continued oxidation these molecules "cracked" splitting into smaller, more volatile molecules. This would account for the loss in weight observed at 96 hours. Little or no change in weight was observed after this, presumably due to the effectiveness of the anti-oxidant.

33. The peculiar shape of the evaporation curves of the uninhibited fluids and the anomalous results, that the viscosity decreased after the evaporation test, are evidence that some change other than vaporization was taking place. In section (d) of this report it is shown that the uninhibited fluids are readily oxidized at 175°F with the formation of volatile acidic products. The oxidation rate of a large number of reactions may be expressed as an exponential function of time. Frequently such reactions are preceded by an "induction" or "activation" period. At some point in the oxidation reaction



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the oxidized molecules "crack" splitting off volatiles at the test temperature. The amount of volatile products formed is a function of the number of molecules oxidized. Thus the vaporization of the cracked products may be represented by an exponential curve starting after an induction period. Such a curve is presented as A in Plate 3. If the material originally contained a volatile component, the weight loss in the absence of any other effect would follow a curve like that of B of Plate 3. The observed evaporation curves of the uninhibited fluids are quite similar to the composite curve C made by summing A and B.

34. The inhibited B-O-100 fluids under these test conditions have evaporation losses of approximately the same magnitude as some familiar naval specification petroleum oils of corresponding viscosities.

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Conclusions

35. The uninhibited B-O-100 fluids showed large changes in weight with time at 150°F. The losses in weight were due to volatiles originally present and to volatiles formed by oxidation and cracking of the polymers during the test, the latter effect becoming dominant after a time. This resulted in a decrease in viscosity and the formation of acidic products. It is evident that the uninhibited fluids cannot be safely used as lubricants in many systems even at a temperature as low as 150°F. However, when the anti-oxidant was added all the B-O-100 fluids showed remarkable decreases in their susceptibilities to oxidation at 150°F. The evaporation rates were comparable to those of petroleum oils of the same viscosities.

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(c) Storage Behavior at Different Temperatures by Hayward R. Baker.

36. At the lowest temperatures of use or storage lubricating fluids should not solidify, develop precipitates, or crystallize. Although the A.S.T.M. cloud and pour point tests have been widely used to determine the tendencies of petroleum products to develop precipitates and to solidify, such tests have not proved entirely satisfactory in predicting the low temperature performance of those products. They are even less reliable for non-petroleum fluids. Also, many fluids exhibit the tendency to supercool. The length of time a liquid can remain in such a metastable state varies considerably with the composition of the liquid, its previous thermal history, and the physical conditions of storage. It is possible (due to supercooling) that no precipitates or crystallization will be observed with the A.S.T.M. cloud and pour point tests. Yet precipitates or crystals may develop on long storage or under different test conditions.

37. Samples of the 135, 250, and 400B-0-100 fluids, both with and without the oxidation inhibitor, were sealed in glass test tubes 25 mm. in diameter and 150 mm. long approximately two-thirds full and stored for one month in a constant temperature chamber held at -40° ± 2°F. The samples were inspected and shaken once each day. At the end of that time no crystals had formed, and all samples were found to flow when the tubes were held at an inclination of 45°.

38. The storage temperature was then reduced to -50°F. After 24 hours it was found that when the tubes were held at an inclination of 45°, flow was observed after the intervals indicated below:

Test Method	Time
Uninhibited	
135B-0-100	5 seconds
250B-0-100	5 "
400B-0-100	25 "
Inhibited	
135B-0-100	5 seconds
250B-0-100	10 "
400B-0-100	35 "

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39. After 24 hours at -60°F none of the six samples would pour and all appeared solid. They were still optically transparent without any indication of the formation of crystals. Even after 24 hours at -80°F no further change in appearance was observed.

40. The temperature was then raised to -50°F where it was held for two weeks. During this time the fluid was observed and shaken once each day. It was found that when the tube was held at an inclination of 45°, flow could be observed after the following intervals:

Test Fluid	Time
Uninhibited	
135B-0-100	5 seconds
250B-0-100	10 "
400B-0-100	30 "
Inhibited	
135B-0-100	5 seconds
250B-0-100	15 "
400B-0-100	40 "

The approximate agreement of the pour points before and after a cooling and heating cycle indicate stability of the pour-point.

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Conclusions

41. The samples of both the inhibited and uninhibited B-O-100 fluids poured after one month of storage at -40°F and also after storage for 24 hours at -50°F. None of these fluids poured after 24 hours of storage at -60°F. During these tests as well as a storage test at -80°F for 24 hours no formation of crystals was observed. The presence of the oxidation inhibitor somewhat increases the pour-point of these fluids.



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(d) Oxidation Characteristics by George M. Hain

42. Stability to oxidation, particularly at elevated temperatures, is one of the most important requirements for a lubricating or a hydraulic fluid. Oxidation of the lubricant may render it unfit for use by causing formation of sludge and lacquer, by generating acid materials which attack metals (particularly the bearing metals) and by greatly changing its viscosity. The results are presented here of tests in glass systems in order to study the oxidation of the B-O-100 fluids under closely controlled reproducible conditions.

43. Samples of 135B-O-100, 250B-O-100, and 400B-O-100 fluids were each exposed to accelerated oxidation at constant temperature by air bubbled through fritted Pyrex glass plates. The containers were 38 x 300 mm. Pyrex test tubes which held the 175 gram samples with enough clearance at the top to avoid high entrainment or foam losses. An oxygen-type air regulator valve with auxiliary needle valves provided a uniform flow of air at 80 cc/min through each tube, a rate high enough to ensure good contact without foaming over. The air from the regulator passed through a tower of soda-lime to remove moisture, carbon dioxide, and acid vapors before reaching the individual needle valves, then through the fritted plates, rising to the surface as fine bubbles, and out to the air through gas washing bottles filled with 100 cc each of distilled water.

44. The progress of any significant oxidation was followed by titrating the water in the gas washing bottles for volatile acid content with 0.1 N potassium hydroxide, using phenolphthalein indicator, and by determining the viscosity and acidity of the oil sample. For the acidity determination, five gram samples of the fluid were titrated in 50 cc of hot, neutral alcohol with 0.1 N aqueous potassium hydroxide, using phenolphthalein indicator. This procedure provided neutralization numbers precise enough to show the end of an induction period without seriously depleting the size of the sample under test.

45. Only a small sample was taken for each viscosity measurement. These determinations were run in modified Ostwald viscometer tubes, after which the samples were returned to their respective test tubes for the remainder of the run.

46. Uninhibited samples of 135B-O-100, 250B-O-100, and 400B-O-100 oxidized very rapidly at 175°F, as evidenced by

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a decrease in the viscosity and a rapid increase in the neutralization number of the fluid (see Table IV). Large quantities of water-soluble acids were evolved. No sludge precipitated out of the oil under the conditions of test, nor was any induction period observed at 175°F for the uninhibited stocks. In contrast to the uninhibited fluid, the B-0-100 fluids containing 2% anti-oxidant showed no sign of breakdown at 175°F in a 200 hour test (see Table IV).

47. A reserve concentration of anti-oxidant is believed to be highly desirable, since the number of hours of protection afforded by it is very roughly proportional to its concentration (see Plate 5). There is also the possibility of a small amount of the inhibitor being decomposed, adsorbed, or extracted. Under any circumstances the system in which the fluid is used should be checked for sources which might lead to such a removal of anti-oxidant.

48. Two percent of anti-oxidant seems to give ample protection since a sample of 135B-0-100 in the absence of catalytic metals for 200 hours at 200°F showed no sign of breakdown when protected by only 0.5% of anti-oxidant (see Table V). Increasing the test temperature to 250°F for the 200 hours test period caused no breakdown of B-0-100 fluids containing 2% anti-oxidant (see Table VI).

49. Metals and their corrosion or oxidation products which normally come in contact with a lubricating oil may accelerate its oxidation, as may certain additives serving as detergents, corrosion inhibitors, penetrants, or film strength improvers. It was therefore advisable to test the B-0-100 fluids containing 2% anti-oxidant in the presence of some of these materials. No significant breakdown occurred at 200°F after 200 hours with 17ST aluminum, copper, brass, or cold-rolled steel (see Table VII) or with any of a number of organic acids, metal soaps, and amines (see Table IX) of interest as rust inhibitors as outlined in Section (i) of this report.

50. Although copper and cold-rolled steel did not cause the inhibited B-0-100 fluids to oxidize past the induction period in 200 hours at 250°F, a three metal combination of high-lead bronze, chromium, and steel caused a powdery sludge to form. The bronze showed a loss of 1.79 mg. per sq. cm. of surface. However, no corroded areas were visible. The weight changes for the other metals were not significant. No varnish appeared on the metal surfaces, although a thin, hard deposit of yellow lacquer was observed at the air-liquid

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line on the glass tubes at this temperature. The three metal combinstion did not carry the fluid past the induction period of oxidation either, although Plates 8 and 9 show evidence of catalysis.

51. Catalyzed by copper naphthenate (Harshaw Chemical Co., 11% copper), however, breakdown started after approximately 70 hours at 200°F with 0.25% anti-oxidant, 140 hours with 0.50% anti-oxidant, and after a period known to be longer than 211 hours with 1% anti-oxidant. The percentage of copper naphthenate used did not appear to be critical (see Table VIII and Plates 5, 6, and 7). These tests suggest a method for evaluating the anti-oxidant's protective action in a shorter time by determining the duration of the induction period for a given sample after diluting it with uninhibited stock to which a small percentage of copper naphthenate has been added. A copper content of .01% gives marked acceleration.

52. Because the B-O-100 fluids contain combined oxygen, a test was also run on the uninhibited 250B-O-100 fluid sealed under nitrogen. Runs were made with and without copper naphthenate. No change in acid values occurred in 140 hours, indicating good thermal stability of the fluids.

53. The oxidation of inhibited B-O-100 fluids appears to take place in two stages. In the first stage little or no change in viscosity is observed; the acid number remains low but often rapidly attains a value somewhat higher than the initial value; volatile acids appear in the effluent air in small amounts, which when dissolved in water result in a pH between 2 and 3. This agrees with the results described in section (b) of this report on the evaporation rates which indicate that one portion of the oxidized products is a low molecular weight fraction. The metals tested, excluding copper, its alloys, and its metal soaps, do not increase the rate of evolution of volatile acid at this stage (see Plates 4, 8, 9, and 10).

54. This first stage of oxidation, which might be called the induction period, represents the period of safe use for the lubricant, and it is followed by a second stage characterized by rapid breakdown. The rate of evolution of volatile acids becomes very rapid, and there occurs a change in the nature of the products. This change is evidenced in the titration for acids since the indicator keeps fading over a wide range of alkali addition evidencing the presence of easily saponifiable matter which is not necessarily acid.

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55. In the breakdown stage the viscosity of the fluid drops very rapidly, and its neutralization value soon reaches a value usually associated with the pure fatty acids (see Plates 5, 6, and 7 and Table VIII).

56. Thus far, no analysis has been made of the complex breakdown products although the presence of formaldehyde as well as a short chain acid such as acetic acid is indicated by the odor of the effluent gas and by the pH of the water in which it has been dissolved. Neither inhibited nor uninhibited fluids form precipitates on oxidation. Uninhibited fluids are not darkened while inhibited fluids, which darken in early stages of oxidation, may become much lighter in color when breakdown occurs.

57. As a consequence of the high susceptibility to oxidation of the unprotected oil, care should be exercised to preserve the anti-oxidant content, even though the reserve protection seems ample under all ordinary circumstances. It can easily be visualized what serious damage could occur in a precision machine running on the broken-down fluid. It is also obvious that filters capable of selectively adsorbing the anti-oxidant are to be avoided.

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Conclusions

58. The inhibited B-0-100 fluids have satisfactory resistance to oxidation. Once the inhibitor has been consumed the rate of oxidative breakdown increases rapidly with a corresponding large rise in the rate of evolution of acidic material, an increased rate of rise in the neutralization number, and a rapid decrease in the viscosity. However, up to a temperature of 250°F the B-0-100 fluids may be expected to be adequately stable toward oxidation. Their inhibitor protection seems ample even in the presence of a copper catalyst. Further tests are now in progress at temperatures above 250°F with a wider range of metals and in comparison with various mineral oils under similar conditions. Due to the evolution of volatile acids after the induction period, caution is recommended in using it in closed or inadequately ventilated systems containing metals susceptible to corrosion by such acids.



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(e) Inflammability by Miles V. Sullivan and John K. Wolfe

59. The fire hazards involved in the use of polymer thickened petroleum-base oils in high pressure hydraulic lines of ships and airplanes have been widely recognized since the commencement of the Second World War.

60. Probably the most frequently encountered fire hazard is the exposure of a fluid to a spark or flame source and the subsequent propagation of the flame over the surface of the liquid. Ignition will occur when the vapor pressure of the liquid is sufficiently high to produce a combustible mixture with air. The probable incidence of fire under these conditions can be ascertained by a determination of the flash and fire points of the fluid.

61. Hydraulic fluids are frequently exposed to certain types of hazards in warfare. One such hazard is the incidence of local ignition when an oil spray or a stream of oil strikes a hot surface such as an exhaust pipe or a high pressure steam line. This hazard may be studied by the measurement of the spontaneous ignition temperature. Another hazard encountered in modern warfare is that resulting from the penetration or rupture of an hydraulic oil accumulator or a high pressure hydraulic line by an incendiary bullet or shell fragment and the production of a fine spray of the hydraulic fluid either directly from the violent explosion or indirectly by rapid passage through an orifice formed. The propagation of a flame front through such a spray can be measured by a spray test developed at the Naval Research Laboratory.

62. The penetration of hydraulic lines by high explosive or incendiary missiles and the subsequent explosion which results from the spray represents another important type of inflammability hazard. The incidence of fire under these conditions can be measured by firing incendiary bullets into samples of the fluids.

63. Flash and fire points were taken under standardized conditions by slowly heating the liquid in an open metal cup (in some cases a closed cup may be used) and passing a small flame over the cup at specified intervals. The flash point was taken as the lowest temperature at which a flash of fire appears on the surface of the liquid. The lowest temperature at which the liquid ignited and continued to burn for a period of at least five seconds was taken as the fire point.



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64. The Cleveland open cup flash and fire points of B-O-100 fluids are given in Table XI. As would be expected, the low viscosity fluid has the lowest flash and fire points. This value, however, is only slightly lower than that observed for the medium viscosity fluid. The heavy fluid (400B-O-100) has flash and fire points in the same range as the medium 250B-O-100 fluid. The presence of the anti-oxidant makes little difference in the flash or fire points observed. It is concluded from this data that relatively little fire hazard exists from the inflammability of these fluids in the presence of a small flame or spark passed over or near these fluids in any apparatus which operates at temperatures below 500°F. This conclusion does not hold when the source of flame carries with it hot metal or combustible materials which will raise the temperature of the fluid in the vicinity of the flame.

65. The spontaneous ignition temperature (also known as the autogenous ignition temperature) is the lowest temperature at which a small volume, usually a calibrated drop, spontaneously ignites when dropped on a hot surface in an enclosed space. The apparatus used consisted of a stainless steel cup with a side inlet to furnish preheated fresh air and provided with a thermocouple for temperature measurement. The steel cup was fitted with a flat cover having a small hole in the center through which the drop of oil was admitted. The air was passed through the cup at a measured rate. Drops of fluid were added and the lowest temperature at which the drop spontaneously ignited was noted. The apparatus used was described by Sortman, Beatty, and Heron in the Symposium of the Petroleum Division of the American Chemical Society, Detroit meeting September 1940, p. 101.

66. Table XI contains the values determined for the spontaneous ignition temperatures (S.I.T.) of the B-O-100 and several other fluids of naval interest. It will be noted that the S.I.T. changed little with viscosity for each type of fluid. As might be expected, the viscosity varied considerably with the chemical nature of the fluid. The addition of the anti-oxidant produced little change in the S.I.T. It is concluded on the basis of these results that the fire hazards resulting from a spontaneous ignition of this fluid are not likely to be encountered at temperatures below 700°F. In predicting the inflammability of a fluid under such circumstances, it is important to note that the value of the S.I.T. must be considered as a minimum above which ignition can occur and at which fire will not necessarily result in actual practice. This is the case since the vapor concentration in the vicinity of the open or exposed hot surface is not directly comparable to the concentration during the measurement of the S.I.T. made in an enclosed space.

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67. The inflammability of an air spray formed from each of these fluids and the ease of propagation of the flame back to the source of the spray was measured by a method recently developed by this Laboratory. This test has been described in NRL report No. P-2165 dated 28 September 1943. The test consists of determining the minimum oxygen concentration of the atmosphere containing the spray to permit a flame to be self-propagating. Ignition of the spray was accomplished by the use of an intense electrical arc discharge. The proportion of oxygen necessary in that atmosphere was used as a relative measure of the inflammability. Insofar as possible, the other factors affecting the determination were held constant.

68. The results of the spray tests on the B-O-100 synthetic fluids are given in Table XI. All of these fluids propagated a flame when dispersed as a mist in an atmosphere containing 52 to 56% oxygen. Small differences were observed between the various samples by this test, but these were not significantly different from the individual variability of samples. The presence of the anti-oxidant made little difference in the amount of oxygen necessary for propagation of the flame. Sprays of petroleum fluids made from low viscosity base stocks were found by this test to propagate a flame in an atmosphere of 12% oxygen, and this percentage held true for many petroleum hydraulic fluids of widely different viscosities. The higher boiling petroleum fractions used as lubricating oils, for example, N.S.-2135, required more oxygen to propagate a flame in the spray. Sprays of "Hydrolube" fluids made from a polymer thickened mixture of ethylene glycol and water required over 80% oxygen to propagate a flame.

69. Incendiary firing tests were conducted at the Naval Proving Grounds at Dahlgren, Virginia through the cooperation of the Research Division of the Bureau of Ordnance. The test consisted of firing a 50 caliber M-1 incendiary bullet through the length of a rectangular one gallon can of the fluid. Two 1/16 inch thick baffle plates were held in front of the can to cause fragmentation of the bullet and thus increase the inflammability hazard. Several independent tests were made on each fluid of interest.

70. A limited inflammability was observed with the 135B-O-100 fluids. Flames 2 to 3 feet in height and of short duration were observed when the incendiary pierced the one gallon can of the fluid. Samples of 250B-O-100 and 400B-O-100 fluids showed even less inflammability by this test method and only occasionally produced a flame discernible from that due to the ignition of the incendiary itself. The

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presence of the anti-oxidant appeared to decrease slightly the inflammability of these fluids. Petroleum fluids of corresponding viscosities produced much larger fires when tested by this procedure. The addition of anti-oxidants to petroleum fluids likewise has been observed to decrease the inflammabilities of these oils under incendiary fire. Hydraulic fluids containing a high percentage of chlorine and fluids prepared with ethylene glycol-water as a base stock showed no fire above that observed for the incendiary bullet itself.

71. Plate 11 contains several pictures of the results of firing one gallon of the O.S.-2943 flat viscosity, petroleum-base hydraulic oil due to one 50 caliber M-1 incendiary bullet. These pictures were printed from a roll of 16 mm Kodachrome film photographed at 64 frames per second. For illustrative purposes the plates printed here are composed of frames taken every 1/20 second. In each case the first frame is the incendiary burst and the last frame shows no fire (except O.S.-2943). In order to save space, only five of the twelve pictures required to show the total fire of O.S.-2943 oil are included in Plate 11. The relatively great extent of the fire (20 to 25 feet) is the main point established by these pictures. Also on Plate 11 is shown the spray formed when an incendiary bullet was fired into a one gallon sample of water under the same conditions.

72. Plate 12 contains pictures made of the firing tests on the 250B-0-100 fluid both with and without oxidation inhibitor present. The uninhibited samples burned producing flames 1 to 2 feet in height and lasting about 1/5 second. The test on the inhibited samples showed no fire.

73. Plate 13 contains pictures made of the firing tests on inhibited 135B-0-100 fluid and the uninhibited 400B-0-100 fluid. The highest viscosity fluid (400B-0-100) showed no fire and was thus less inflammable than the medium viscosity fluid 250B-0-100 (Plate 12). The lowest viscosity fluid (uninhibited 135B-0-100) burned producing flames 2 to 3 feet in height and lasting 1/5 second. The extent of this burning was slightly greater than that observed for inhibited 250B-0-100 fluid (Plate 12).

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Conclusions

74. These inflammability tests can be summarized as follows:

(a) The inflammability of the B-0-100 fluids increases with decreasing viscosity.

(b) The presence of the 2% of anti-oxidant appears to decrease the inflammability.

(c) The B-0-100 fluids are definitely less inflammable than petroleum lubricating oils of the same viscosities and are much less inflammable than Army and Navy Specification petroleum hydraulic oils made from a low viscosity base stock and a polymer thickener.

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(f) Surface Tension by Herbert A. Pohl

75. The surface tension is an index of the relative ability of a substance to leak through crevices and holes in containers, and to creep over surfaces.

76. Table XII gives the surface tensions of the various B-O-100 fluids received. The measurements were made using a Cenco du Nuoy tensiometer. The Harkins correction factors were applied to the apparent surface tensions as obtained from the dial readings. (W. D. Harkins and H. F. Jordan, J.A.C.S. 52, 175 (1930).

Conclusions

77. The surface tensions of the B-O-100 fluids are not greatly different from those of petroleum lubricants consequently, no abnormal behavior in this respect would be expected.

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g) Specific Gravity and Thermal Expansion by Helen F. Fobes and Charles M. Murphy

78. The specific gravities (referred to water at 60°F) of the inhibited B-0-100 fluids were determined over the temperature range -20° to 210°F. A Westphal balance was used, and the observed apparent specific gravities at temperatures other than 60°F were corrected for the changes in volume of the glass plummet.

79. The results are presented in Plate 14. The specific gravities of the three fluids increased with their viscosities, while the specific gravity versus temperature curves were parallel straight lines over the temperature range -20°F to 130°F. Between 130 and 210°F a slight upward curvature was found.

80. Comparisons of the specific gravities of the inhibited B-0-100 fluids with those of typical petroleum oils of comparable viscosities are given in Table XIII. The inhibited B-0-100 fluids are considerably more dense than petroleum oils having comparable viscosities. The differences are greater for "paraffin base" (1000 series) than for "naphthene base" (2000 series) oils.

81. The specific volumes or volumes per unit mass were calculated from the specific gravities of the inhibited B-0-100 fluids and are graphed in Plate 15. These curves also are parallel straight lines over the temperature range except for a slight upward curvature between 130° and 210°F.

82. The coefficients of thermal expansion of the inhibited B-0-100 fluids were calculated from the data on specific volumes of Plate 15 and are given in Table XIV. The coefficients of expansion of petroleum oils have been found to vary for the different specific gravity fractions. The petroleum oil group in the specific gravity range of 0.8504 to 0.9965 (referred to water at 60°F) has a coefficient of expansion of 0.00040 per degree Fahrenheit according to the "Supplement to National Bureau of Standards Circular C-410". This gravity group includes most of the lubricating oils in the SAE 10 to SAE 70 classifications.

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Conclusions

83. The specific gravities of the inhibited 135, 250, and 400B-0-100 fluids are close to unity at 50°, 60°, and 80°F respectively. They increased as the temperature decreased reaching values of from 1.01 to 1.02 at the freezing point of water. These fluids were considerably more dense than petroleum oils of comparable viscosities. The specific gravity versus temperature curves were linear and parallel over the temperature range -20° to 130°F with a slight upward curvature occurring between 130° and 210°F. The coefficient of thermal expansion of the inhibited B-0-100 fluids was found to be approximately 0.00040 per degree Fahrenheit, a slight increase occurring above 130°F. Petroleum oils of comparable viscosities have approximately the same coefficient of thermal expansion as the B-0-100 fluids.



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(h) Emulsion Characteristics and Water Solubility by Hayward R. Baker and William A. Zisman

84. In many types of instruments and machinery it is not possible to exclude water or moisture completely from the lubricating system. In internal combustion engines water is a product of combustion and also is used as an engine coolant. From both sources and from atmospheric condensation water may accumulate in the sump. Hence, data relative to the solubility for water and the water emulsifying characteristics of the B-O-100 lubricating fluid are essential.

85. Where much water is expected to accumulate in a lubricating system some means for removing it is often provided in the form of a centrifuge or a settling chamber. However, this will remove only that portion of the water separating from the emulsion and insoluble in the lubricant.

86. It will be shown in section (i) of this report that the corrosion caused by water dispersed or dissolved in the B-O-100 fluids can be prevented by using an organic rust inhibitor dissolved in the lubricant. However, even when such inhibitors are used it is desirable that the total amount of water present be kept at a minimum; otherwise water separating out from the emulsion will accumulate in pockets. Complete inhibition against corrosion under such circumstances is difficult to obtain.

87. If an emulsion of oil and water forms its viscosity must not be much greater than that of the original oil. Thus if a mayonnaise-like emulsion forms, it obviously will be difficult to pump through the lubricating system and the oil film responsible for the actual lubricating process will be difficult to maintain.

88. The characteristics of emulsions of B-O-100 fluids and water were determined as follows: The B-O-100 fluid and the water to be emulsified were preheated to the desired test temperature and were stirred while held at constant temperature for 10 minutes. During this time they were emulsified by a vigorous stirring at 5000 r.p.m. using a Hamilton Beach No. 33 milkshake stirrer. The emulsion formed was immediately transferred to a 100 ml, graduate and observations were made on the rate of separation. The volumes of the emulsion and the separated water were observed at the end of 5 minutes, 10 minutes, 30 minutes, 60 minutes, and 24 hours.

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89. The results of the emulsification tests on the B-O-100 fluids are recorded in Table XV. The observations are expressed in terms of the percentage of the total water placed in the system which had separated from the emulsion at the time specified. The effect of temperature on the stability of the emulsion was observed by repeating this test at temperatures of 34°, 77°, 140°, and 180°F.

90. It is evident from Table XV that the more viscous the polymer fluid the more stable the emulsion; also, the less water present the more stable the emulsion. As is characteristic of most emulsions, increasing the temperature decreases the stability. The emulsions with 10% of water of the unoxidized B-O-100 fluids containing the oxidation inhibitor did not break rapidly on standing. Even at 140°F after standing for 24 hours, 10% of the 135B-O-100 fluid, 40% of the 250B-O-100 fluid, and 50% of the 400B-O-100 fluid were still in the emulsion. Such relatively stable emulsions were always dispersions of water in oil.

91. The nature of each emulsion was observed by microscopic examination of the emulsions prepared with an oil-soluble or a water-soluble dye to color the separate phases. As a check the electrical conductance of each emulsion was measured by using a convenient platinum wire electrode conductance cell and a microammeter.

92. All emulsions of the inhibited 135B-O-100 fluids at room temperature containing 10% or more of water were almost entirely of the oil-in-water type. The inhibited 250B-O-100 fluid formed oil-in-water emulsions when 20% or more of water was present. The inhibited 400B-O-100 fluid required 30% or more of water to form oil-in-water emulsions. In every case when a large enough proportion of water was present mixed emulsions formed. If a small proportion of water was present mixed emulsions were formed; i.e. water-in-oil and oil-in-water dispersions were found in the same emulsion, and in most cases oil drops were found inside water drops and vice versa. These observations can be explained on the basis of the well-known empirical rules of emulsification. It would appear that the high molecular weight polymers present in these fluids are oil-soluble and are water-in-oil emulsifiers, while the low molecular weight polymers are water-soluble and oil-in-water emulsifiers.

93. An emulsion of inhibited 250B-O-100 and 10% of water was prepared at 100°F, and two minutes later the viscosity was measured and found to be 67.6 cs. The original viscosity of the fluid at 100°F was 56.1 cs., representing an increase

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in viscosity of 17%. Emulsions of the inhibited 400B-0-100 fluid and 10% water tested the same way resulted in an increase in viscosity on emulsification of 14%. Evidently under these conditions mayonnaise-like emulsions did not form.

94. Emulsion tests using the uninhibited B-0-100 fluids resulted in stabilities somewhat less than the values obtained with the inhibited fluids.

95. A sample of the inhibited 250B-0-100 fluid was exposed to oxidation conditions by bubbling air through it at the rate of approximately 80 cc. per minute for 200 hours at 175°F. Samples of the resulting fluid were then emulsified with 10% and 50% of water and the stabilities were measured. The results are recorded in Table XVI. Although the emulsions formed were somewhat more stable than those of the unoxidized fluids, the viscosity did not increase sufficiently to cause pumping or lubricating difficulties.

96. The oxidation inhibitor used by the Carbide & Carbon Chemicals Corporation is flesh-colored when first prepared. If, however, it is allowed to come into contact with the atmospheric air for a considerable time, it oxidizes and becomes reddish-brown in color. It was found that the B-0-100 fluids inhibited with such a partially oxidized inhibitor formed more stable emulsions with water than resulted with the fluid inhibited by the anti-oxidant purified.

97. Plate 16 gives the approximate solubility of water in the different inhibited B-0-100 fluids. The uninhibited and unoxidized fluids dissolved approximately 1% less water. These values were determined by adding distilled water 1 ml. at a time to a 100 ml. volumetric flask containing approximately 90 ml. of the B-0-100 fluid. After shaking it thoroughly and allowing it to stand the contents were observed for undissolved water using a flashlight beam to give the Tyndall effect. If 1 ml of water completely dissolved, the second ml. was added, etc. until the Tyndall effect was first observed. This procedure was carried out at the temperatures indicated on Plate 16.

98. As might have been expected water is more soluble in the 135B-0-100 fluid than in the more viscous fluids. These differed from most fluids in the effect of temperature on solubility, for as the temperature increased the solubility for water decreased. From the large proportion of hydroxyl-containing molecules (see section n), the molecular



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structure of these fluids, and this unusual temperature-solubility effect, it is suggested that much of the water dissolved in the B-0-100 fluid was present as water of hydration.

99. It was observed that the water contained in the B-0-100 fluids, whether in the dissolved or dispersed state, was easily and readily evaporated when an emulsion was stirred at temperatures above 140°F. With this occurring in an internal combustion engine the water would be quickly eliminated. However, unless a rust inhibitor is used such dissolved water is likely to be detrimental when the fluids are in use or storage at temperatures below 180°F.



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Conclusions

100. The inhibited B-O-100 fluids were found to dissolve from 1 to 2% of water in the range of 140°F to 180°F and from 3 to 6% near 32°F. The abnormal effect of a decrease in solubility as the temperature is increased is believed due to the water being present in the hydrated state. The inhibited B-O-100 fluids emulsified readily with water to form emulsions which were less stable the higher the temperature and the lower the viscosity. Oil-in-water emulsions were formed when the ratio of water to oil was large, and water-in-oil emulsions were formed when the ratio was large. It was concluded that the emulsifying properties of the high and low molecular weight polymers present in these fluids were entirely different. The water-in-oil emulsions were by far the more stable. An emulsion formed with 10% of water at 140°F after standing 24 hours still contained 33% of the unbroken emulsion of 135B-O-100, 40% of the 250B-O-100 and 50% of the 400B-O-100 inhibited fluids. However, the emulsions were not very much more viscous than the original fluids even after exposure to considerable oxidation.

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(i) Rust Inhibitors Soluble in B-O-100 Fluids by Hayward R. Baker and William A. Zisman.

101. When lubricants are used in equipment exposed to rain, mist or atmospheric condensation, some water is likely to accumulate in the fluid system to be dissolved or dispersed by the movement of the equipment. The petroleum industry has learned to add small percentages of oil-soluble organic compounds to reduce or prevent corrosion or rusting of metals in systems which are exposed to such water.

102. The B-O-100 fluids were tested using a modification of the standard A.S.T.M. turbine oil corrosion test designated as D665-42T. Because of the limited quantities of B-O-100 fluids available at the commencement of these studies, a modification of the standard turbine oil corrosion test was necessary. Instead of using the standard 300 ml sample with 30 ml. of water, 9 ml. of the B-O-100 fluid and 1 ml. of distilled water were tested in a flat bottomed pyrex glass 80 x 18 mm. cylindrical container. The specimen of cold-rolled steel used was 4-1/2" x 1/4" x 1/32" and was revolved at 1080 ± 20 r.p.m. to stir the mixture of oil and water. The lower 45 mm. of this specimen was observed for corrosion as it alone was in contact with the mixture. The specimen was so placed that the lower end came within 1/4" of the bottom of the container. The test was carried out at 140°F for 24 hours.

103. As shown in Plate 17 the B-O-100 fluids offer practically no protection against attack by water, and therefore a corrosion inhibitor is needed. It is believed that the greater water solvent power of this fluid (indicated in paragraphs 92-97) as compared with that of the uninhibited petroleum reference oil caused the rusting of the steel specimens to be worse for the B-O-100 fluids. In section (h) of this report it is indicated that at low temperatures as much as 6% of water can be dissolved in the B-O-100 fluids. These properties make it imperative that rust inhibitors be added for most conditions of use.

104. An earlier research project of this Laboratory was concerned with the type of rust inhibition obtainable when small portions of pure organic polar compounds or mixtures were added to petroleum oils. The knowledge developed of this protective action made it evident that the same principles could be applied to select such inhibitors for the B-O-100 fluids. It was realized that among the qualities to be looked for in a suitable inhibitor were that the additive must not

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be too soluble in the temperature range of operation, it must be sufficiently soluble not to precipitate out at the lowest temperature of storage, it must not be conducive to the formation of viscous emulsions, and it must not chemically deteriorate in use to form corrosive or other undesirable products.

105. With these requirements in mind the solubilities at various temperatures and the extent of corrosion inhibition by the modified turbine tests at 140°F were studied for each compound of interest. It was found that many inhibitors could be selected which were capable of performing in a satisfactory manner when used in quantities of less than 1% by weight. In this investigation any corrosion inhibitor needed in a concentration exceeding 1% was not studied further. The cold storage tests were made successively at 77°, 0°, -20° and -40°F, and were stopped when a temperature was reached at which precipitation of the inhibitor occurred.

106. In the absence of an anti-oxidant the B-O-100 fluids are readily oxidized in use and reactions may take place between the corrosion inhibitor and the oxidation products. Under such circumstances as soon as the corrosion inhibitor is rendered useless by the reaction, the water present can attack the steel in the system. When an effective anti-oxidant is present such as that recommended and used by the Carbide & Carbon Chemicals Corporation for these fluids, little oxidative breakdown of the B-O-100 fluids occurs, and the problem of obtaining a satisfactory rust inhibitor becomes simplified.

107. A numerical rating system was used in tabulating the results obtained in these tests. The corrosion of cold rolled steel observed under the test conditions using a Texaco uninhibited petroleum oil (Navy symbol 2135) was assigned a rating of 10. Samples of the fluid under test exhibiting a greater amount of corrosion were given ratings in excess of 10 while those showing less corrosion were rated less than 10. A rating of 0 was used to denote that no corrosion was observed under the test conditions.

108. That rust inhibitors have been found is evident from the data of Table XVII and also by a comparison of the photographs of Plates 17 and 18. Twelve of the corrosion inhibitors listed did not precipitate after two months of storage at -40°F and were effective at concentrations of less than 1%. Three other inhibitors were found of equal effectiveness as long as the storage temperatures did not go below -20°F. Many other products closely related to these compounds were also effective. These inhibitors were found

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to be stable under oxidation test conditions as reported in section (d) and did not interfere with the effective action of the anti-oxidant. It is also important to note that they did not cause any significant increases in the stabilities or viscosities of emulsions formed with water. This will be evident from an inspection of Table XVIII on the effect of the rust inhibitor on the stabilities of the emulsions formed by the procedure described in section (h).

109. Modified turbine corrosion tests were made using synthetic sea water instead of distilled water. At 140°F it was found that within one hour enough water had evaporated from the emulsion to cause the salts in the sea water to separate out as crystals. These deposited over the surface of the cold-rolled steel specimens resulting in corrosion.

110. To minimize this difficulty caused by evaporation, tests were run at 90°F. Even at that temperature the water in the emulsions evaporated completely during the 24 hour test period, and the steel specimens again became coated with the crystals. The procedure finally adopted was to add 10% of distilled water at the end of every 8 hours to replace the evaporated water and to maintain the salt water concentration very approximately the same as that of sea water.

111. Using the above procedure it was generally found that the presence of the salt made it necessary to increase the quantity of rust inhibitor added to the B-O-100 fluids to secure adequate protection. The results are given in Table XIX. In most cases two or three times the concentration used for inhibition against corrosion by distilled water was needed with sea water. Cold storage tests on these fluids containing the increased amounts of corrosion inhibitor were run, and as indicated in Table XIX no precipitation occurred in a number of cases after two months of storage at -40°F.

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Conclusions

112. Turbine corrosion tests on the B-O-100 fluids containing the anti-oxidant revealed that a rust inhibitor was necessary for use in any system likely to exposure to water of condensation, spray, or rain. A number of effective rust inhibitors have been found. These are effective in weight concentrations of from 0.5 to 1%. When sea water is likely to be present the concentration of inhibitor should be two or three times greater. A number of these inhibitors will not increase the rate of oxidative breakdown or the emulsion stability of the lubricant.

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(j) Foaming and Defoaming of the B-O-100 Fluids by Harrison Shull and William A. Zisman.

113. The various inhibited B-O-100 fluids have been tested in a preliminary series of experiments to determine whether any undesirable foaming tendencies are present. Experiments have also been conducted in order to discover an effective defoamer for these fluids.

114. The foaming apparatus used consisted of a pyrex column 12 inches tall, at the bottom of which was a fritted Corning glass filter of medium porosity, 3 centimeters in diameter. An air stream was passed at the rate of 166 ml. per minute through the fritted glass into the oil as very fine bubbles. The air was previously passed through a cotton filter and a Fischer and Porter rotameter which served to indicate accurately the flow-rate. McBain and his collaborators (NACA report under contract NAw-1734 of June 1943) have shown that the results obtained by using several of the well-known procedures in foaming tests can be related with each other in a satisfactory manner. The foaming procedure used was found to give reproducible results of the comparative foaming tendencies of the various oils.

115. The standard apparatus as prescribed by the Coordinating Lubricants Research Committee of the Coordinating Research Council (November 1943) is too large for exploratory work on synthetics due to the large samples required. It is intended to correlate these small-scale tests with the CLR method in the near future.

116. Because various tubes differing slightly in diameter (and perhaps in porosity of the fritted disc) were used, different runs were correlated through the use of a single batch of Navy symbol 2135 non-additive petroleum oil made by the Texas Oil Company. The height of the foam was plotted against the time elapsed after the air flow cut for all runs made in a single tube. The intercept and slope relative to that of the 2135 oil in the same tube were determined, and these relative figures were used in obtaining the final results. In Plate 19 are given the graphs comparing the foaming tendencies of these oils and several widely used petroleum oils.

117. There are two factors of interest in the foaming of any oil. One is the equilibrium amount of foam formed, and the other is the rate at which foam disappears after it is no longer being formed. These two factors may be expressed in a single "Foam Index" by dividing the intercept by the



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slope of an oil relative to that of 2135 oil and multiplying by 100 for convenience. The index thus formed expresses the relative foaming tendencies of various oils in a single figure. An oil that does not foam at all has an index of 0. Navy symbol 2135 oil is arbitrarily given an index of 100, and those oils having greater foaming tendencies than this have indices higher than 100. The indices of the oils of interest in this report are:

Navy symbol 2135 oil	100
Inhibited 400B-0-100	176
Inhibited 250B-0-100	64
Inhibited 135B-0-100	34
Esso hydraulic oil as per spec. AN-VV0-366a	21
Uninhibited 250B-0-100	75

The graphs in Plate 19 for the three B-0-100 fluids indicate very clearly the effect of viscosity on the rate of decrease of foam height. Evidently the relatively longer duration of foam life in the more viscous oils is due to the slower drainage of oil from the foam. In this connection it should be noticed that AN-VV0-366a is a relatively light oil and that 2135 at room temperature lies between 250B-0-100 and 400B-0-100 in viscosity. That the foaming of these fluids is not caused by the inhibitor present is shown by the general agreement between the foaming indices of the inhibited and uninhibited 250B-0-100 fluid.

118. Because this amount of foaming in the case of the B-0-100 fluids may be found to be excessive in certain applications, small amounts of silicone polymer fluids were tried as anti-foaming additives. It was found that very small quantities of selected silicone polymer fluids were fully as effective as anti-foam additives in the B-0-100 fluids as they were in the high-boiling petroleum oils (see NRL confidential report No. P-2227). A solution of 0.074% by weight Dow Corning silicone fluid 230-3-208-2 (12,700 cs. at77°F) in 135B-0-100 inhibited fluid decreased the foam to a maximum of 0.4 units. On the above foam index scale this solution had a rating of 1.6. The foam index for the 250B-0-100 inhibited fluid plus 0.076% of the same silicone was 2.4. With 0.024% added to the 400B-0-100 inhibited fluid no foam was observed, the foaming index being 0. General Electric silicone polymer fluid 0-38 (131 cs. at 77°F) in 0.014% solution in 400B-0-100 inhibited fluid resulted in a foam index of 2.6. It is therefore apparent that the silicone polymer fluids are very effective anti-foaming additives for the B-0-100 fluids. The solutions mentioned above were cloudy, showing that the silicone fluids were partially insoluble in the concentrations used.

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Conclusions

119. Foaming tests on the inhibited B-0-100 polymer fluids revealed them to foam in amounts comparable to well-known petroleum oils, depending upon the viscosity of the fluid. The higher the viscosity the more slowly the foam broke. Because this amount of foaming is undesirable in certain applications, the use of defoamers may become desirable. Silicone polymer additives were found highly effective in concentrations as low as 0.01%. The investigation is being continued.



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(k) Effect of Exposure to Ultraviolet Light by Helen F. Fobes and Charles M. Murphy

120. The stabilities of the B-O-100 fluids to ultraviolet light were examined in order to obtain an accelerated test of the relative stabilities of these fluids and naval specification petroleum oils to sunlight.

121. A 15 ml. sample of each fluid in contact with air at room temperature was exposed to the rays of a Hanovia S type Analytic Model Lamp using an "Alpine Sun Burner Luxor Type" high pressure, quartz mercury arc lamp. The fluids were contained in stoppered, clean, dry, clear quartz test tubes which were 15 ml. outside diameter and 115 ml. long. The center of the lamp was at a distance of 18 inches from the test samples and the axis of the lamp was at right angles to the long axis of the test tubes. The lamp was used with a metal reflector but without a filter. Therefore, the radiation used extended from 5460 Angstroms down to approximately 1849 Angstroms. The energy distribution as given by the manufacturer is shown in Plate 20. It will be noted that the relative intensities of the wave lengths dropped rapidly below wave lengths of 2537 Angstroms.

122. The viscosities of the B-O-100 fluids were determined at intervals during the test period of 240 hours and the results are presented in Table XX. The uninhibited fluids showed an initial rapid increase in viscosity followed by a slower and more uniform rate of increase. The changes in the viscosities as the result of 240 hours of exposure were 7.36%, 9.32%, and 15.55% respectively for the 135, 250, and 400B-O-100 fluids. Thus it appears that the effect of ultraviolet light on the viscosity of these fluids is greater the more viscous the fluid.

123. Although the color of the samples did not change because of the exposure to the ultraviolet light, an odor developed, indicative presumably of oxidation, while the neutralization number increased considerably. The neutralization numbers found at the end of the test were 0.87 and 0.57 respectively for the uninhibited 135 and the 400B-O-100 fluids. These fluids had neutralization numbers of 0.40 and 0.17 respectively at the commencement of the test.

124. In marked contrast the viscosity of the inhibited fluids changed but slightly on exposure to the same condition of illumination by ultraviolet light. The increases found were 1.69%, 1.04%, and 0.88% respectively for the 135, 250

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and 400B-0-100 fluids. Changes of that magnitude would be considered practically insignificant and are of the same order as petroleum oils. All of the inhibited B-0-100 fluids darkened in color on exposure to ultraviolet light. Whereas the 135B-0-100 fluid became quite dark the 250 and 400B-0-100 fluids showed less pronounced color changes. The inhibited fluids also changed somewhat less in acidity than the non-inhibited fluids.

125. In Table XX will be found comparisons between the effects of such ultraviolet exposure tests on the viscosities and neutralization numbers of the B-0-100 fluids and four naval specification petroleum oils of approximately comparable viscosities. It will be noted that the changes in the viscosities were of the same order of the same order of magnitude, but the neutralization numbers of the B-0-100 fluids increased much more.



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Conclusions

126. The effect of ultraviolet light on the viscosities of inhibited B-0-100 fluids in contact with air is greater than on naval specification petroleum oils of comparable viscosities. The neutralization number rises more rapidly for the former than for the latter. In addition, exposure to ultraviolet light causes a darkening in the color of the inhibited B-0-100 fluids. The presence of the anti-oxidant causes a marked improvement in the ultraviolet light stability of the B-0-100 fluids. It is considered that, except for extended storage under conditions of exposure to direct sunlight, the B-0-100 fluids are adequately stable to light. Like petroleum oils, it is advisable to keep them stored in the dark where possible.



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(1) Refractometric Analysis for Anti-oxidant by Herbert A. Pohl

127. It is often of value to have available a simple method for the evaluation of the amount of anti-oxidant in a sample of a B-O-100 fluid to aid in the specification or analysis.

128. The method is as follows: Two small samples of the fluid to be examined are percolated, one through dry silica gel and the other through a double bed of dry silica gel and activated charcoal. The refractive index of each percolate is measured, and from the difference of the refractive indices of the two percolates, Δn_D, the percentage of anti-oxidant present may be calculated.

129. In checking the suitability of this method the refractive indices for the sodium "D line" were measured with an Abbé-type refractometer thermostatted at 25° ± 0.05°C. The adsorption columns used were only 3 cm. high and 4 mm. in diameter. The lower end of the adsorption tube was constricted and contained a glass wool plug. The adsorbents were poured in and packed by tapping the entire tube many times. One ml. of the fluid to be percolated was then forced through the column under 10 pounds per square inch of nitrogen (or air) pressure.

130. The refractive index of the first drop or two to penetrate was measured and Δn_D determined. A number of adsorbents were tried including silica gel, Brockmann's alumina, an activated charcoal prepared for the Chemical Warfare Service of over 10 mesh, and Norite charcoal. Silica gel, Norite, and the charcoal proved usable in the analysis.

131. Using Brockmann's alumina it was found that solutions of the anti-oxidant in B-O-100 fluid came through the percolation column richer in anti-oxidant than before percolation. This means that the solvent was preferentially adsorbed.

132. The graphs of n_D against the concentration of anti-oxidant as changed by direct addition, or the removal of anti-oxidant by percolation, are given for the 135, 250, and 400B-O-100 fluids in Plate 21. As they are parallel straight lines, the results for percent anti-oxidant can be expressed as the simple equation derived from the slope of the graphs of Plate 21: Percent anti-oxidant = 420 x n_D. This relation is valid for the concentration range 0 to 5% and is correct to ± 0.1% anti-oxidant. The validity for high concentrations was not investigated since two percent has been found to be an adequate amount of anti-oxidant.

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133. The indices of refraction of the various B-O-100 fluids are given in Table XXI.

Conclusions

134. The determination of the amount of the anti-oxidant in B-O-100 fluids has been based on the change of the refractive index of the fluid after differential percolation through small adsorption columns of silica gel and activated charcoal. This method is simple and requires only a small sample. This method is believed limited in usefulness to the unoxidized, uncontaminated B-O-100 fluids containing only one additive, the Carbide & Carbon anti-oxidant. Its application to the analysis of used B-O-100 fluids is not recommended without an extensive investigation. The refractive indices of the B-O-100 fluids received were found to be comparable in magnitude with the values for petroleum fluids of similar viscosities.

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(m) Solvent Power, Hydrolysis, and Hydroxyl Value by Kenneth L. Temple and John K. Wolfe

135. Several synthetic oils under investigation by this Laboratory have been found to increase in acidity when heated in the presence of water. Such an increase in acid strength, if continued over a long period, will produce substances corrosive to metal systems. For example, fluids prepared from chlorinated organic substances have been particularly troublesome in this regard due to the formation of HCl making it necessary to place certain requirements as regards hydrolysis on liquids proposed for use as non-inflammable fluids.

136. From known facts concerning the chemical constitution of the B-O-100 fluids it was not expected that corrosive acids would be formed from them in the presence of water at the usual operating temperatures. However, it was considered advisable to check this possibility.

137. Hydrolysis tests on the B-O-100 fluids were made by heating under reflux equal quantities of B-O-100 fluids with distilled water for a period of hours. The mixture was allowed to cool and separate into two layers. The aqueous layer was tested for the presence of acids. In preliminary titrations of this aqueous layer it was found that two drops of the alkali solution were enough to change the indicator to the alkaline color. Therefore, this lack of sensitivity made it advisable to use the pH meter to measure the acidity.

138. In Table XXII are the values of pH obtained in several tests for hydrolytic acids from 250B-O-100 fluids. Uninhibited 250B-O-100 fluid, when mixed with freshly distilled water without heating and allowed to separate, was shown to have present no acids since the pH of the water was between 6.0 and 7.0. Heating this mixture under reflux for 24, 46, and 100 hours lowered the pH gradually to 2.7. Since the hydrolysis reaction may have been complicated by a simultaneous oxidation reaction occurring in atmospheric air, the mixture was heated in an atmosphere of nitrogen. The pH of the water separated after heating in this experiment did not differ from that previously observed during the experiment carried out in air. Under the conditions of this experiment, the oxidation reaction did not produce serious amounts of corrosive acids, probably because of the blanketing effect of the water vapor during the hydrolysis test. The hydrolysis tests were therefore not complicated by a simultaneous oxidation reaction.

139. Inhibited 250B-O-100 fluid was tested in a similar manner and when extracted with a little water initially showed



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acids present since the pH was lowered to 4.2. This behavior was in contrast to that of the uninhibited fluid which showed no lowering of the pH of extracted water. Refluxing the mixture of the inhibited material and an equal volume of water lowered the pH of the water to only 3.5 over a period of 100 hours heating. On the basis of these tests it is concluded that no large amounts of corrosive acids are produced in these inhibited fluids by the hydrolysis reaction at 210°F.

140. Hydrolysis tests were run on the inhibited fluid in the presence of synthetic sea water, and the pH of the water at the end of 100 hours was nearly the same as that previously observed in the presence of distilled water. Therefore, sea water did not cause more hydrolysis than did distilled water. It is possible that this result is due in part to a buffering action of the salts in sea water.

141. Hydroxyl determinations were made on the uninhibited B-0-100 fluids by both the acetic anhydride-pyridine and the metallic sodium methods. Results of these determinations are presented in Table XXIII. The 135B-0-100 fluid showed the highest hydroxyl value and the 400B-0-100 the lowest and the 250B-0-100 was intermediate.

142. The acetic anhydride-pyridine method involves mixing a weighed sample of the fluid to be tested with a standard mixture of acetic anhydride-pyridine and allowing acetylation to take place. After esterification has occurred the mixture is poured into a water-ice mixture and the acid produced is titrated in the cold. By difference between this titration and that of a blank the amount of hydroxyl group in the original sample is calculated.

143. The sodium method of determining the proportion of hydroxyl groups present is to mix a weighed sample of the fluid to be tested with anhydrous amyl ether and then add sodium metal. The volume of hydrogen evolved is measured and from it the percent hydroxyl in the original sample is calculated.

144. The agreement obtained between the two methods is shown in Table XXIII. Values by the sodium method are slightly higher than those observed by the acetic anhydride method. Water, if present in these fluids, would cause high hydroxyl values to be observed. To check this possibility, a sample of B-0-100 fluid was treated with anhydrous barium oxide to remove completely the dissolved water. This anhydrous sample was found to have only a very slightly lower hydroxyl value.

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145. Inhibited B-O-100 fluids were not used for hydroxyl determinations since the inhibitor was found to increase the hydroxyl value observed on the uninhibited material.

146. The B-O-100 fluids have been found to exert a powerful solvent action on certain painted and coated surfaces. From their chemical nature this action is to be expected. The paint coating on the inner part of the test engines was removed by the B-O-100 fluids during the engine test runs. This solvent action was also observed outside the engines when the fluids were spilled onto painted surfaces in the Laboratory. This solvent action is even more troublesome with oxidized mixtures of the B-O-100 fluids, having high neutralization numbers.



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Conclusions

147. The inhibited and uninhibited B-O-100 fluids have been found to give only a small amount of acidic substance on hydrolysis with water at 212°F. The observed hydrolysis occurs principally during the first 24 hours and thereafter decreases gradually approaching zero. The small amount of acidic material formed by hydrolysis will usually not be an important factor in the use of these fluids. Hydroxyl values determined on the B-O-100 fluids indicate that these liquids are polar substances increasing in viscosity with an increase in molecular weight. The B-O-100 fluids exert a powerful solvent action, and therefore systems in which use is contemplated should be thoroughly examined for the presence of painted or coated surfaces which could be dissolved or softened on contact with the fluids.

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(n) Neutralization Number and Corrosion by Hayward R. Baker, Charles M. Murphy, and William A. Zisman

148. The neutralization number (the number of milligrams of potassium hydroxide required to neutralize 1 gram of oil) was originally used to determine if an acid-treated petroleum oil had been washed free of the highly corrosive mineral acids used in the refining process. The significance of the neutralization number as a measure of the corrosiveness of a used oil has been and still is the subject of controversy. Inasmuch as high neutralization numbers were found in the oxidation tests reported in section (d), it is necessary that their significance be discussed.

149. During use a petroleum lubricant or hydraulic fluid is subject to contamination and to various chemical reactions one of which is oxidation to form organic acids. The neutralization number has evolved into a commonly employed measure of the acidity or corrosiveness of a used oil. But the compounds present in such a petroleum oil that may consume the potassium hydroxide in the neutralization of the sample include organic and inorganic acids, phenolic compounds, easily saponifiable esters, and lactones. Also many inhibitors and detergents now used in additive-type oils may react with the alkali. It is evident that the neutralization number cannot in general be a measure of the acidity of a used oil.

150. Even where the presence of organic acids alone leads to observed neutralization numbers, it is difficult to relate such measurements to the corrosiveness of the oil. Organic acids vary considerably in strength even in a homologous series, the first members being the strongest acids and the acid strength decreasing with increasing chain length. Also, as is well-known, the introduction of functional groups and changes in structure of the molecules may cause marked changes in the acid strength. The corrosion and wear caused by organic acids also vary with the nature of the metals present in the system, the softer bearing alloys being particularly susceptible to attack.

151. The type and design of the system notably influence the nature and amount of corrosion observed. The corrosion of metal parts not ordinarily covered or wetted by the oil is often caused by water or by volatile acids. If water or water vapor is present, not only will the volatile acids be dissociated and increase corrosion but also the water in direct contact with the iron or steel will cause rusting.



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152. The concentration of the volatile acids and the corrosion due to their presence can be reduced by a properly designed breathing system or by the use of protective paints or coatings for the portion of the metal surface exposed to vapor phase corrosion. This type of corrosion protection is necessary in steel turbine systems. Usually automotive engines are sufficiently aerated to be free from such attack. There are mechanical systems, however, which for one reason or another must be nearly or completely closed. Examples of these are hydraulic systems, recording instruments, torpedo mechanisms, gyroscopes, clock work mechanisms, compasses, etc. A rate of formation of organic acids which would not cause troublesome corrosion in an automotive engine may cause serious corrosion in such closed or poorly ventilated systems.

153. Organic acids dissolved in the oil can be responsible for the corrosion of immersed metal parts. The mechanism involved appears to be much misunderstood. This has been discussed in NRL Report No. P-1849 of March 1942 and some of the discussion presented therein is appropriately repeated here. The dissolved acid molecules migrate or diffuse to the metal-oil interface, the rate depending upon the viscosity of the oil, the molecular weight, and the geometrical shape or configuration of the molecule as a whole. Thus the low molecular weight acids, such as the short-chain saturated fatty acids, diffuse much more rapidly than do the long-chain. The acid molecules reaching the metal-oil interface adsorb there to form an oriented film such that the hydrocarbon portion of each molecule is exposed to the oil while the acid or polar group is in contact with the metal surface. If the metal is chemically reactive with the acid, a subsequent reaction occurs to form a metallic compound. This surface reaction will progress more rapidly the greater the strength and concentration of the acid and the more reactive the metal involved. If the resulting compound dissolves or if it is washed away by the oil, the reaction of acid and metal can continue until all the acids have reacted. If the concentration of the acid is large and this surface reaction can continue, this reaction will lead to easily observable etching, pitting, and marked loss of weight.

154. Many of the high molecular weight acids can protect metals from serious corrosion and rusting. The mechanism involved is not complex. These acids diffuse through the oil to the metal-oil interface and adsorb there to form an oriented monolayer. The more closely packed the molecules of this film the less readily is the underlying metal surface wetted by either the oil or water dispersed in it. A thin protective coating is formed whose permeability may be considered



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proportional to its thickness (or the chain length in case of aliphatic acids) and to the closeness of packing of the molecules in the monolayer. In other words, the high molecular weight acids attack the metal creating a monolayer which prevents the reaction from continuing beyond the extent corresponding to the formation of a complete monomolecular film covering the exposed metal surface.

155. The practical effectiveness of such high molecular weight acids as rust inhibitors for turbine oils and other specialty oils is well-known to the petroleum industry. The addition of used or oxidized oils to new steam turbine lubricating systems to eliminate or minimize the rusting frequently encountered during the break-in run is an example of the application of this kind of protective action.

156. Oils containing organic acids of high molecular weight can cause troublesome and even serious corrosion of alloy bearing surfaces such as copper-lead, bronze, cadmium-silver, and Babbitt. This will be accelerated under conditions of high temperature. Friction in the rubbing surfaces causes the continuous removal of the adsorbed monomolecular film, and hence the total amount of acid reacted with the metal is greater than that corresponding to the coating of the metal surface with a monomolecular film. This results in etching and pitting of alloy bearings, and it can cause considerable increase in the apparent rate of bearing wear. In addition, as the concentration of the metallic reaction product dissolved or dispersed in the body of the petroleum lubricating oil increases, the rate of the oxidative deterioration of the oil increases due to the well-known accelerating effect of metallic naphthenates on the oxidative reactions.

157. From the above discussion it can be concluded that the neutralization number should not be used as a general criterion of the corrosiveness of a used petroleum oil. A sample of used oil with a high neutralization number should be considered to be potentially corrosive where rubbing surfaces are continually exposed to such oil. It is also important to point out here that the change of neutralization number with time of oxidation is always a valuable means of following the course of the oxidation reaction.

158. These conclusions resulting from past research experience with petroleum oils were found to apply to the B-O-100 fluids also, as indicated in the following experiments. The B-O-100 fluids were first given accelerated corrosion tests when in contact with a variety of metals. The test procedure used was a modification of the F.S.B. method (Federal



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Specification Board Method 530-31, Copper Strip Corrosion at 212°F). The metal specimens were approximately 1-1/2" long, 1/4" wide, and 1/16" thick and were polished with emery paper and degreased with hot benzene. Sealed test tubes containing the strips completely immersed in samples of the fluid were maintained at 212°F in a constant temperature bath. The specimens were examined after 3 hours and again after a total of 24 hours of exposure to test conditions.

159. No corrosion was observed in 24 hours by this test procedure when the 250B-0-100 fluid containing 2% of anti-oxidant was in contact with any one of the following metals: lead, magnesium, silver, tin, cold-rolled steel, Babbitt, bronze, Dow metal H and Dural 17ST. A slight tarnish was found on the copper strip as is usually the case with petroleum oil samples. This behavior under the test conditions was entirely satisfactory.

160. A sample of uninhibited 250B-0-100 fluid which had been oxidized at 175°F for 24 hours using a well aerated system as indicated in section (d) was tested at 212°F by the above procedure using strips of copper, brass, Dural 24ST, cold-rolled steel, and Babbitt. Although the neutralization number had increased from the initial value of 0.1 to a final value of 43, no tarnish or corrosion was observed on the copper, brass, or steel specimens. However, a slight reaction on the surface of the Babbitt metal occurred at room temperature, and further reaction occurred at 212°F. After six hours at 212°F the Dural 24ST was also slightly corroded. At the end of 24 hours at that temperature the strips of Dural 24ST and Babbitt were cleaned and reweighed. The losses in weight found were 0.106 and 0.08 milligrams per square centimeter of surface respectively.

161. In confirmation of these results with the F.S.B. corrosion test, a 72 hour test in the single-cylinder Onan engine with the uninhibited fluid caused the neutralization number to rise to 56. Evidence was found of corrosion of the copper-lead crankshaft bearing and copper alloy parts. (The Onan test engine has been described in NRL Report No. P-1857.)

162. Two samples of the 250B-0-100 fluid containing 2% of the anti-oxidant were studied, one of which had been run for 101 hours in a small Delco one-cylinder four-cycle test engine of 2-1/2 horse power rating. The average sump temperatures during the engine runs on these oils were 140°F and 195°F respectively. No corrosion or discoloration was observed on any of these specimens in the F.S.B. corrosion test using

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copper, brass, bronze, Babbitt, Dural 24ST and cold-rolled steel. After the engine runs the neutralization numbers had become 1.0 and 30.3 respectively; yet no evidences of corrosion were found in the engines. The differences in the final neutralization numbers indicated a more rapid oxidative breakdown in the Delco than in the Onan engine.



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Conclusions

163. The unreliability of the neutralization number as an index of the corrosiveness of an oil has been discussed. The formation of low molecular weight oxidation products (notably organic acids) may cause corrosion in systems containing reactive metals if a condition of inadequate breathing or ventilation exists, or if these products can accumulate in the oil. Where high molecular weight acids (or other high molecular weight oil-soluble oxidation products) are formed, they usually act as corrosion inhibitors. Etching, pitting, or apparent increased rates of wear will be found where rubbing or sliding of the lubricated metals occurs and where the metals involved are able to form adsorbed chemical compounds with the organic acids developed. Tests with the B-O-100 fluids demonstrate that these theoretical conclusions apply to them just as fully as they do to petroleum oils. Thus, whereas the Federal Specification Board corrosion tests show little or no corrosion to copper and brass regardless of the neutralization number of the oxidized or used uninhibited B-O-100 fluid, engine tests showed corrosion of copper-lead bearings and copper-alloy parts. No signs of corrosion were found in the F.S.B. corrosion test or in engine tests when the inhibited B-O-100 fluids were used. Therefore, it is believed the inhibited B-O-100 fluids were used. Therefore, it is believed the inhibited B-O-100 fluids are satisfactory as regards freedom from bearing corrosion in automotive engines or other mechanisms provided with adequate ventilation.

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(o) Results of Four-Ball Machine Tests by Harrison Shull

164. The Carbide & Carbon B-O-100 inhibited fluids were subjected to tests on the Shell Four-Ball Lubricant Testing Machine in order to obtain an evaluation of the load-carrying ability indicated by this machine. The same test method was applied also to several well-known petroleum oils in order that a comparative picture might be obtained.

165. The Shell Four-Ball machine has already been fully described in the literature (National Petroleum News, 31, December 27, 1939; Instit. Mech. Engineers, General Discussion on Lubrication, Vol. II, pp. 274-284). The test chamber consists of three 1/2 inch steel balls tightly clamped together so that they cannot revolve, upon which a fourth steel ball is rotated at about 1700 r.p.m. All four balls are immersed in the test lubricant during the time the machine is in operation. A convenient lever loading device is so arranged that various loads may be applied between the upper and the three lower balls. At the conclusion of the test, there is a circular ring of wear on the upper ball, and a circular wear spot on each of the lower balls. The average wear-spot diameter is taken as the criterion of the amount of wear occurring at each load. The torque indicator gives a measure of the friction occurring between the rotating and non-rotating balls, and helps to detect seizure. The procedure used in these tests was to determine the pressure-wear index (PWI) as developed at the U.S. Naval Engineering Experiment Station at Annapolis, Md.

166. The PWI is calculated from two points, P_1 and P_2, on the graph of average wear spot diameters against applied total load on log vs. log paper. Diameters are measured in millimeters; loads are measured in kilograms. At very low loads, no seizure between the upper and the three lower balls is obtained. The largest load at which no seizure is consistently observed corresponds to the point P_1. At high loads it has been found that a straight-line relationship exists between wear diameters at various loads when plotted on log vs. log graph paper. The lowest load at which the wear diameters fall consistently on this line corresponds to the point P_2.

167. Having determined points P_1 and P_2 by a series of tests at various loads, the PWI may be calculated by the use of the following formula:

$$PWI = \frac{L_2 - L_1}{W_2^2 - W_1^2}$$

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where L_2 and W_2 are the load and average wear diameter respectively at point P_2, and L_1 and W_1 are similar quantities for the point P_1.

168. At a meeting of representatives of laboratories owning Four-Ball machines held on January 11-12, 1944 in Annapolis, the Engineering Experiment Station reported that although points P_1 and P_2 changed slightly from day to day, the PWI was remarkably reproducible over long periods of time. It was also found that the PWI showed a rough correlation with extreme pressure properties of lubricants. For example, a very good extreme-pressure lubricant was found to have a PWI value of 40-45 or higher. Mineral oils in general were found to have PWI's of 0-2. Mild extreme-pressure lubricants usually fell in between these values, often being 15 - 20. The pressure-wear index was reported to be somewhat affected by viscosity (a change from SAE 40 to SAE 50 may affect the PWI by one or two units).

169. In Table XXIV are given the values found by this Laboratory for the following oils of interest in connection with this report:

(1) Carbide & Carbon inhibited 250B-0-100 fluids (PWI measured twice on different days).
(2) Carbide & Carbon inhibited 400B-0-100 fluid.
(3) Carbide & Carbon inhibited 135B-0-100 fluid.
(4) Navy Symbol 2135 non-additive, low V.I. petroleum oil (Socony Vacuum).
(5) Navy Symbol 9250 detergent oil (Esso HD 30).
(6) Navy Symbol 1080 non-additive, high V.I. petroleum oil (Socony Vacuum).

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Conclusions

170. The fact that some extreme pressure lubricants have PWI values of over 40 helps to define the position of the B-0-100 fluids in relation to other well-known types of lubricants. These data indicate that the inhibited B-0-100 fluids can be classified as very mild extreme-pressure lubricants which presumably have better load-carrying characteristics than straight mineral oils. It must be emphasized, however, that, due to the present limited knowledge of the characteristics of this test machine, the PWI is an empirical factor referring only to the behavior of a given lubricant in the Four-Ball machine, and it therefore is impossible to apply this information to other lubricating systems where load, velocity, configuration, and composition of the bearing surfaces are different.



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CONCLUSIONS

171. The three B-O-100 fluids studied had viscosity indices of from 140 to 145 and A.S.T.M. viscosity slopes of from 0.6 to 0.7 and hence were superior as regards viscosity-temperature characteristics to conventionally refined petroleum oils not containing polymer thickening agents. However, they are inferior in these respects to the methyl silicone polymer fluids. The evaporation rates of the inhibited B-O-100 fluids were comparable to those of petroleum oils of the same viscosities. Even after a month of cold storage all three fluids had pour points between -50°and -60°F. No crystals or precipitates were observed even after 24 hours of storage at -80°F. The more favorable viscosity-temperature characteristics and lower pour points indicate that these fluids would be advantageously substituted for conventionally refined petroleum oils where extreme cold weather makes difficult the starting of cold motors. The S.A.E. system of classification based on viscosity cannot be applied to these fluids.

172. The uninhibited B-O-100 fluids were unstable to oxidation even at 150°F. In the presence of adequate air the oxidation process resulted in the splitting of the polymer molecules with the evolution of volatile organic acids and aldehydes, but no precipitates were formed. The addition of two percent of the anti-oxidant as recommended by the manufacturer was found remarkably effective in inhibiting the oxidative breakdown. At a temperature of 250°F and in the presence of any of the metals likely to be present in engines, the inhibited B-O-100 fluids were found adequately stable to oxidation. Copper and bronze acted as catalysts to accelerate the rate of oxidation while cold-rolled steel, chromium, and Dural 17ST did not. Copper naphthenate was found an effective oxidation accelerator as is the case with petroleum oils. Once the anti-oxidant was consumed by oxidation, the fluid deteriorated rapidly with the evolution of volatile acids and aldehydes accompanied by a rapid decrease in the viscosity and a large increase in the neutralization number of the fluid. Due to the importance of the anti-oxidant for safe use of these fluids in valuable equipment, it is important that any oil filters employed be types which are not selective in adsorbing the anti-oxidant.

173. The B-O-100 fluids were found to be definitely less inflammable than petroleum lubricating oils of the same viscosities, and they were very much less inflammable than present Army and Navy specification polymer-thickened "flat-viscosity" hydraulic oils. This was indicated by the high percentage of oxygen necessary in the atmosphere to allow



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the propagation of a flame in an oil mist or spray. Incendiary firing tests at the Naval Proving Grounds at Dahlgren, Virginia on one gallon samples resulted in the same conclusions.

174. The surface tensions of the B-O-100 fluids were not much different from those encountered with petroleum lubricants. This characteristic and their low volatility indicate that these fluids should not creep or leak more than petroleum oils of corresponding viscosities. The densities of these fluids are very nearly equal to that of water at room temperature, and they increase a few percent as the temperature approaches the freezing point of water. These fluids are therefore considerably more dense than petroleum oils of comparable viscosities. The coefficients of thermal expansion are approximately the same as those of petroleum oils of comparable viscosities.

175. The 135, 250, and 400B-O-100 fluids dissolved from 5% to 3% of water at 32°F and from 2% to 1% at 140°F, the more viscous fluids dissolving the smallest amounts of water. They emulsified readily with water, the emulsions being less stable the higher the temperature and the lower the viscosity of the B-O-100 fluid, this being an example of a general rule of emulsion stability. Oil-in-water emulsions were formed when the ratio of water to oil was large; otherwise, water-in-oil emulsions developed. The water-in-oil emulsions were definitely the more stable. The emulsions formed with water and either new or used oils were not much more viscous than the original fluid.

176. The B-O-100 fluids were found to lack rust inhibiting properties. Due to their ability to dissolve so much water and their good emulsifying properties, a suitable rust inhibitor will be needed. Rust inhibitors have been found many of which were effective even in the presence of sea water. A number of these will not precipitate from oil solution after one month of storage at -40°F nor will they increase the stability of the emulsion with water or the rate of oxidative breakdown.

177. Laboratory foaming tests revealed that the inhibited B-O-100 fluids foam to an extent comparable to some well-known and much used Navy specification petroleum oils, and it is believed this property may be troublesome in some applications. However, it was discovered that the silicone polymer additives when present in concentrations as low as 0.01% were entirely effective and satisfactory for use as defoaming agents.



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178. Ultraviolet exposure tests for stability to sunlight indicated these new fluids were comparable to petroleum fluids in these respects, and like them it is advisable that they be kept in the dark during long-time storage. Without the anti-oxidant the fluids are less stable to sunlight than petroleum oils. A useful refractometric method has been developed of analyzing the unoxidized fluids for the anti-oxidant content.

179. The B-O-100 fluids were found to be adequately stable to hydrolysis even at 212°F. Hydroxyl determinations demonstrated these fluids to be polar substances increasing in viscosity with the average molecular weight. These fluids are powerful solvents and therefore lacquered or painted systems in which they are to be used should be tested previously to be sure they are coated with a material not dissolved or softened by contact with them.

180. As with the petroleum oils the neutralization numbers of the used or oxidized B-O-100 fluids were found to correlate poorly with their corrosion tendencies. The uninhibited fluids caused some corrosion of copper-lead bearings in small-scale test engines. No corrosion of copper or brass occurred when the Federal Specification Board corrosion test was applied to the used oils. The used inhibited fluids did not cause engine bearing corrosion even when the neutralization number was as high as 30. The reasons for this are outlined in terms of the monomolecular adsorption of acidic products to form chemiadsorbed films and the increased rates of removal of such films through bearing friction. It is concluded that the inhibited B-O-100 fluids should be satisfactory lubricants as regards freedom from bearing corrosion where operated in ventilated systems under conditions where the anti-oxidant supply is adequate.

181. Measurements of the Pressure-Wear Index with the Shell Four-Ball machine indicated that the inhibited B-O-100 fluids could be classified as "very mild" E.P. lubricants, presumably with better load-carrying capacities than straight mineral oils.

RECOMMENDATIONS

182. (a) The B-O-100 fluids deserve serious consideration for use as lubricants for internal combustion engines where good low temperature starting properties are essential.

(b) It is also recommended that these fluids be tested in aircraft engines because of their good low temperature

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starting properties and also their unique oxidation breakdown products the greater proportion of which are volatile materials eliminated through the exhaust system.

(c) These fluids are satisfactory for use as hydraulic oils in systems not likely to accumulate more than 5 to 10% of water provided a suitable rust inhibitor is used. However, the change in viscosity with temperature is not sufficiently small to permit their use as "flat viscosity" hydraulic oils in aircraft or naval gun control hydraulic systems.

(d) Due to their excellent resistance to inflammability under incendiary fire these fluids deserve consideration where that property is essential. It is entirely possible that they may be satisfactory for use as non-inflammable fluids for hydraulic systems not requiring a fluid with a smaller change in viscosity with temperature.

(e) The B-O-100 fluids are particularly recommended for lubricants in systems where the ability to dissolve or tolerate from 3 to 6% of water is highly advantageous.

(f) It is recommended that the addition to the B-O-100 fluids of inhibitor be required if these fluids are to be used where water can accumulate and remain in the system for long intervals.

(g) The B-O-100 fluids do not appear to be any more troublesome as regards foaming than many commonly used petroleum oils. If oil foaming problems are encountered the use of a small percentage of one of the higher viscosity Dow Corning or the General Electric methyl silicone polymers as a defoamer is highly recommended.

(h) In connection with recommendations (a) and (b), tests are in progress on the use of these fluids for engine lubricants in small scale 2-1/2 H.P. stationary Onan and Delco engines. As the results are of much interest they will be reported in the near future.

(i) Further laboratory research is in progress on the oxidation stability and products of oxidation of the inhibited B-O-100 fluids in aerated systems at high temperatures and in the presence of a wider variety of metals. Comparisons are being made with suitable naval specification petroleum oils. Oxidation tests in closed systems are also being made to determine if the inhibited fluids are satisfactory for use in completely enclosed mechanisms. High pressure oxidation tests at 212°F and from 100 to 1500 psi

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for 48 hours in the presence of common metals are also being made in order to indicate the suitability of these fluids for use in hydropneumatic systems such as recoil systems.



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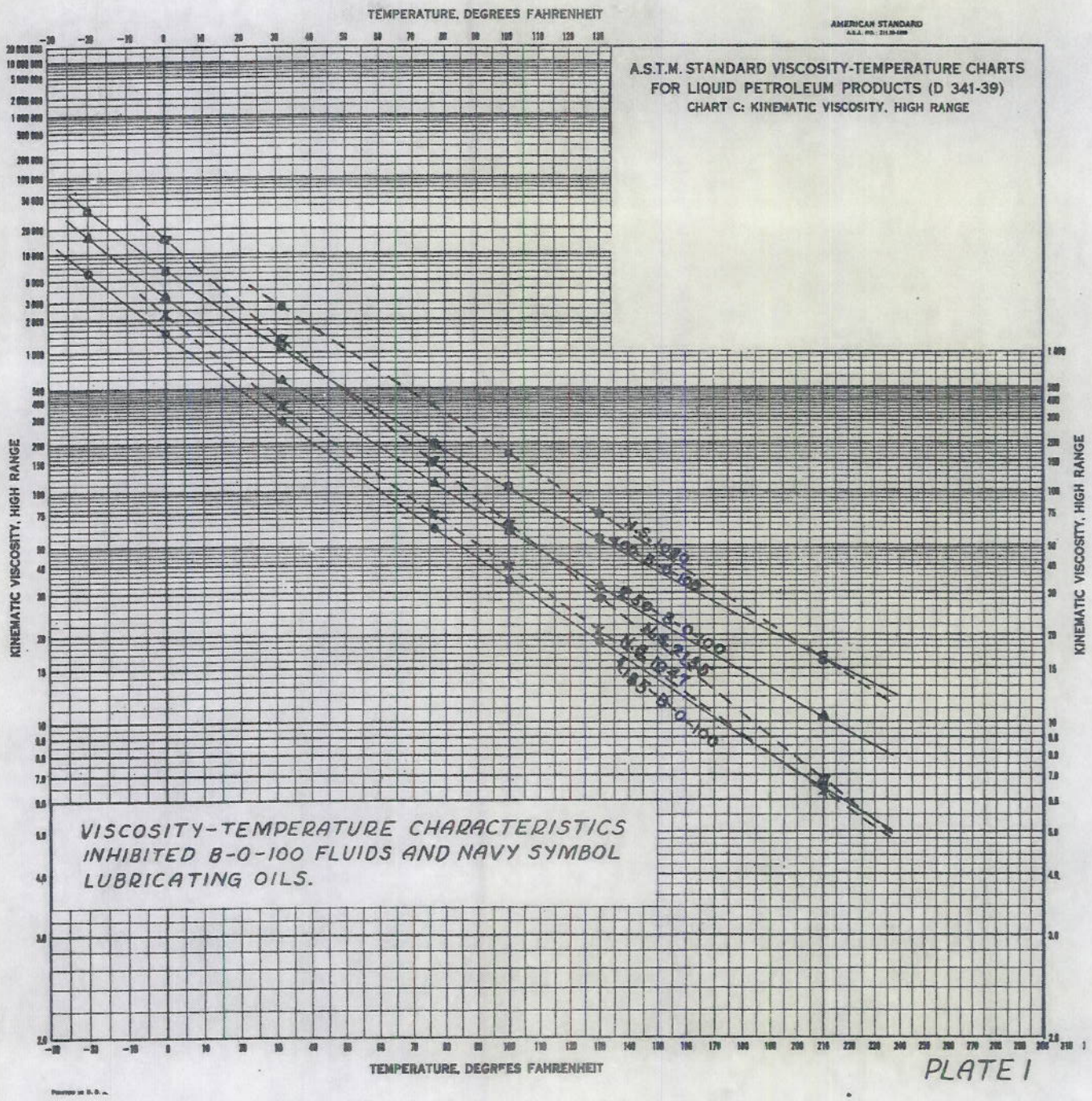
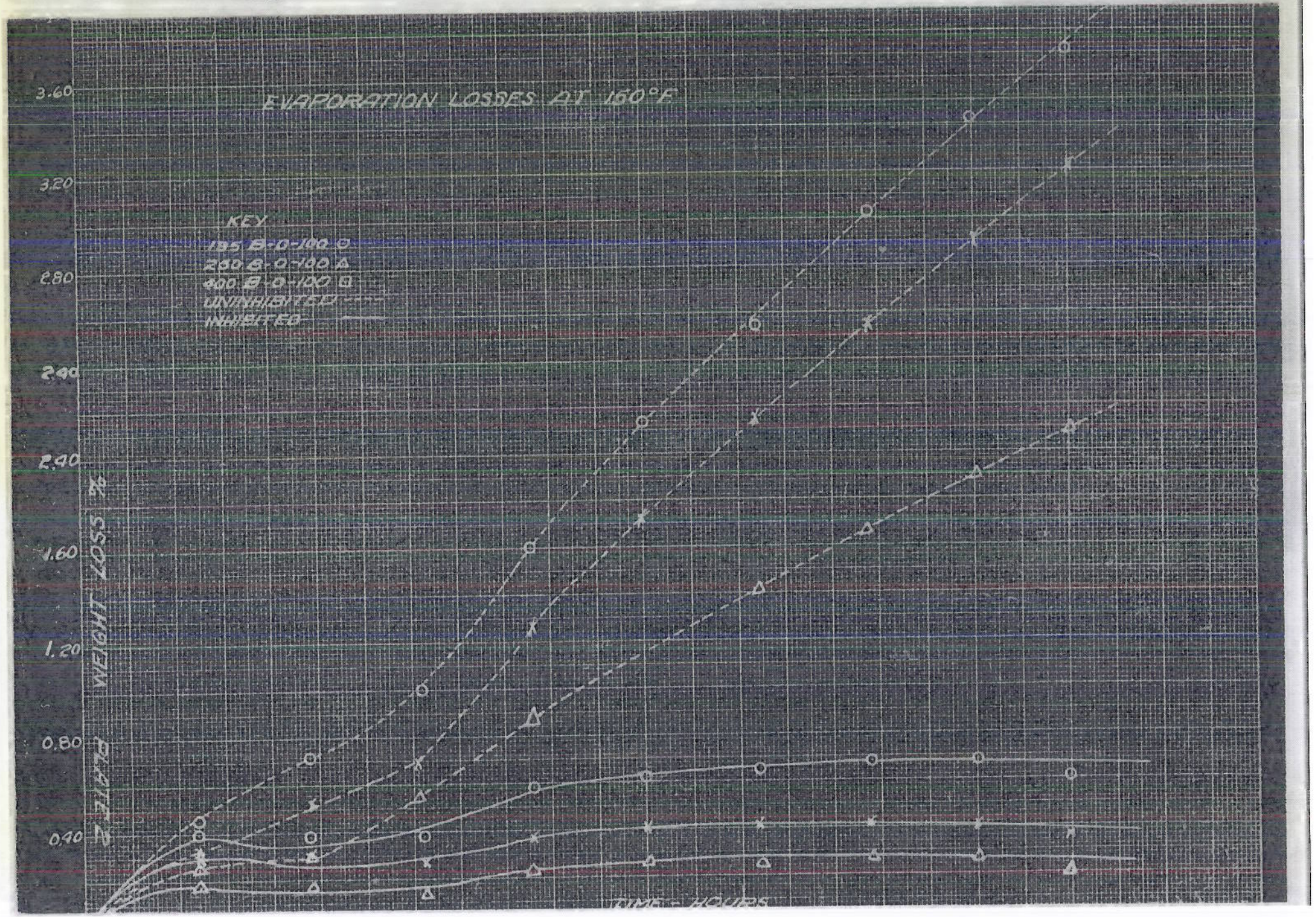
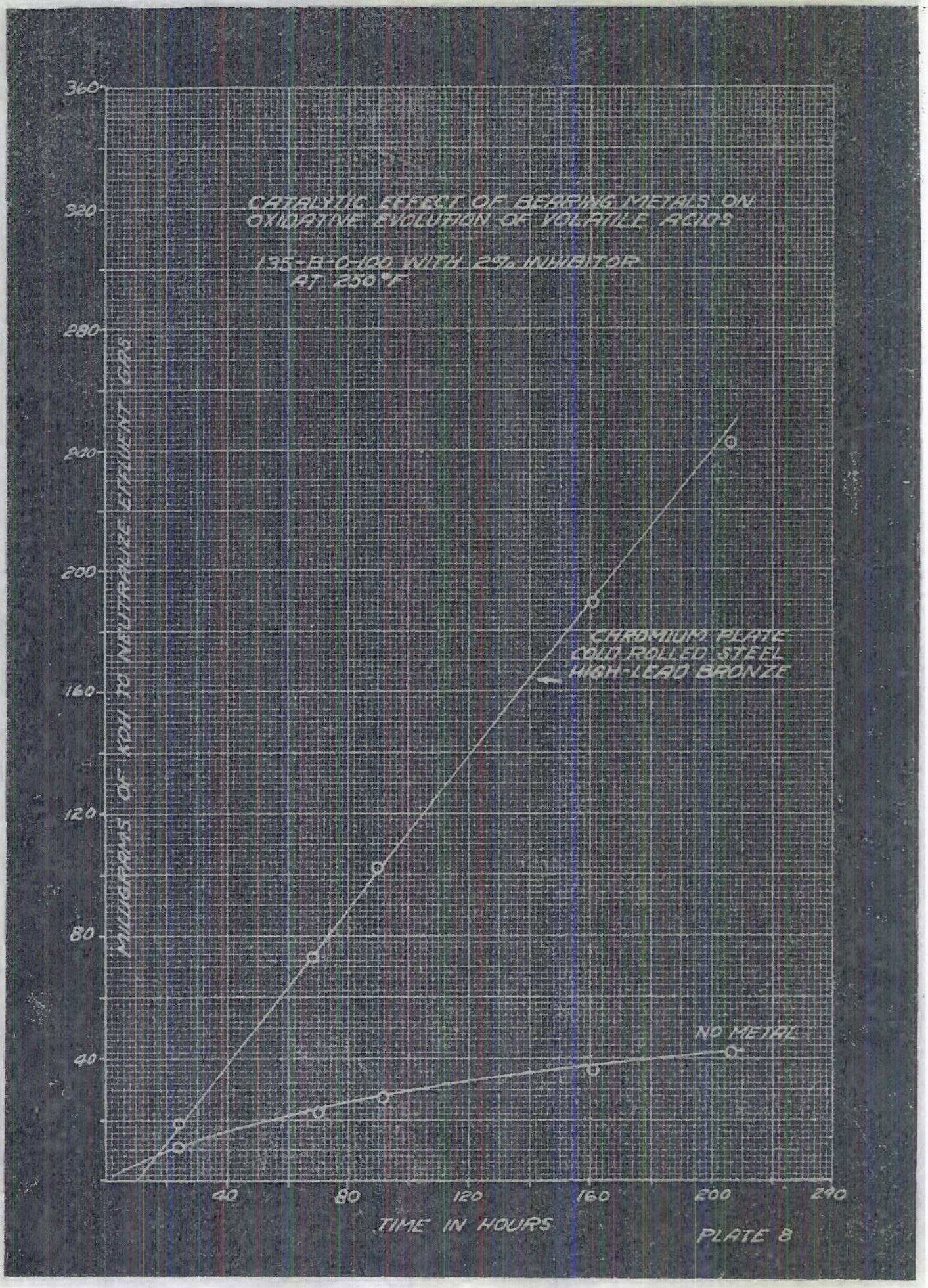


PLATE I

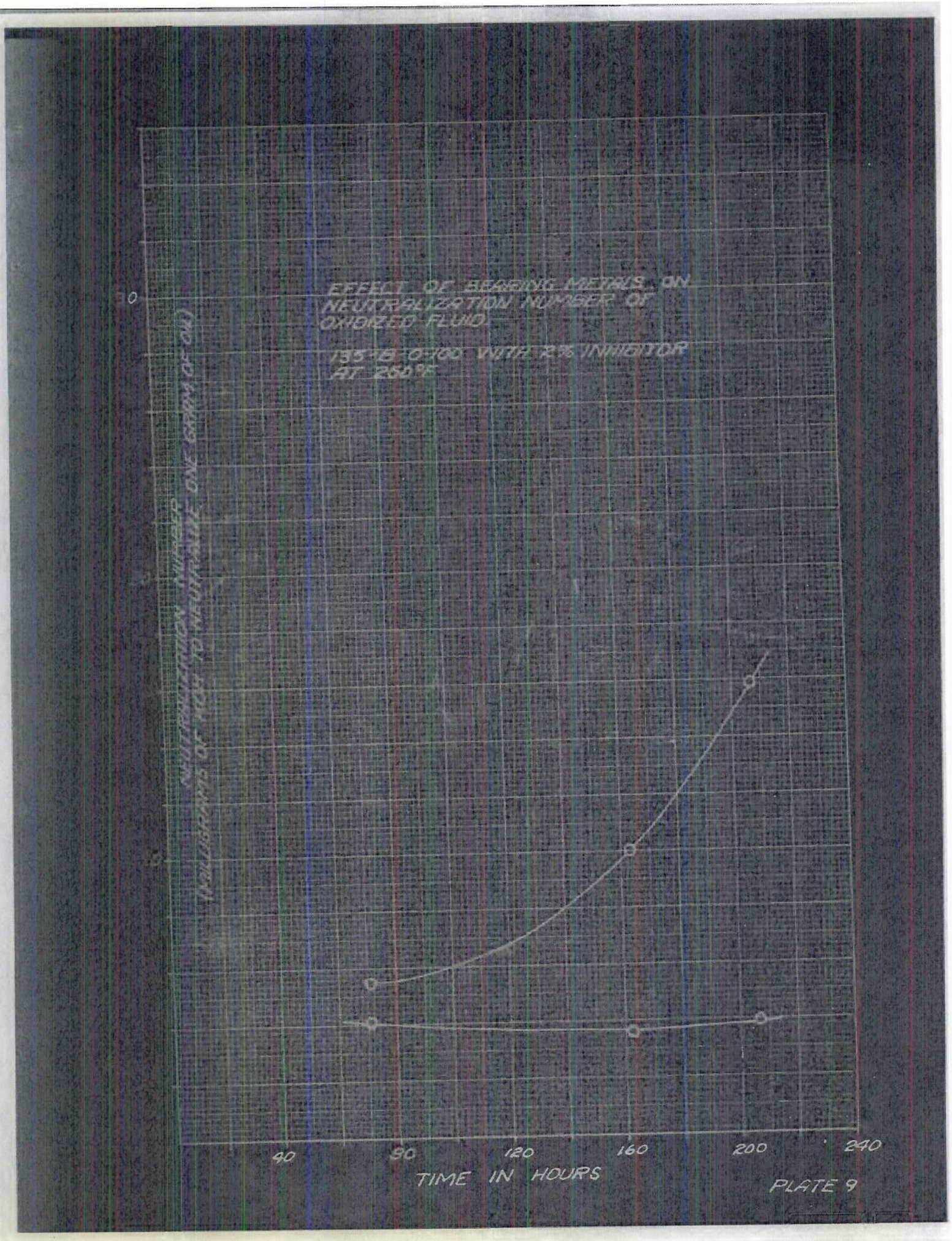
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EFFECT OF BEARING METALS ON NEUTRALIZATION NUMBER OF OXIDIZED FLUID
135-B 10-100 WITH 2% INHIBITOR AT 200°F
NEUTRALIZATION NUMBER OF OXIDIZED FLUID
0
.5
40
80
120
160
200
240
TIME IN HOURS
PLATE 9

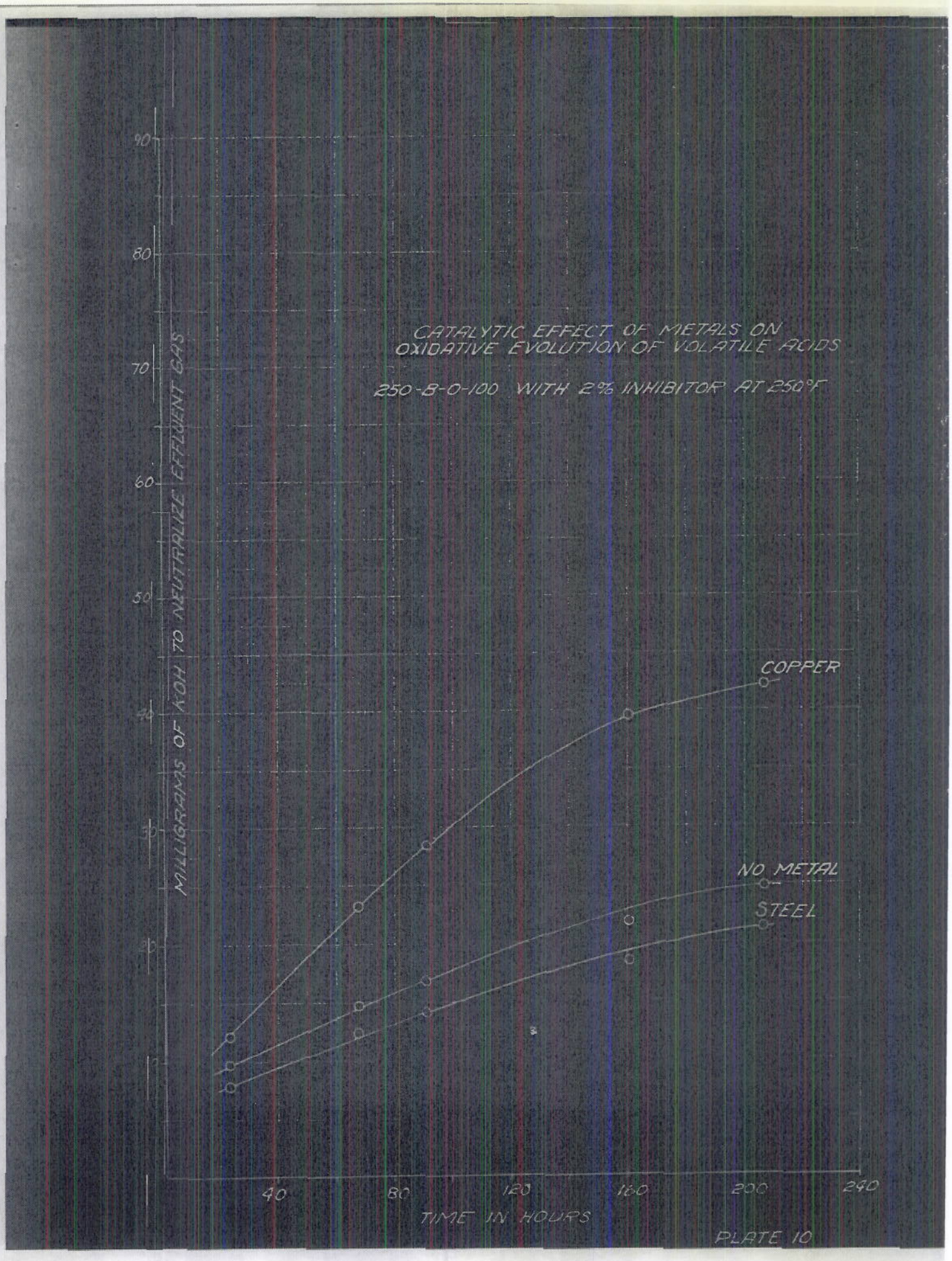
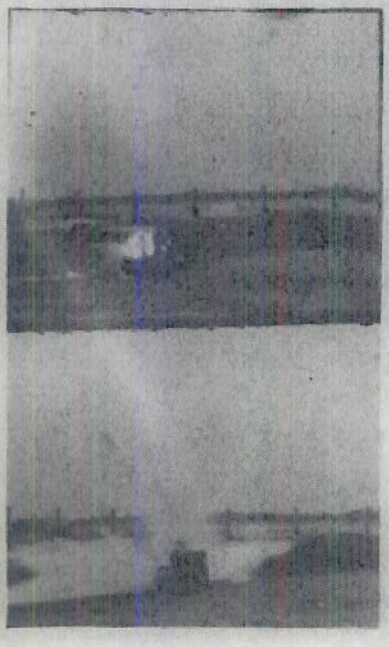
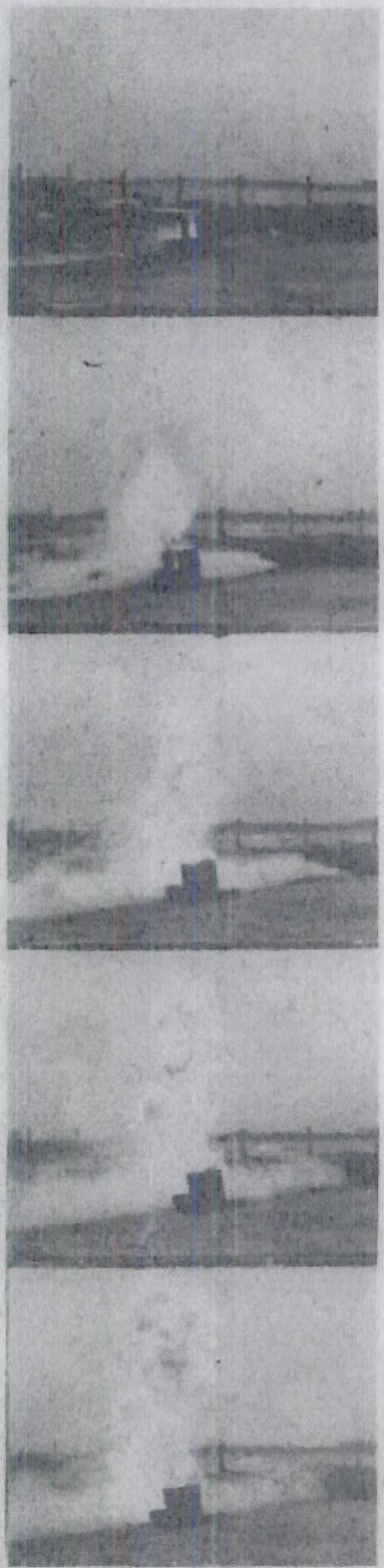

CATALYTIC EFFECT OF METALS ON
OXIDATIVE EVOLUTION OF VOLATILE ACIDS
250-8-0-100 WITH 2% INHIBITOR AT 250°F
MILLIGRAMS OF KOH TO NEUTRALIZE EFFLUENT GAS
90
80
70
60
50
40
30
20
10
COPPER
NO METAL
STEEL
40
80
120
160
200
240
TIME IN HOURS

PLATE 10

WATER

O.S. 2943





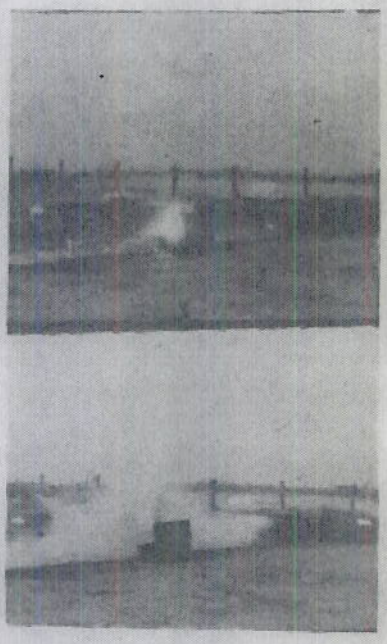
INCENDIARY FIRING TESTS
ON WATER AND OS 2943

[redacted]

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PLATE 11

250 - B-O-100 + 2% ANTIOXIDANT



250 - B - O - 100



INCENDIARY FIRING TESTS
ON 250 - B - O - 100

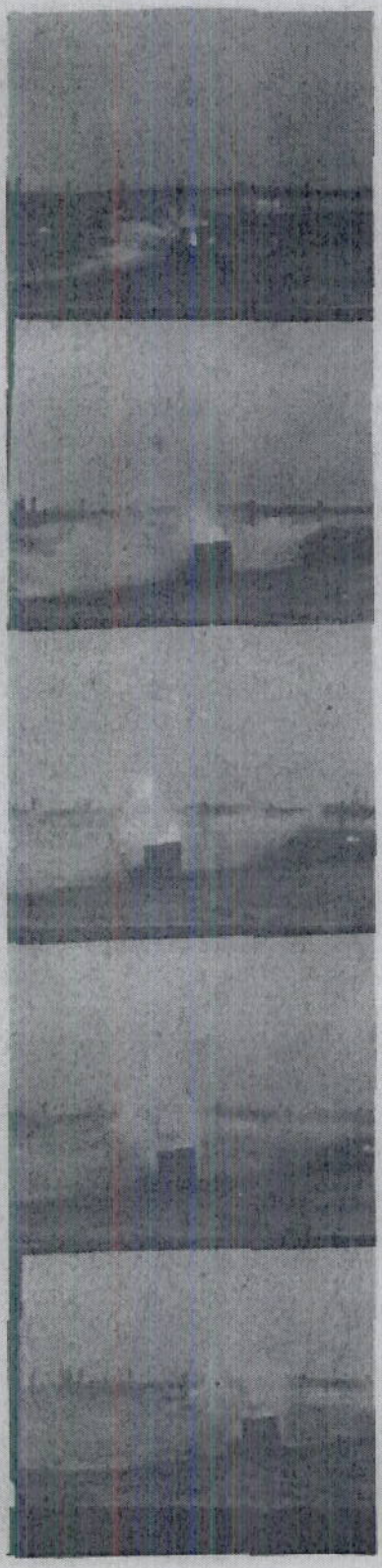
PLATE 12

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135-B-0-100 2% ANTIOXIDANT



400-B-0-100



INCENDIARY FIRING TESTS
ON 135-B-0-100 AND
400-B-0-100

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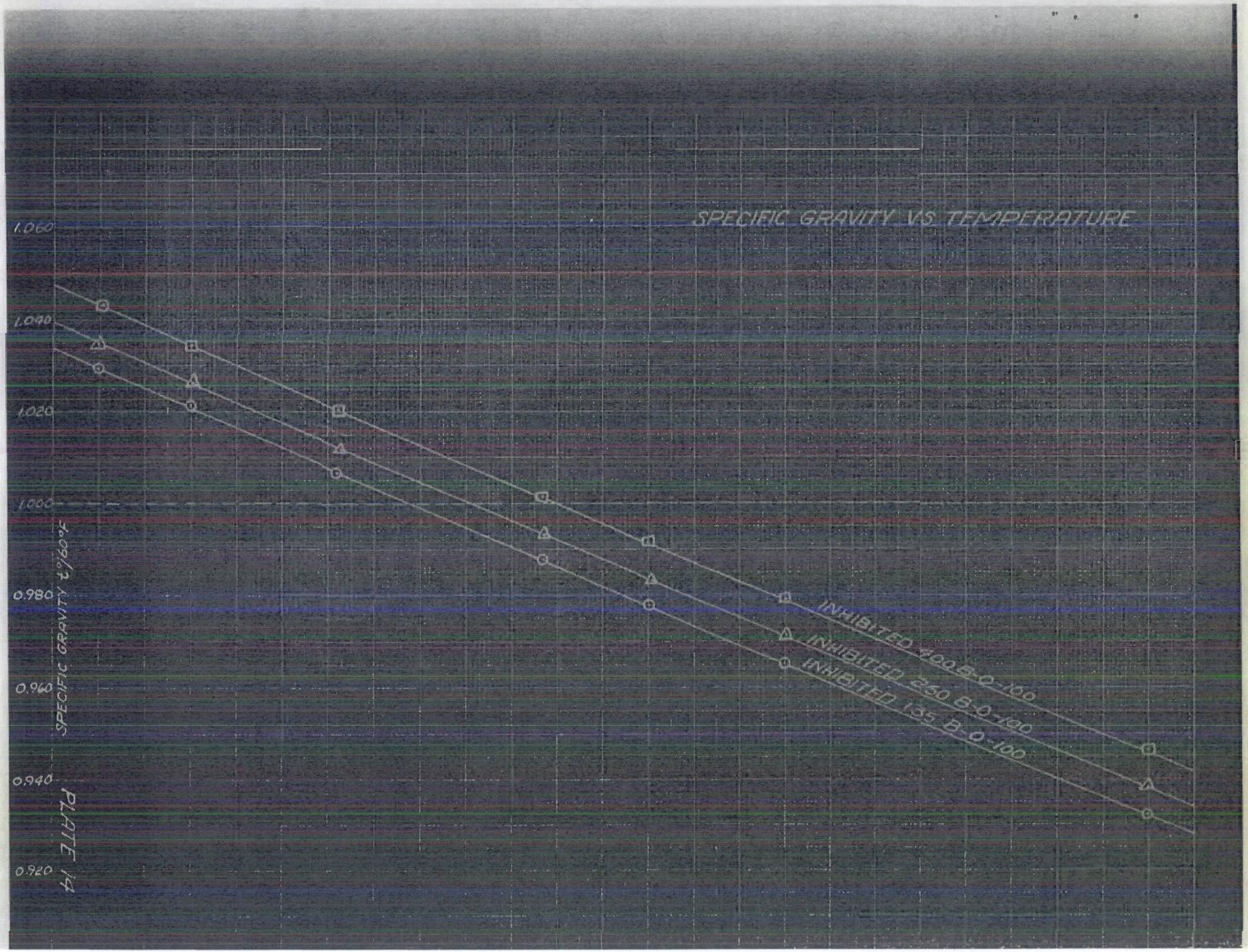
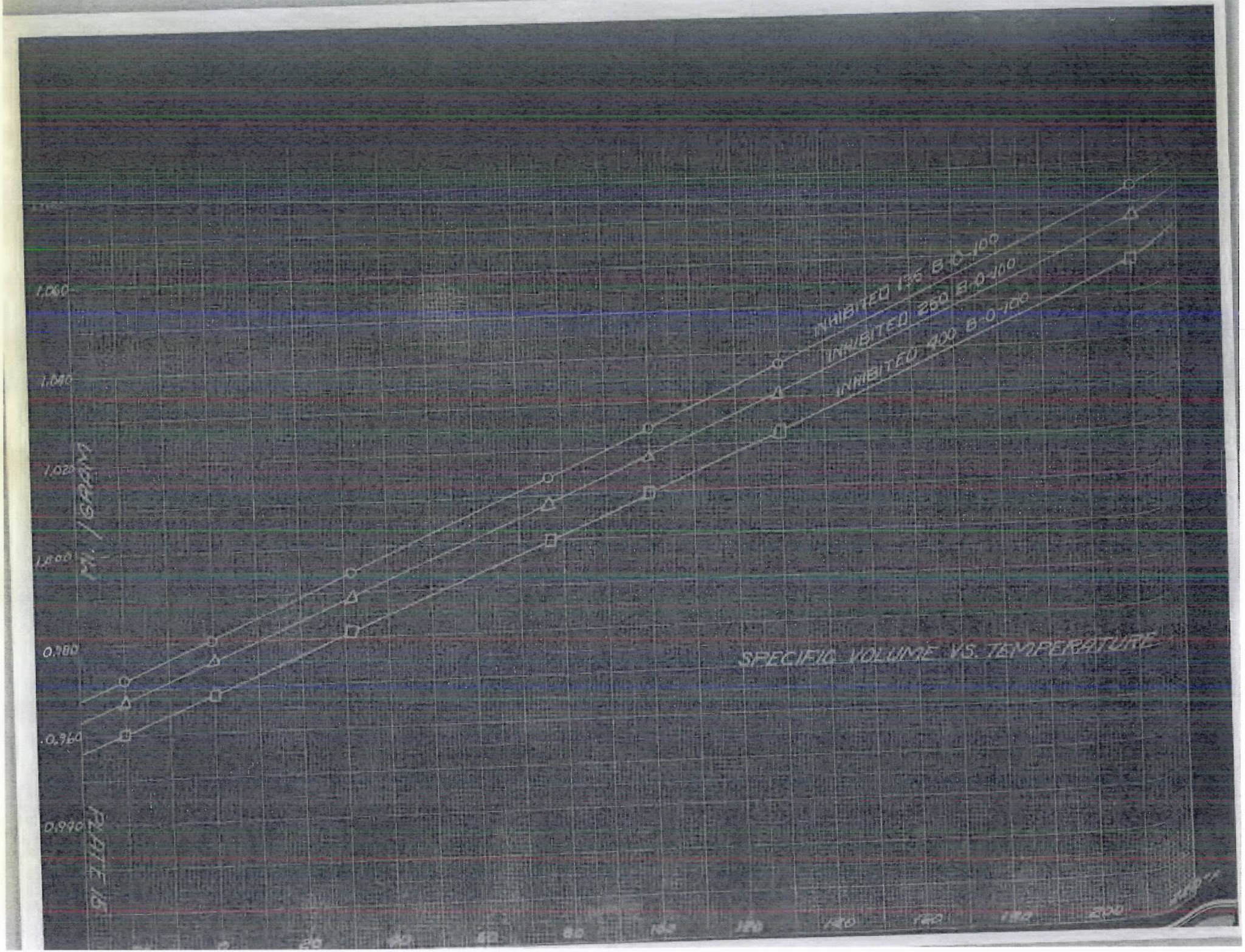
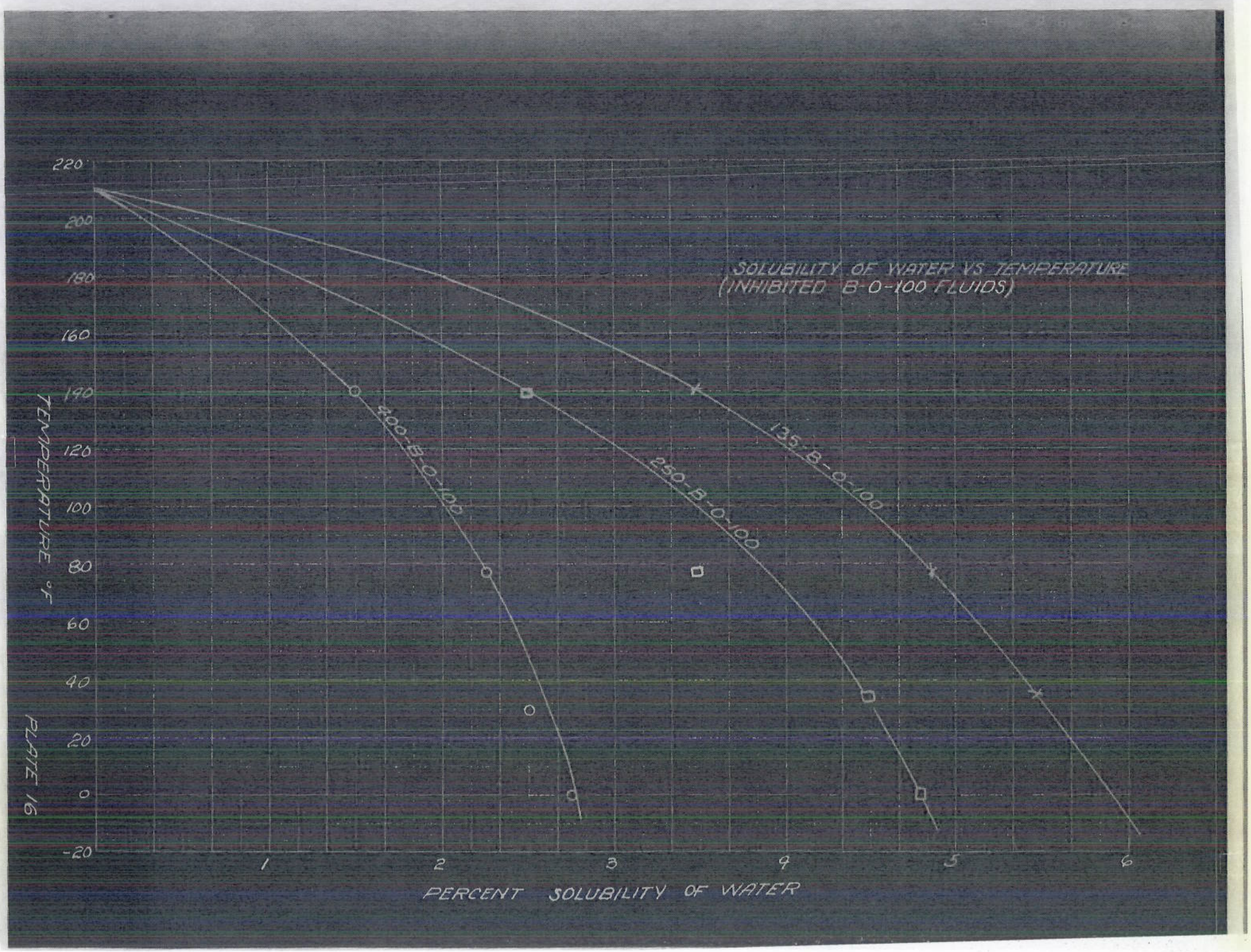

SPECIFIC GRAVITY VS TEMPERATURE
1.060
1.040
1.020
1.000
0.980
0.960
0.940
0.920
SPECIFIC GRAVITY @ 60°F
INHIBITED 400 B-O-100
INHIBITED 250 B-O-100
INHIBITED 135 B-O-100

PLATE 14


SPECIFIC VOLUME VS. TEMPERATURE
INHIBITED 145 B-0-100
INHIBITED 250 B-0-100
INHIBITED 400 B-0-100
1.060
1.040
1.020
1.000
0.980
0.960
0.940
cu. ft. / pound
60
80
100
120
140
160
180
200


SOLUBILITY OF WATER VS TEMPERATURE
(INHIBITED B-O-100 FLUIDS)
135-B-O-100
250-B-O-100
400-B-O-100
TEMPERATURE °F
220
200
180
160
140
120
100
80
60
40
20
0
-20
1
2
3
4
5
6
PERCENT SOLUBILITY OF WATER
PLATE 16

MODIFIED TURBINE CORROSION TESTS

In Absence of Additives



I35-B-0-I00
without
oxidation inhibitor



CONTROL RUN
(using Navy symbol
2I35 oil and I0%
distilled water)



I35-B-0-I00
containing 2%
oxidation inhibitor



250-B-0-I00
without
oxidation inhibitor



250-B-0-I00
containing 2%
oxidation inhibitor



400-B-0-I00
without
oxidation inhibitor



400-B-0-I00
containing 2%
oxidation inhibitor

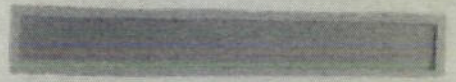
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PLATE 17

MODIFIED TURBINE CORROSION TESTS

In Presence of Additives

I35-B-0-I00	250-B-0-I00	400-B-0-I00
		
no additive	no additive	no additive
		
with 0.5% phenyl stearic acid	with I.0% sorbitan monooleate	with I.0% mixture of n-mono octadecenyl and n-mono octadecendienyl amine
		
with 0.5% naphthenic acid M.W.440	with 0.5% zinc naphthenate	with 0.2% p.n.dodecyloxy benzoic acid

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PLATE 18

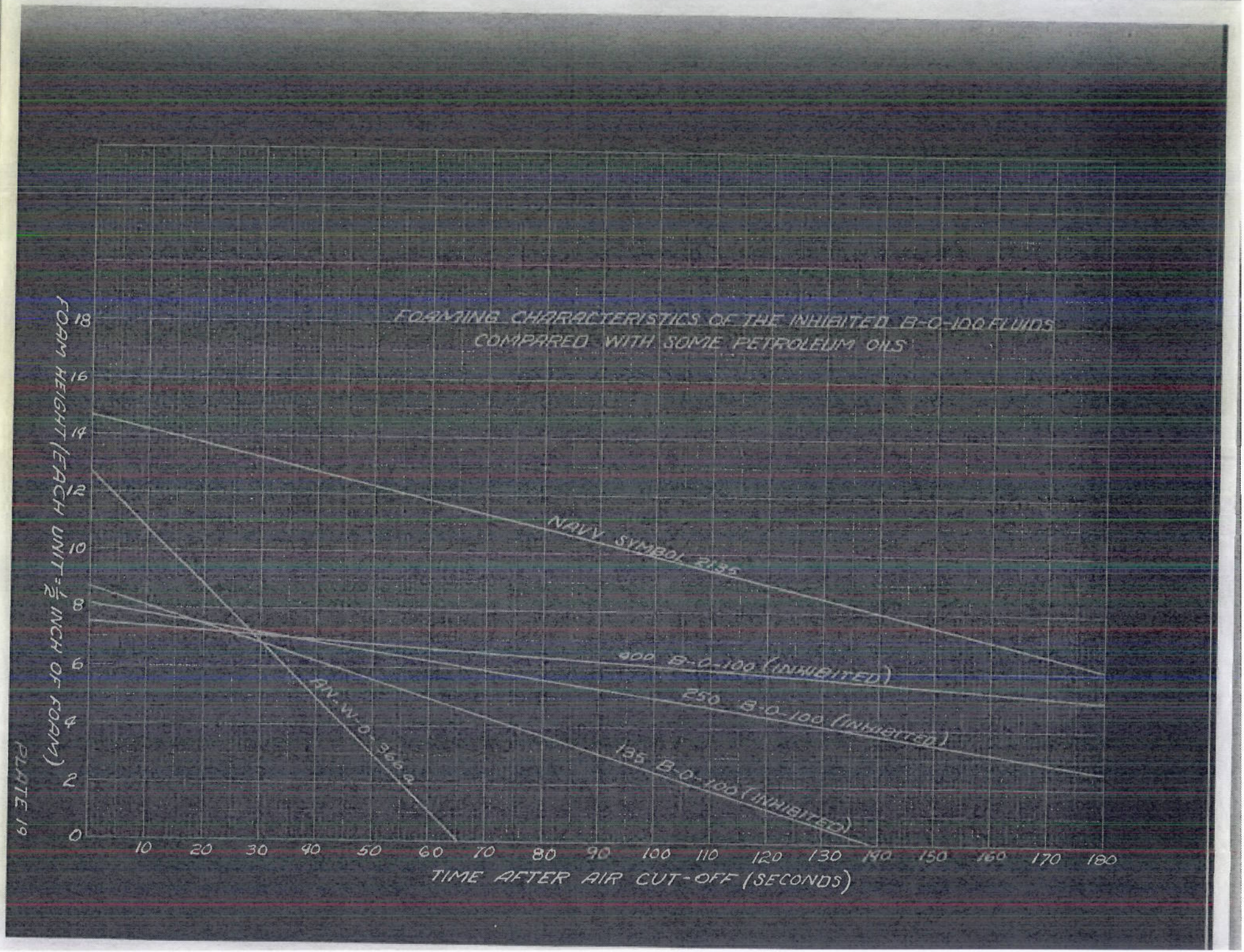
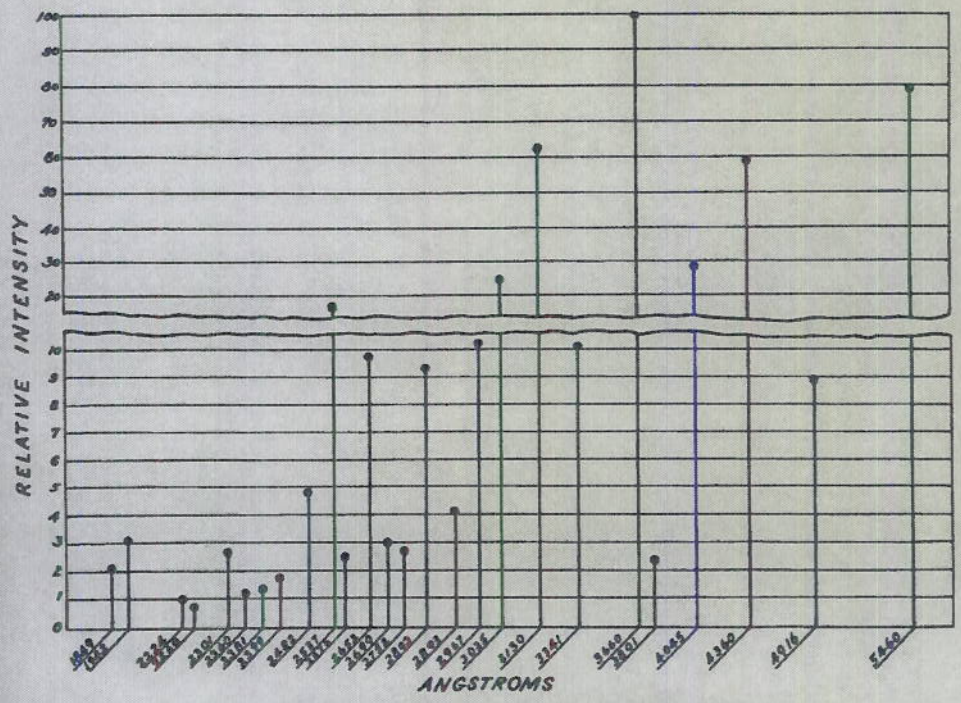

FOAMING CHARACTERISTICS OF THE INHIBITED B-O-100 FLUIDS COMPARED WITH SOME PETROLEUM OILS
FOAM HEIGHT (EACH UNIT = ½ INCH OF FOAM)
TIME AFTER AIR CUT-OFF (SECONDS)
NAVY SYMBOL 2135
300 B-O-100 (INHIBITED)
250 B-O-100 (INHIBITED)
135 B-O-100 (INHIBITED)
AN-W-O-366a
0
2
4
6
8
10
12
14
16
18
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

PLATE 19

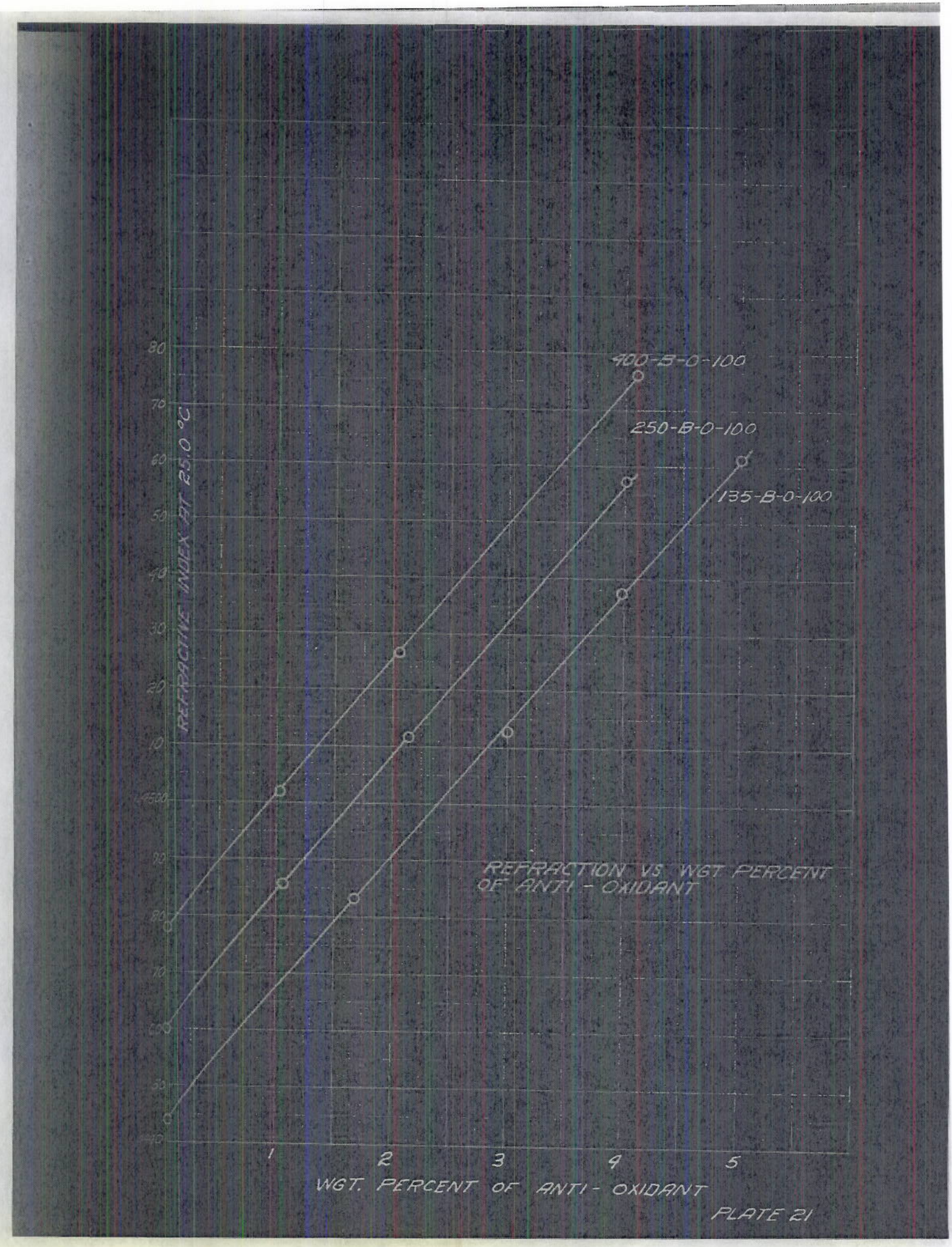
SPECTRAL ENERGY DISTRIBUTION CURVE IN ULTRA VIOLET SOURCE *



* REPRODUCED FROM MANUFACTURERS DATA

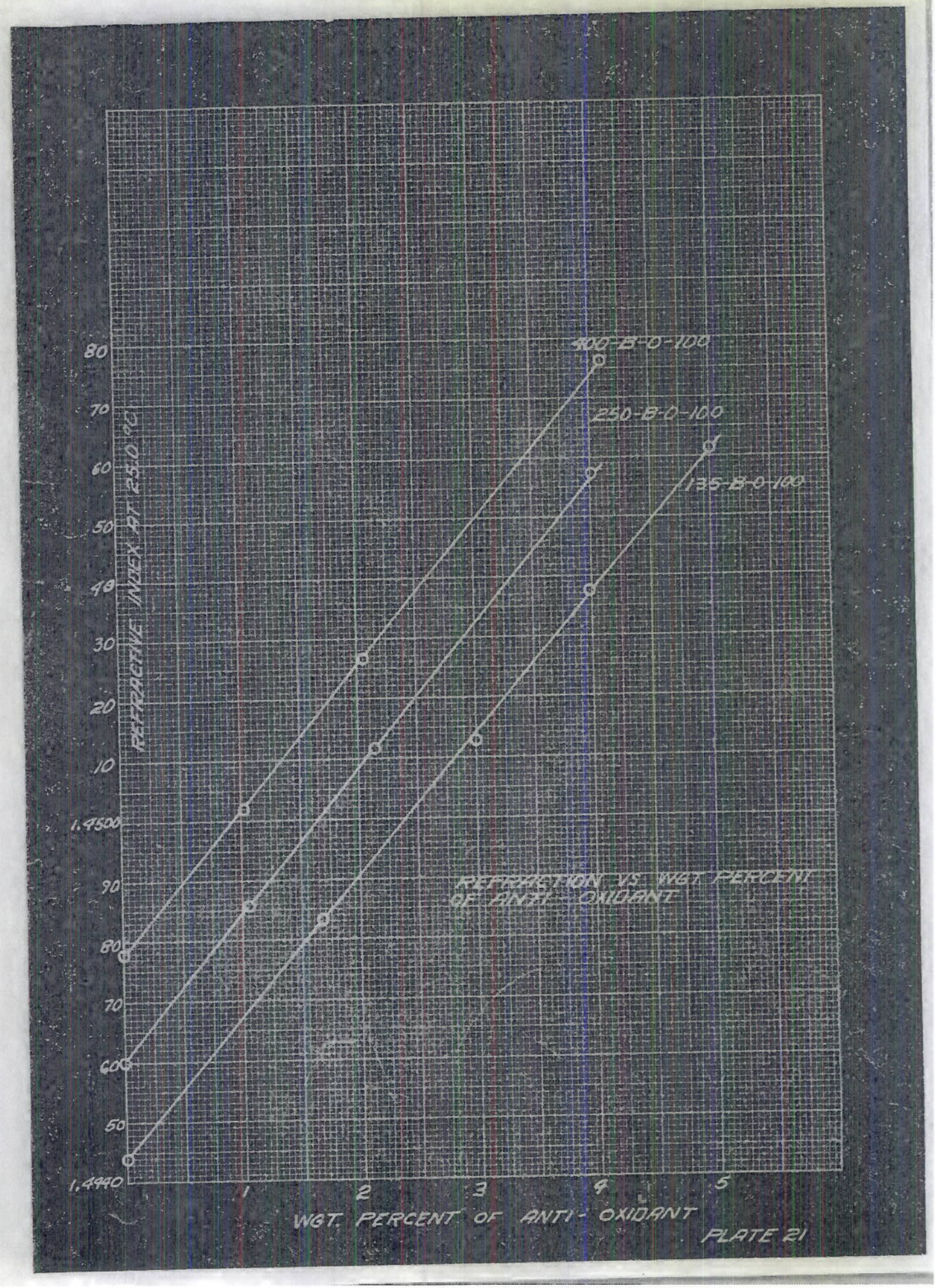
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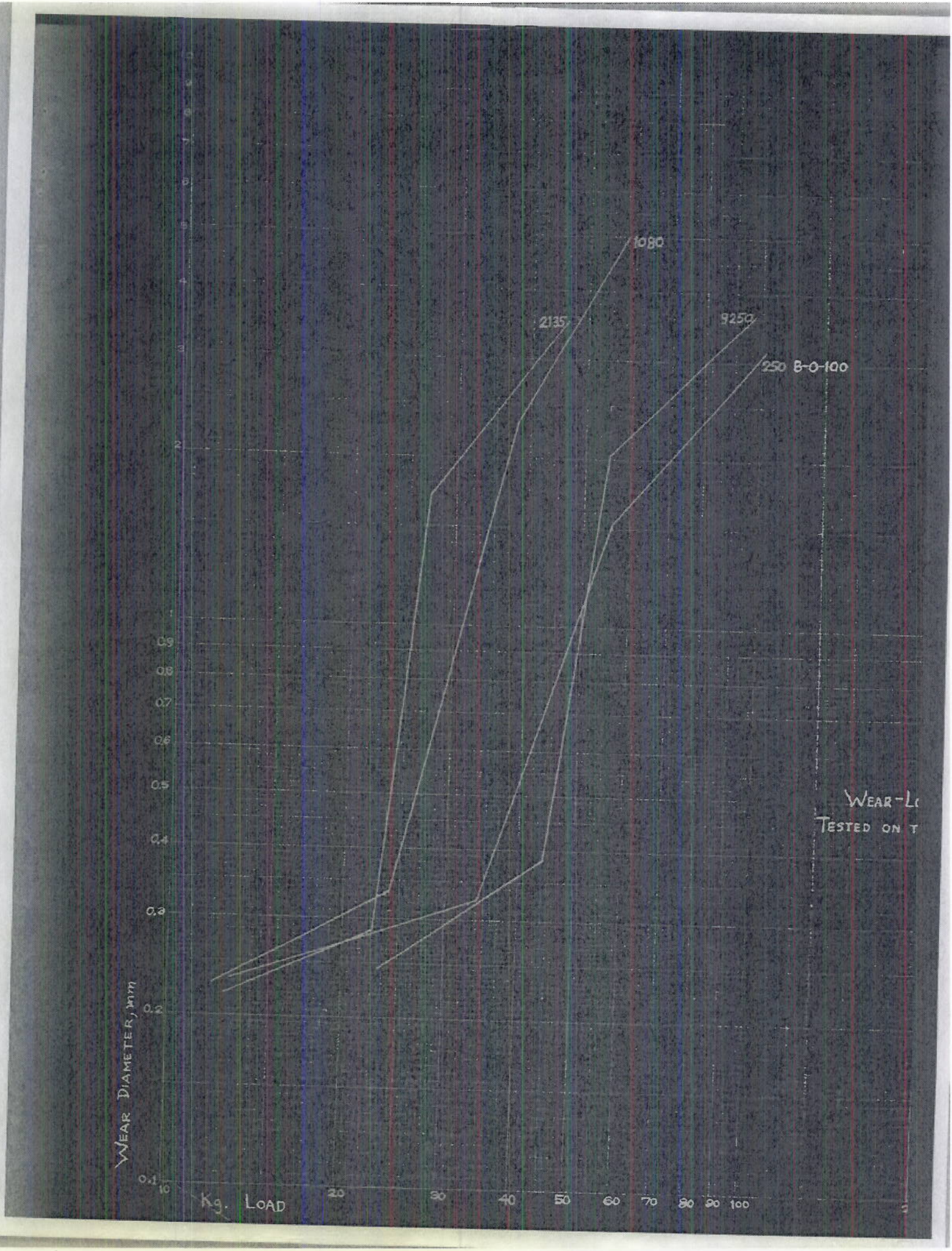
PLATE 20

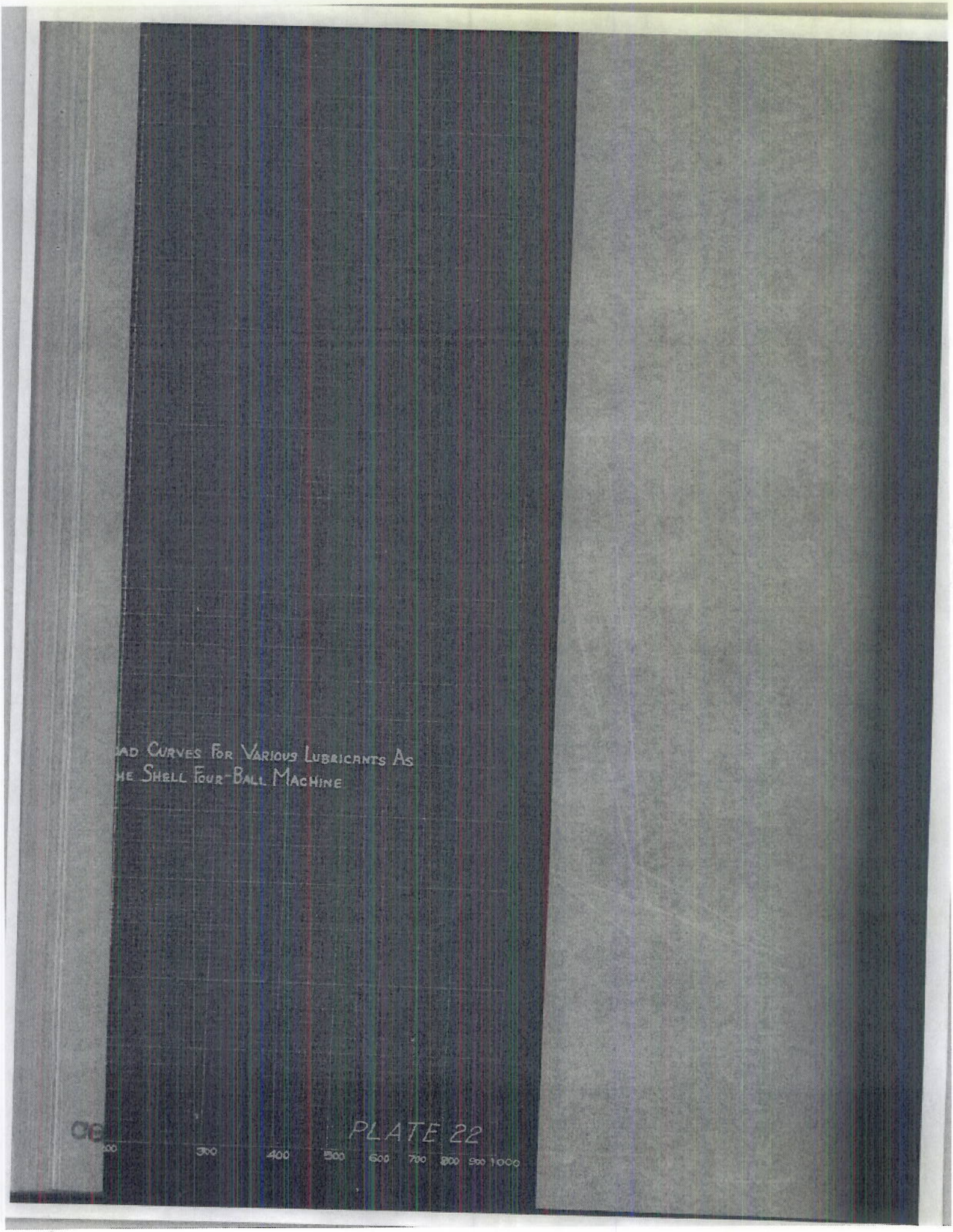


REFRACTION VS WGT PERCENT OF ANTI-OXIDANT

PLATE 21


400-B-0-100
250-B-0-100
135-B-0-100
REFRACTIVE INDEX AT 25.0°C
80
70
60
50
40
30
20
10
1.4500
90
80
70
60
50
1.4440
1
2
3
4
5
WGT. PERCENT OF ANTI-OXIDANT
REFRACTION VS. WGT. PERCENT OF ANTI-OXIDANT
PLATE 21


1080
2135
9250
250 B-O-100
WEAR-L
TESTED ON T
WEAR DIAMETER, mm
Kg. LOAD
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
2
10
20
30
40
50
60
70
80
90
100


AD CURVES FOR VARIOUS LUBRICANTS AS
HE SHELL FOUR-BALL MACHINE
PLATE 22
300
400
500
600
700
800
900
1000

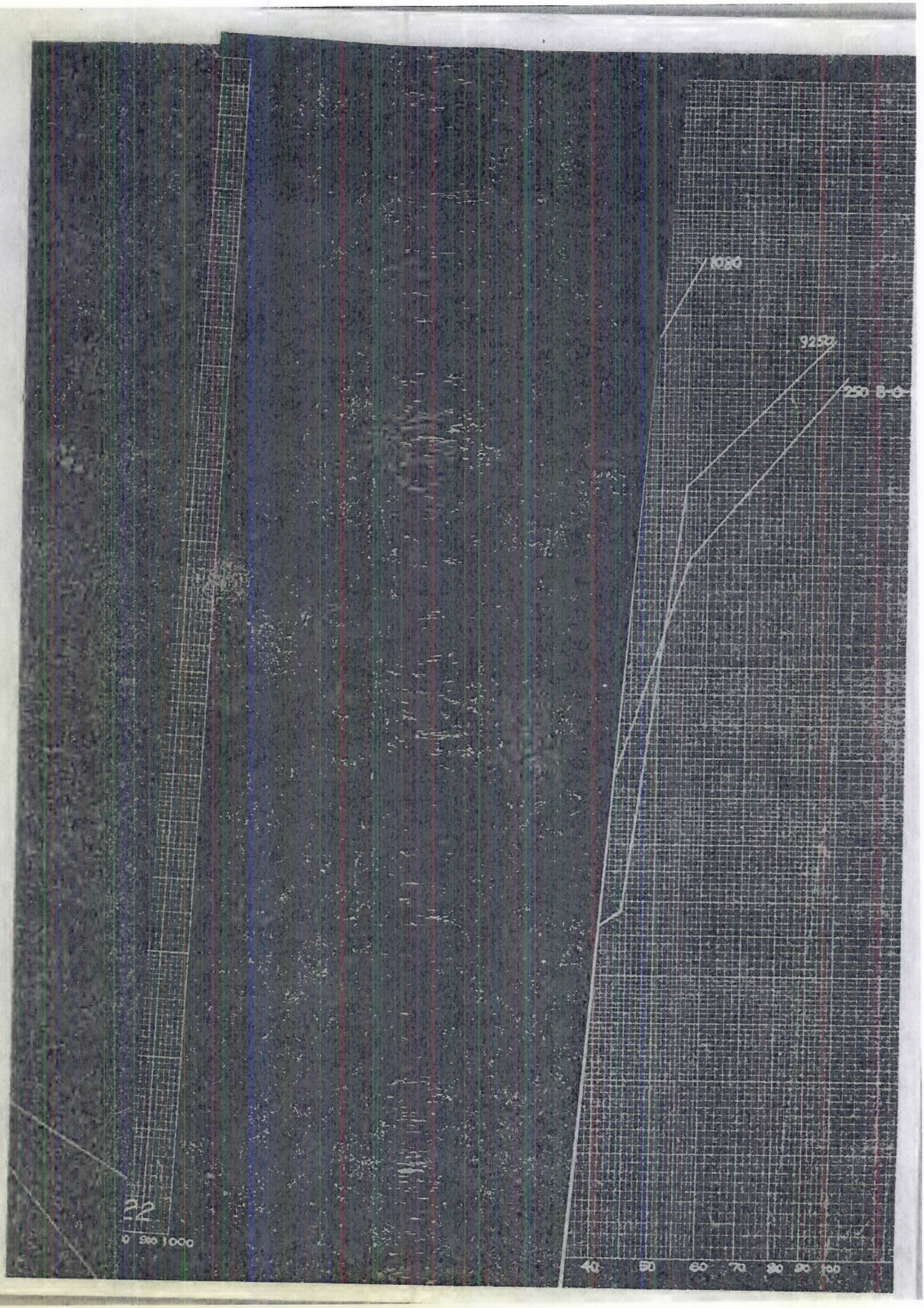

1080
925
250
40
50
60
70
80
90
100
900
1000

Table I

Manufacturer's Identification of B-O-100 Fluids

Amt. rec'd	Manufacturer's identification		Viscosity code	Inhibited by mfgr.	Date rec'd
	Serial No.	Works Lab. No.			
5 gal.	11679	S-61927	250B-0-100	No	10-14-43
5 "	1500 *		400B-0-100	No	10-14-43
15 "	VB 12464 and 12461**	S-61927	250B-0-100	Yes	11-16-43
5 "		N3-38	135B-0-100	No	12-4-43
5 "		N3-38	135B-0-100	Yes	12-4-43
5 "	24450	S-74853	250B-0-100	No	12-19-43
55 "	V.S. 44403	S-74853	250B-0-100	No	1-17-44
5 "	44400	23293	400B-0-100	No	1-17-44
5 "	44397	23293	400B-0-100	No	1-17-44
5 "	44398	23293	400B-0-100	No	1-17-44
5 "	44399	23293	400B-0-100	No	1-17-44
5 "		75406	135B-0-100	No	1-17-44
5 "		75406	135B-0-100	No	1-17-44
5 "		75406	135B-0-100	No	1-17-44
5 "		75406	135B-0-100	No	1-17-44

* Batch #1
** Obtained from drums shipped by mfgr. to U.S. Naval Engineering Experiment Station, Annapolis, Md.

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Table II

Viscosity Temperature Characteristics

Identification	Viscosity in centistokes 210°F	130°F	100°F	77°F	32°F	0°F	-20	-40°F	ASTM slope 0°-210°F	Visc. index
B-0-100 fluids										
135 (uninhibited)	6.348	18.02	31.82	55.59	248.4	1189	4759	26400	0.676	150
135 (+ 2% anti-oxidant)	6.430	18.72	33.80	60.25	285.0	1469	5989		0.691	144
250 (uninhibited)	10.19	30.72	56.06	101.5	503.0	2740	11750	82700	0.629	147
250 (+ 2% anti-oxidant)	10.35	31.92	59.70	110.5	585.8	3413	16010		0.645	143
400 (uninhibited)	16.37	51.53	96.93	178.8	938.7	5551	25670		0.587	144
400 (+ 2% anti-oxidant)	16.52	53.54	102.6	195.0	1087	6092	34010		0.591	141
Petroleum oils										
Navy Symbol 2110	5.478	18.69	36.99	70.59	394.3	**			0.765*	89
Navy Symbol 1047	6.232	20.66	39.81	74.08	361.7	2200#	**		0.730	113
Navy Symbol 2135	6.725	28.65	65.86	148.0	1340	15000#	**		0.828	40
Navy Symbol 1080	15.88	74.73	171.5	377.3	2800##	**			0.664*	102

\# Non-Newtonian.
Apparent viscosity increased with each determination (Non-Newtonian behavior).
* A.S.T.M. slope, 32° to 210°F.
** Will not flow.

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Table III

Weight Losses and Change in Viscosity with Evaporation
(after 210 hours at 150°F)

Identification	Viscosity in centistokes at 100°F			% loss in weight
	Original	After 210 hrs.	% change	
B-O-100 fluids				
135 uninhibited	31.82	30.21	-5.06	3.7
135 inhibited*	33.80	34.24	+1.30	0.7
250 uninhibited	56.06	53.61	-4.37	2.1
250 inhibited*	59.70	60.26	+0.94	0.3
400 uninhibited	96.93	78.24	-19.27	3.2
400 inhibited*	102.6	103.9	+1.25	0.4
Petroleum oils				
Navy Symbol 2110	36.99	37.23	+0.65	0.3
Navy Symbol 1047	39.81	39.95	+0.35	0.1
Navy Symbol 2135	65.86	67.42	+2.27	0.9
Navy Symbol 1080	171.5	172.2	+0.41	0.05

* 2% by weight of anti-oxidant.

Table IV

Effect of Inhibitor on Oxidation Rate at 175°F

Viscosity (cs. at 100°F)	135B-O-100 anti-oxidant	250B-O-100 anti-oxidant			400B-O-100 anti-oxidant
	0%	0%	1/2%	2%	0%
Initial	31.8	56		59.9	96.9
After 26-1/2 hrs.	24.79	38.4	57.3	59.6	46.8
After 197 hrs.			57.4	59.7	
Neutralization number (mg. KOH/gm.)					
Initial	0.0	0.0	0.0	0.0	0.0
After 26-1/2 hrs.	45.0	43.0	0.3		39.0
After 197 hrs.			0.1	0.1	
Volatile acids (mg. of KOH)					
After 26-1/2 hrs.	500	88	17	17	30.3

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Table V

Effect of Inhibitor on Oxidation Rate at 200°F

	135B-0-100 anti-oxidant 0%	135B-0-100 anti-oxidant 1/2%	135B-0-100 anti-oxidant 2%
Viscosity (cs. at 100°F)			
Initial	32.82		33.97
After 22 hrs.	18.24		
After 197 hrs.		32.25	33.82
Neutralization number (mg. KOH/gm.)			
Initial	0.0	0.0	0.0
After 6 hrs.	32.0		
After 22 hrs.	108.0	0.17	0.11
After 197 hrs.		0.22	0.22
Volatile acids (mg. of KOH)			
After 22 hrs.	1800		
After 197 hrs.		6.4	18.2

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Table VI

Oxidation Stability at Elevated Temperatures

	135B-0-100 2% anti-oxidant 200°F	135B-0-100 2% anti-oxidant 250°F	250B-0-100 2% anti-oxidant 175°F	250B-0-100 2% anti-oxidant 250°F
Viscosity (cs. at 100°F)				
Initial	33.97	33.97	59.93	59.81
After 200 hrs.	33.82	34.36	59.70	60.74
Neutralization number (mg. KOH/gm.)				
Initial	0.0	0.0	0.0	0.0
After 200 hrs.	0.2	0.2	0.1	0.2
Volatile acids (mg. of KOH)				
After 200 hrs.	18.2	22.4	17	24.8

Table VII

Effect of Common Metals on Oxidation Stability*

	Cold-rolled steel	17ST aluminum	Copper	Brass
Viscosity (cs. at 100°F				
Initial	59.81	59.81	59.81	59.81
After 211 hrs.	61.12	60.17	60.29	59.80
Neutralization number (mg. KOH/gm.)				
Initial	0.0	0.0	0.0	0.0
After 211 hrs.	0.1	0.1	0.2	0.2
Volatile acid (mg. KOH)				
After 116 hrs.	9.8	12.3	16.3	12.9
After 211 hrs.	10.9	14.3	21.9	16.5

* Temperature 200°F; 250B-0-100; 2% anti-oxidant; and 22 $cm.^2$ metal.

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Table VIII

Effect of Dissolved Catalytic Agent on Oxidation Stability

	1/4% anti-oxidant .06% copper naphthenate	1/2% anti-oxidant .12% copper naphthenate	1% anti-oxidant .25 copper naphthenate
Viscosity (cs. at 100°F)			
Initial			58.82
After 48 hrs.	57.06	58.51	60.53
After 115 hrs.	38.97	58.58	60.05
After 167 hrs.	15.72	52.24	61.00
After 211 hrs.	7.03	40.13	60.69
Neutralization number (mg. KOH/gm.)			
Initial			0.78
After 115 hrs.	56	2.3	1.34
After 167 hrs.	169	13.4	1.1
After 211 hrs.	314	59.5	1.3
Volatile acids (mg. of KOH)			
After 115 hrs.	360	90	1.01

* Using 250B-O-100 fluid at 200°F

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Table IX

Effect of Anti-Rust Additives on Oxidation Stability*

Additive	Visc. cs. at 100°F		Neutralization No.	
	Initial	After 200 hrs.	mg. KOH/gm Initial	After 200 hrs.
0.5%palmitic acid	59.87	59.9	1.40	1.40
1.0% stearolic acid		60.64	2.33	2.13
0.5% phenylstearic acid		60.67	0.9	0.90
0.2% p.n.-dodecyloxy benzoic acid		60.41	0.4	0.45
1.0% naphthenic acids		61.07	3.0	2.50
1.0% sorbitan mono-oleate		61.05	0.28	0.22
1.0% n-hexadecyl amine		61.26	0.20	0.17
1.0% mixture of mono-octadecenyl amine & monooctadecendienyl amine		60.93	0.34	0.34
0.5% monohexadeceyl ammonium stearate		60.38	0.10	0.67
0.75% phenyl alpha naphthyl ammonium stearate		61.26	2.19	1.06

* 250B-0-100, 2% anti-oxidant, and anti-rust additive listed above.

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Table X

Effect of Metals on Oxidation Stability at Extreme Temperatures*

	135B-0-100 2% anti-oxidant				250B-0-100 2% anti-oxidant		
	No metal	72 cm^2 35 cm^2 35 cm^2	bronze c.r. steel chromium		No metal	22 cm^2 c.r. steel	22 cm^2 copper
Viscosity (cs. at 100°F)							
Initial	33.97	33.97			59.81	59.81	59.81
After 208 hrs.	34.36	38.02			60.74	60.24	62.23
Neutralization number (mg. KOH/gm)							
Initial	0.0	0.0			0.0	0.0	0.0
After 208 hrs.	0.2	1.6			0.2	0.3	0.3
Volatile acids (mg. of KOH)							
After 68 hrs.	11.1	73.0			14.5	12.3	23.6
After 163 hrs.	19.2	190.5			21.7	18.5	37.4
After 208 hrs.	22.4	244.0			24.8	21.3	42.4

* Temperature 250°F.

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Table XI

Inflammability Data

Fluid tested	Approx. visc. in cs. @ 100°F	Flash# pt. °F	Fire# pt. °F	Flame propagation % oxygen	S.I.T. °F	Incendiary tests		
						No of samples	Height flame	Duration of fire
0.S.-2943*	27	225	235	12		1	20-25'	0.6 sec.
N.S.-2135**	66	340		34	680	4	15-20	0.6
135B-0-100	32			52-56	720	0		
135B-0-100, inhibitor	34	380	445	52-56	720	2	2-3	0.2
250B-0-100	56	442/455	486/500	52-56	725	3	1-2	0.2
250B-0-100, inhibitor	60	465	570	52-56	745	3	0	0
400B-0-100	97	446	468	52-56	727	1	0	0
400B-0-100, inhibitor	103			52-56	752	0		
Hydrolube 2229-C	29	none	none	over 80	885	10	0	0

* Standard Oil of New Jersey hydraulic gear oil (Ordnance spec. 2943)
** Texaco non-inhibited petroleum oil (Navy Symbol 2135)
\# Data reported by Carbide & Carbon Chemicals Corporation

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Table XII

Surface Tensions at 25°C

Fluid	Surface tensions dynes per cm.
135B-0-100 Batch N3-38 as rec'd	30.6
250B-0-100 Can 24450 as rec'd	30.8
400B-0-100 Batch #1 as rec'd	31.4
135B-0-100 Batch N3-38 with 2% anti-oxidant as rec'd	30.2
250B-0-100 Annapolis Batch with 2% anti-oxidant, as rec'd	31.0
400B-0-100 with 2% anti-oxidant	31.4
Petroleum (kerosene)	30-31
Petroleum (lubricating oils)	30-37

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Table XIII

Comparison of Specific Gravities of B-O-100 and Petroleum Oils

Fluid	Visc. cs. at 100°F	Specific Gravity	
		100°/60°F	60°/60°F
135B-O-100	33.80	0.977	0.995
Navy Symbol 2110	36.99	0.870	0.885
Navy Symbol 1047	39.81	0.847	0.861
250B-O-100	59.70	0.983	1.000
Navy Symbol 2135	65.86	0.902	0.916
400B-O-100	102.6	0.991	1.008
Navy Symbol 1080	171.5	0.871	0.886

Table XIV

Thermal Expansion Data

Fluid	Coefficient of expansion/°F	
	-20 to 130°F	130° to 210°F
135B-O-100	0.00043	0.00046
250B-O-100	0.00043	0.00045
400B-O-100	0.00041	0.00044

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Table XV

Emulsification of Inhibited B-O-100 Fluids
(in percent of total water separated)

Temp °F	Percent water used to make emulsion	Percent of emulsified water separated after: 5 min.	10 min.	30 min.	1 hr.	24 hrs.
		(a) 135B-O-100				
34	10	0	2	10	25	50
77		0	0	5	20	45
140		0	5	10	30	68
180		0	10	25	50	85
34	20	15	40	65	70	75
77		10	30	60	70	80
140		20	50	75	80	85
180		80	82	86	90	91
34	50	76	90	92	94	94
77		72	84	90	92	95
140		86	94	96	96	96
180		94	96	96	96	96
		(b) 250B-O-100				
34	10	0	0	0	0	30
77		0	0	0	0	20
140		0	0	0	10	60
180		0	0	5	15	70
34	20	0	30	50	70	80
77		0	20	60	60	70
140		10	40	70	70	80
180		70	75	80	80	90
34	50	90	92	94	94	94
77		86	88	92	92	96
140		90	94	94	94	96
180		92	96	96	96	98
		(c) 400B-O-100				
34	10	0	0	0	5	30
77		0	0	0	0	20
140		0	0	0	10	50
180		0	0	10	30	60
34	20	0	0	0	15	60
77		0	0	0	10	50
140		0	0	5	20	70
180		0	0	10	15	80
34	50	0	0	5	20	88
77		0	0	0	15	84
140		0	0	40	65	92
180		0	0	50	70	92

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Table XVI

Emulsions of Oxidized 250B-0-100 Fluid

Temp. °F	Percent water used to make emulsion	Percent of emulsified water separated after:				
		5 min.	10 min.	30 min.	1 hr.	24 hrs.
34	10	0	0	0	0	20
77		0	0	0	0	10
140		0	0	0	5	50
180		0	0	0	10	60
34	50	90	94	94	94	96
77		80	84	88	90	94
140		86	88	90	92	96
180		92	94	94	94	98

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Table XVII

B-0-100 Soluble Corrosion Inhibitors
(Tested with distilled water)

Additive	Percent by wt.	Corrosion rating	Cold storage effect in 2 mo.
Lauric acid	0.5	0	S (-40°F)
Myristic acid	0.5	0	S (-20)
Palmitic acid	0.5	0	S (0)
Phenyl stearic acid	0.5	0	S (-40)
Xylyl stearic acid	0.5	0	S (-40)
Ethyl phenyl stearic acid	0.75	0	S (-40)
Stearolic acid	1.0	0	S (-40)
p.n.-dodecyloxy benzoic acid	0.2	0	S (-40)
Palmitic alpha hydroxy acid	0.5	0	S (-40)
Naphthenic acid, M.W. 240	1.0	0	S (-40)
Naphthenic acid, M.W. 440	0.5	0	S (-40)
Hexadecyl amine	1.0	0	S (0)*
Mixture of n mono-octodecenyl and n mono-octadecendienyl amine	1.0	0	S (-40)
Zinc naphthenate	0.5	0	S (-40)
Zinc stearate	0.2	0	S (-20)*
Sorbitan monooleate	1.0	0	S (-40)
Phenyl alpha naphthyl-ammonium stearate	0.75	0	S (0)
Mono-dodecyl ammonium stearate	1.0	0	S (-20)
Mono-tetradecyl ammonium stearate	0.75	0	S (0)
Mono-hexadecyl ammonium stearate	0.5	0	S (0)
Mono-octadecyl ammonium stearate	0.5	0	S (0)

* Slightly murky on standing, but dissolves readily when agitated.
S Soluble.

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Table XVIII

Emulsification of 250B-O-100 Fluids Containing Rust Inhibitors

Temp. °F	Percent water used to make emulsion	Percent of emulsified water separated after: 5 min.	10 min.	30 min.	1 hr.	24 hrs.
(a) 0.5% phenyl stearic acid used as inhibitor						
34	10	0	0	0	0	50
77		0	0	0	0	10
140		0	0	5	20	60
180		0	0	10	30	80
(b) 1.0% stearolic acid used as inhibitor						
34	10	0	0	0	0	75
77		0	0	0	0	10
140		0	0	5	10	70
180		0	0	10	20	80
(c) 0.2% p.n.-dodecyloxy benzoic acid used as inhibitor						
34	10	0	0	0	0	70
77		0	0	0	0	10
140		0	0	5	10	75
180		0	0	10	20	85
(d) 0.5% naphthenic acid, M.W. 440, used as inhibitor						
34	10	0	0	0	0	70
77		0	0	0	0	10
140		0	0	10	15	70
180		0	0	20	30	80
(e) 1.0% mixture of n-mono octadecenyl and n-mono octadecendienyl amine used as inhibitor						
34	10	0	0	0	0	60
77		0	0	0	0	10
140		0	0	5	10	70
180		0	0	10	20	80
(f) 0.5% zinc naphthenate used as inhibitor						
34	10	0	0	0	0	50
77		0	0	0	0	10
140		0	0	5	5	75
180		0	0	10	20	80

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Table XIX

B-0-100 Soluble Corrosion Inhibitors
(Tested with synthetic sea water)

Additive	Percent by wt.	Corrosion rating	Cold storage effect of one week	Cold storage effect of one week
Lauric acid	0.5	1/2		S (-40)
" "	1.0	1/16	S (-40)	
Phenyl stearic acid	0.5	5		S (-40)
" " "	1.5	0	S (-40)	
Stearolic acid	1.0	6		S (-40)
" "	3.0	0	S (-20)	
p.n.-dodecyloxy benzoic acid	0.2	5		S (-40)
p.n.-dodecyloxy benzoic acid	0.6	0	S (-20)	
Naphthenic acid, M.W. 240	1.0	3		S (-40)
" " " "	2.0	1/8	S (-40)	
" " " 440	0.5	4		S (-40)
" " " "	1.5	0	S (-40)	
Mixture of n-mono octo-decenyl and n-mono octa-decendienyl amine	1.0	4		S (-40)
Mixture of n-mono octo-decenyl & n-mono octa-decendienyl amine	3.0	2	S (-20)	
Zinc naphthenate	0.5	5		S (-40)
" "	1.5	2	S (-40)	
Zinc stearate	0.2	4		S (-20)*
" "	0.4	2	S (+50)*	
Sorbitan monooleate	1.0	1/4		S (-40)
" "	2.0	0	S (-40)	
Mono-tetradecyl ammonium stearate	1.0	5		S (0)
Mono-tetradecyl ammonium stearate	2.0	1/8	S (+77)	

* Slightly murky on standing, but dissolves readily.
S Soluble.

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Table XX

Comparison Between Ultraviolet Stabilities of B-O-100 Fluids and Naval Specification Petroleum Oils

Identification	Viscosity in centistokes at 100°F					Percent change in viscosity	Neutralization No.	
	Original	After 24 hrs.	After 48 hrs.	After 96 hrs.	After 240 hrs.		Orig.	After 240 hrs.
B-O-100 fluids								
Uninhibited								
135B-O-100	31.82	32.38	32.60	32.80	34.16	7.36	0.40	0.87
250B-O-100	56.06		58.95	59.78	61.29	9.23	0.11	
400B-O-100	96.93	102.0	104.1	106.7	112.0	15.55	0.17	0.57
Inhibited								
135B-O-100	33.80	33.89	34.03	34.16	34.37	1.69	0.25	0.87*
250B-O-100	59.70	59.33	59.88	60.08	60.32	1.04	0.06	0.44
400B-O-100	102.6	102.4	102.7	102.9	103.5	0.88	0.17	0.44
Petroleum oils								
Navy Symbol 2110	36.99	37.11	37.10	37.25	37.38	1.03	0.07	0.12
" " 1047	39.81	39.63	39.85	39.76	39.97	0.29	0.07	0.12
" " 2135	65.86	66.19	66.21	65.78	66.49	0.96	0.08	0.12
" " 1080	171.5	174.3	173.2	174.1	172.4	0.53	0.10	0.18

* Sample so dark that the end point of the titration was obscured.

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Table XXI

Refractive Index at 25.0°C

Fluid	Refractive index
135B-0-100 (Batch N3-38 as rec'd)	1.4445
Same but with 2% anti-oxidant	1.4490
250B-0-100 (Can 244050 as rec'd)	1.4463
Same but with 2% anti-oxidant	1.4507
400B-0-100 (Batch #1 as rec'd)	1.4481
Same but with 2% anti-oxidant	1.4526
Light and Heavy petroleum oils	1.41 to 1.48

Table XXII

Hydrolysis Tests

Sample	Hours refluxed	Atmosphere	pH
Distilled water	0	air	6.0-7.0
Uninhibited 250B-0-100 & distilled water	0	air	6.0-7.0
" "	24	nitrogen	3.5-4.2
" "	24	air	3.8-3.9
" "	46	air	3.2
" "	100	air	2.7
Inhibited 250B-0-100 & distilled water	0	air	4.2
" "	24	nitrogen	3.68
" "	46	air	3.5
" "	100	air	3.4
Inhibited 250B-0-100 & sea water	100	air	3.5

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Table XXIII

Hydroxyl Determinations

Sample	Percent hydroxyl: Pyridine-acetic anhydride method	Sodium method
135B-0-100 uninhibited	2.70	
250B-0-100 "	1.83	1.88
400B-0-100 "	1.10	1.3
400B-0-100 uninhibited dried over BaO	1.04	

Table XXIV

Data of Four-Ball Machine Tests on B-0-100 Fluids and on Some Well Known Petroleum Oils

Identification of oil	L_1(kg.)	W_1(mm.)	L_2(kg.)	W_2(mm.)	PWI
250B-0-100 inhibited	34	0.33	56	1.51	10,1
250B-0-100 " *	36	0.34	54	1.62	7.2
400B-0-100 "	37	0.34	56	1.60	7.8
135B-0-100 "	34	0.33	55	1.72	7.4
Navy Symbol 2135#	22	0.27	27	1.70	1.77
Navy Symbol 9250**	44	0.39	55	1.99	2.89
Navy Symbol 1080##	24	0.34	38	2.30	2.70

* Duplicate run on another day.
** Detergent type petroleum oil.
# Non-additive, low V.I. petroleum oil.
Non-additive, nigh V.I. petroleum oil.

Note: L_1 and W_1 are applied load and average wear diameter respectively at P_1. L_2 and W_2 are similar quantities at point P_2. All Carbide & Carbon fluids were inhibited with 2% anti-oxidant.

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