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The Laboratory Evaluation of Hydraulic Oils

I - Methods for Inflammability Test

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Data Collected by: Miles V. Sullivan, Contract Employee

Robert W. Miller, Ens. USNR

John K. Wolfe, Contract Employee

Prepared by: John K. Wolfe, Contract Employee

Reviewed by: W. A. Zisman, Senior Chemist, Lubrication
Section

P. Borystrom, Head Chemist, Superintendent
Chemistry Division

Approved by: A. H. Van Keuren, Rear Admiral, USN
Director

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TABLE OF CONTENTS

ABSTRACT	Page
INTRODUCTION	
(a) Authorization	1
(b) Statement of the Problem	1
(c) Known Facts Bearing on the Problem	2
(d) Narrative of the Original Work Done at this Laboratory	5
METHODS	
(a) Apparatus Used	8
A. Flame Propagation Chamber	8
B. Electrical Ignition System	9
C. Gas Measurement and Control Device	10
D. Oil Metering Device	12
(b) Experimental Procedure	12
DISCUSSION OF RESULTS	
(a) Data Obtained	14
(b) Comparison of the Present Method with other Inflammability Tests	18
A. Flash points	18
B. Fire points	19
C. Spontaneous Ignition Temperature	20
D. Incendiary Firing Tests	20
CONCLUSIONS AND RECOMMENDATIONS	20
BIBLIOGRAPHY	
APPENDICES	
	Table
Legend of Apparatus Described in Plate 1	1
Legend of Apparatus Described in Plate 3	2
Inflammability Results on Hydraulic Oils and Fluids Tested	3
	Plate
Propagation Chamber and Spray Equipment	1
Propagation Chamber - Top View	1A
Spray Gun	2
Gas Measurement and Control Equipment	3
Mixing Chamber Detail	4
Electrical Circuit	5
Oil Metering Device	6

ABSTRACT

This is the first in a series of studies of hydraulic oils undertaken to develop non-inflammable fluids for use in the hydraulic systems of naval vessels and aircraft. It is necessary that laboratory tests be devised to simulate the fire hazards of actual practice under battle conditions in order that the hydraulic oils developed in the present program may be properly evaluated.

This report describes a new method for inflammability tests on hydraulic oils by measurement of the oxygen concentration necessary for flame propagation of a finely atomized spray of the oil when a high temperature ignition source is present. The atomized spray is ignited by means of an electric arc and the propagation of the flame back to the spray source is observed. The gas used in producing this spray is an oxygen-nitrogen mixture whose composition can be continuously varied. The oxygen-nitrogen ratio of gas is changed until the mixture just propagates the flame back to the source and this critical oxygen concentration is a measurement of the inflammability.

The analogue of this test in actual practice is the inflammability of an oil spray resulting from the penetration or rupture of a high pressure hydraulic line and the subsequent propagation back to the line or the hydraulic reservoir. The ignition under actual conditions may arise from a shell fragment, incendiary bullet or from contact with a hot metal surface such as an exhaust pipe, but in any case an estimation of the relative inflammability can be made by a propagation measurement with increased oxygen present. The present method is compared with the known methods for evaluation of hydraulic oils and their use individually and collectively in predicting inflammability of fluids is discussed.

It is recommended that proposed hydraulic oils and their base stocks be evaluated for inflammability behavior by measurement of:

- a. Flash point
- b. Fire point
- c. Spontaneous ignition temperature
- d. Flame propagation of the spray
- e. Incendiary firing tests

Other reports on the results of inflammability measurements using these methods together with the results of incendiary firing tests will be submitted at a later date.

INTRODUCTION

(a) Authorization

1. This research was authorized by Bureau of Ships as Project No. 1121/42.

(b) Statement of the Problem

2. In connection with the War Effort a considerable amount of research has been sponsored by the Navy Department in developing less inflammable or non-inflammable oils for use in hydraulic systems and in other naval equipment where the fire hazard is a potential danger to naval personnel and equipment. It is necessary that laboratory tests be devised to simulate the fire hazards of actual practice under battle conditions in order that with such tests the hydraulic oils developed in the present program may be properly evaluated.

3. One of the potential fire hazards frequently encountered in naval practice is the penetration or rupture of a high-pressure hydraulic line by a bullet or shell fragment and the production of an atomized spray of the liquid hydraulic oil by passage through the orifice. When this spray strikes a hot surface, spontaneous ignition may result. The likelihood of ignition under these circumstances may be determined roughly by a measurement of the spontaneous ignition temperature. The laboratory value obtained must be considered as a minimum point at which ignition can occur and fire will not necessarily result in actual practice since the vapor concentrations in the proximity of the open hot surface are not directly comparable to the measurements of spontaneous ignition temperature made in a closed system. This standard test, however, is of little help in predicting the ease of propagation of the flame throughout the spray and back to the break in the line. This report describes a method by which atomized sprays produced from oils proposed for use in naval military equipment may be evaluated according to their relative ability to propagate a flame.

4. Several oils have been submitted to the Naval Research Laboratory by groups under Navy contract to perform research work on the development of non-inflammable or less-inflammable fluids. Such liquids vary widely in their inflammability characteristics when measured and evaluated by ordinary procedures useful in evaluating the fire hazards of petroleum base oils. Contradiction between laboratory measurements of inflammability and practical experience often occur and are extremely difficult to explain. By the method herein described it is possible to evaluate in the

laboratory the probable incidence of fire under a condition known to occur in actual practice.

(c) Known Facts Bearing on the Problem

5. The determination of flash point has been used extensively as a method of measuring and evaluating the inflammability of petroleum fluids. This test consists of 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 is taken as the temperature at which a flash of flame appears on the surface of the liquid. Evaluation of the fire hazard of light petroleum fractions by means of the flash point has been found to be very useful, particularly on liquids in the gasoline, kerosene, and light fuel oil ranges, but its usefulness in predicting the fire hazard of relatively non-volatile petroleum fractionation cuts is not well established. In many instances, however, the flash point has been used to describe the relative fire hazard of petroleum and non-petroleum hydraulic fluids. This procedure has been found here to be inadequate, and a number of different methods will be necessary to match the nature of the inflammability circumstance involved, each designed to obtain a true evaluation of relative fire hazard.

6. The fire point may be determined in the same apparatus used for the determination of flash point. The fire point is taken as the temperature at which the oil takes fire and continues to burn for a period of at least five seconds. With many low boiling petroleum fractions as well as with many volatile organic liquids the flash and fire points are almost identical, the fire point occurring only a few degrees above the flash point. Higher boiling, less volatile oils and fluids have a wider temperature differential between flash and fire points. This difference may have some added significance especially when applied to non-petroleum fluids. A rough correlation has been observed between this difference and the flame propagation tendencies in that the greater the differential, the more difficult the spray is to ignite.

7. 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 onto a hot surface in an enclosed space. The determination of Autogenous Ignition Temperatures by the A.S.T.M. method is accomplished by heating a 125 ml Erlenmeyer flask in a bath of liquid solder. Small amounts of sample are admitted and the temperature of the bath recorded at the lowest ignition point. The temperature of ignition is noted by measurement on a thermocouple or a thermometer placed in the solder bath. The volume of sample admitted

is changed until the amount just producing ignition is determined. This quantity is then used to determine the minimum Autogenous Ignition Temperature. It is necessary to keep a slow stream of air flowing during the interval between samples to remove the exhaust fumes and provide a fresh oxygen supply.

8. A modification of the Spontaneous Ignition Determination which permits observation at much higher temperatures has been made. This apparatus consists of a stainless steel cup with a side inlet to furnish preheated fresh air, and provided with a thermocouple for temperature measurement. Ignition temperatures obtained in this apparatus may be much lower than those obtained in the A.S.T.M. glass vessel since the metal by catalysis may lower the Spontaneous Ignition Temperature.

9. The conditions under which the Spontaneous Ignition Temperature is determined must be specified in order that the reported value may be reproducible. Some of the factors which should be controlled and reported are:

- (a) Size of sample drop used
- (b) Rate of supply of air to ignition chamber
- (c) Volume of ignition chamber
- (d) Temperature of incoming air
- (e) Material used for construction of chamber and material upon which the drop impinges
- (f) Time interval between droplets

10. A fire hazard known to exist in actual practice is one resulting from the penetration of a hydraulic system by high explosive or incendiary missiles. Tests at both the Naval Proving Ground, Dahlgren, Virginia and Wright Field, Dayton, Ohio showed that many fires were started by shooting bullets through experimental cans (1 gal.) containing the petroleum base oils of the type now used in naval equipment. Although fires can be started when 50 caliber high explosive or incendiary bullets are fired into the oils directly, a much larger percentage of fires can be obtained when there is placed in the line of fire metal plates or "baffle plates" thick enough to fragment and ignite the bullet on impact. If the one gallon can of oil is placed endwise so that the bullet passes through the largest dimension with the burning missile in contact with the oil for the maximum time, the greatest percentage of fires is obtained. Practically 100% of fires can be obtained using this method on petroleum base oils. The use of these incendiaries in modern warfare greatly increases the fire hazard and vulnerability of naval equipment whose proper function at present depends upon the use of inflammable liquids. Armor piercing shells are also known to produce fires, but as the inflammability is produced by the heat of impact, the percentage of fires is much less than under incendiary fire.

12. Several new techniques have been proposed and used by the Standard Oil Development Company of New Jersey to obtain indications of the inflammability of hydraulic oils under varying conditions.⁴ One of these is to spray the oil onto a hot surface and observe, by means of an optical pyrometer pointed at the hot surface, the approximate temperature at which the material burns. Fires can be obtained in this way using some volatile and some non-volatile oils, but the values are not very reproducible. However, many common hydrocarbons, for example benzene, which is usually considered a serious fire hazard, could be burned only with great difficulty requiring temperatures over 1700°F when tested by this method at the Naval Research Laboratory. Another inherent disadvantage aside from such an obvious discrepancy is that the ratio of concentrations of vapor and air is not constant but varies from point to point throughout the system and is not known at or near the point on the surface where the ignition occurs. Eddy currents of air, which are ever present in open laboratory tests, constantly carry the vapors of the material under test from the spot of contact with the hot surface, and the occasional flame is produced at the point where a favorable ignition mixture comes in contact with the proper catalytic surface. Once ignition has started, the remainder of the gaseous mixture in the immediate vicinity burns readily from all directions to the periphery where the volatile vapor concentration is too low to be inflammable.

13. A modification of this hot plate test has likewise been used by the Standard Oil Development Company in which drops of the oil or liquid to be tested are allowed to fall on a spiral-coil type of electrical heating element in an open system. The temperature of the element wire is increased until ignition occurs. The temperature is then determined by means of an optical pyrometer. Here again no attempt has been made to control or ascertain the gaseous mixture in the immediate vicinity of the surface. In addition to this spontaneous ignition, a determination was made in a similar manner with a small flame just over the point of contact of oil and heating element. This value is designated as the ignition point on a hot surface in the presence of a flame.

14. Inflammability studies at the Petroleum Research Laboratories at Pennsylvania State College have been carried out using a ceramic-like "globar" heating element.⁷ In this test, the element was heated electrically with the rate of heating regulated by means of a "variac" voltage controller. Droplets of liquid were allowed to fall on the globar and the presence or absence of ignition was noted. An optical pyrometer was used to measure temperatures at which ignition occurred. Results of attempts to determine inflammability by this method showed that droplets of liquid sometimes burned and sometimes bounced off without igniting or vaporizing completely. A medicinal atomizer was used to spray

the liquids onto the bar and under these circumstances certain volatile low-flash point hydrocarbons tested appeared to burn (e.g. benzene and n-hexane) while the non-volatile hydrocarbon hydraulic oil base stock failed to ignite. The effect of surface catalysis on the ignition was considerable since it was found that metal, when used in place of the globar, caused ignition more readily than did the ceramic globar, although the metal was kept at a lower temperature. In other tests a small stream of liquid was allowed to impinge on the globar and under those conditions all the liquids that were tested ignited. It was concluded, on the basis of these tests, that under sufficient impinging pressure, even the less inflammable materials will burn. An important variable in these tests was the non-uniformity of concentrations of liquid, vapor and air at the point on the surface where contact with the globar, and subsequent ignition, occurred. Since these concentrations are uncontrolled and are affected by eddy currents and room fluctuations, the ignition values cannot be expected to be reproducible. Some preliminary results obtained in the manner described above were reported as follows:

Volt esso 36 (approx. 300° flash point)	-- Burned
Ethylene glycol	-- "
Dimethyl silicone	-- "
Trimethyl phosphate	-- "

No further data was given on these inflammabilities.

15. Flame Propagation Speed Tests have been used by the Underwriters Laboratories on mixtures of combustible gases used as refrigerants as a method for the evaluation of the inflammability which approximates conditions met with in actual practice. These tests consist of igniting a mixture of gases to be tested in an explosion cylinder made of heavy steel. The ignition of the gaseous mixture is done by means of a spark plug and the progress of the flame front is determined by photographs made at regular intervals along the tube as the flame front passes from the ignition source. In these tests, it was found that propane, gasoline, and methyl formate all propagated flame at about the same speed, whereas methyl chloride had a different and much slower propagation rate. The composition of gas or vapor-air mixtures employed was carefully controlled and selected as that mixture producing the maximum pressure in explosion pressure tests previously made. This method, although requiring a more elaborate apparatus, has many obvious advantages for research purposes, and has the soundest basis from a theoretical standpoint.

(d) Narrative of the Original Work Done at this Laboratory

16. It early became apparent that the flash point, fire point, and spontaneous ignition temperature measurements either alone or together were not always appropriate measurements for

evaluating the fire hazard with regard to all practical circumstances. Two notable hazards which are not adequately evaluated by these usual determinations are (1) the penetration of hydraulic systems by incendiary bullets or shells and (2) the ignition of a fine spray of oil from a broken high pressure hydraulic line impinging on a hot metallic surface and the propagation of the flame back through the mist to the oil reservoir or line.

17. Tests begun at the Naval Research Laboratory to evaluate the fire hazards with standard petroleum tests resulted in many apparent discrepancies and irregularities. A literature investigation was then made to find methods more suitable for use in the further evaluation of inflammability. After an extensive search it was apparent that none was available. Investigations were then directed toward the development of methods of test which simulated, as nearly as possible, actual fire hazards existing in naval practice. The first crude tests consisted of dropping lighted rods of magnesium into the liquid under examination and observing whether or not the liquid ignited and burned after the magnesium had dissipated itself or become quenched. Many of the petroleum base oils burned readily, whereas several chlorinated organic compounds quenched the burning metal and did not burn. Although the temperature of the burning magnesium was high, making the test severe, the relative heat capacities of magnesium and oil sample were involved making the test unreproducible and much too qualitative.

18. The next step in the investigation was the construction of an apparatus which consisted of a tin can mounted vertically, holding in the top a burning rod of magnesium suspended on a wire. A hole was provided in the side of the can close to the bottom, and through it was sprayed a fine mist of oil in air. A household insecticide sprayer was used for the dispersion of the oil as a spray. Petroleum oils fired readily in this apparatus and the resulting rapid combustion readily blew the top off the can. This explosion and subsequent removal of the can top was taken as a positive inflammability test. Chlorinated hydrocarbon mixtures under test did not fire or explode when the same experimental conditions were used. However, considerable evolution of smoke was observed from the spray of these liquids in contact with the burning magnesium.

19. This simple vertical apparatus was improved by mounting, within the can, an electric arc consisting of two horizontal electrodes, and allowing the electrodes to touch; upon release they sprung apart and an arc discharge developed igniting the spray more efficiently than the burning magnesium. Later it was found desirable to use an electrical spark discharge apparatus so that a high voltage spark jumped the arc gap and automatically started the arc by reducing the gap resistance. This modification made use of a push button to start the arc in place of the manual touching of the electrodes.

20. The results of preliminary tests on this propagation apparatus showed that some oils would burn readily in air while others were non-inflammable under these conditions. A division of the results was made into these two groups. Inasmuch as the oxygen content of air varies with the altitude it was thought advisable to evaluate the oil mist inflammability by decreasing the oxygen-nitrogen ratio until the critical value was reached where inflammability just could be noticed. It was also realized that this critical value at atmospheric pressure may be used to rate the various oils as regards relative inflammability.

21. Apparatus was therefore constructed for producing a known oxygen-nitrogen mixture for spraying the oil. The necessary oxygen and nitrogen tanks were provided with valves, gas mixing equipment and flowmeters to permit controlling and metering the oxygen-nitrogen ratio of the mixture. As the test unit is now constructed, the oxygen percentage of the mixture can be increased to a point where the explosion or fire results. This oxygen percentage is the basis for evaluating the fluid as to its flame propagation ability when in spray form.

22. It was found that the spray gun carried the liquid into the nozzle by the aspirator effect of the passing air. Since this effect was constant throughout the determinations and the orifice size was likewise constant, the amount of oil passing to the orifice varied inversely with the viscosity of the fluid. For example, benzene had a fast rate whereas SAE #60 motor oil had an extremely slow flow. To make the apparatus independent of viscosity, a metering tube was inserted in the system and the flow to the spray gun was controlled to any desired value regardless of the aspirator effect of the gun or the pressure effect of the liquid column in the burette.

23. Apparatus has also been constructed for the determination of spontaneous ignition temperature in a stainless steel apparatus provided with a constant stream of warm air. Many determinations have been made by this method in an attempt to evaluate the data and interpret it as an indication of the probable fire hazards in naval equipment. It was noted that many discrepancies which existed when attempts were made to evaluate the fire hazard on the basis of flash point, fire point or spontaneous ignition temperature alone disappeared when the data obtained by the spray test herein described are also taken into consideration.

24. Incendiary firing tests are being made on newly developed hydraulic oils to evaluate their inflammability characteristics toward this actual practice hazard and to permit correlation with the results of the laboratory tests. A detailed report of these findings covering all the hydraulic fluids investigated will be made when tests are completed.

METHODS

(a) Apparatus Used

A. Flame Propagation Chamber

25. The method herein described for the measurement of the spray inflammability of hydraulic oils by determination of the flame propagation of the oil in spray form utilizes an apparatus consisting of a cylindrical propagation chamber constructed from two concentric brass tubes. The top view of the chamber (Plate 1A) shows the integral parts and the dimensions of the tube as constructed. A and M are brass tubes of 1/4" diameter, silver soldered into the outer brass tube serving as water outlet and inlet, respectively, for the cooling jacket. B and C are the concentric cylinders made of brass telescope tubing to form the main body of the tube. Brass tubing of 5/8" diameter (D) was silver soldered into the main body tube to serve as holders for the two porcelain insulators (E) fitted on each side of the tube. Electrodes (F) were constructed of 5/16" copper rod and were fitted securely into threaded brass holders. The hexagonal nuts (G) were fastened to the base and nuts designated (I) were soldered to the threaded brass electrode holder. Those nuts (I) were painted with consecutive numbers about the hexagonal sides so that the number of turns could be easily read and thus from the number of threads per inch, the electrode spacing could be ascertained. The dotted lines shown as L indicate the lead wires from a thermocouple having its hot junction at K. This couple is placed midway between the electrodes and the rear end of the tube. The lower end is closed with a tightly fitting brass cap (N) which holds a porcelain thermocouple insulator in the top and a brass rod (O) passing through the center of the cap to serve as a handle. A 0.235" diameter hole was made near the bottom of the cap to accommodate the nozzle of the spray gun. Underneath the cap near the lower edge, a hole 1/16" diameter, provided an outlet for excess oil drippings which collect in the lower end of the tube. The forward end of the tube was left open to the atmosphere to decrease the explosion danger. The velocity of air through the tube was sufficient to prevent appreciable diffusion in from the atmosphere. The chamber was rigidly supported by two pairs of 4" x 5" shelf brackets using the long side at one end and the short side at the lower end to give a slope of about 5 degrees upward.

26. Plate 1 shows the propagation chamber mounted with the spray gun (Q) held in position by means of a laboratory clamp (R). Air supply connection to the apparatus was made through rubber hose (T) and was fastened to the gun by threaded metal connectors. The oil burette (W) was connected to the spray gun with a metal fitting (U) joined to the glass by a short length of Resistoflex PVA oil-resistant tubing. The two leads designated

as S are thermocouple wires from the chromel-alumel couple and are connected directly without the use of a cold junction to a 0 - 10 millivoltmeter. A small hole placed at P released excess oil from the propagation chamber to a glass crystallizing dish placed beneath this point. A legend giving the detailed description of the apparatus shown in Plate 1 is listed in Table 1.

B. Electrical Ignition System

27. In view of the danger involved during the burning of some of these oils in oxygen-rich atmospheres it was considered advisable to strike the arc by means of a high tension spark, rather than use a manual system of arcing. In addition, the danger of electrical shock to the operator is minimized. After construction, the spark ignition method was found to give the added advantage of making possible a more uniform spacing of the electrodes. Movement of the individual electrodes to start the arc is unnecessary, and an initial adjustment is allowed to remain during several inflammability tests.

28. The system of spark ignition used in this work was essentially the same as that described by Brockman and Hockosgang with the exception that 110 volts D.C. was used for the arc instead of 250 volts D.C. The method consisted in the production of high voltage alternating current which was used to produce the igniting spark. Upon impression of the spark across the gap, the 110 volt D. C. arc ignited and continued to burn. Since it was only necessary for a momentary spark to be used, a button type contact switch was used and upon release of pressure the A. C. circuit was broken. Plate 5 shows the electrical circuit as used with the apparatus herein described. The parts are listed as follows:

- S-1 Double pole line switch
- S-2 Relay, 1.5 amp., 110 V.
- S-3 Push button switch
- M-1 Electric clock, synchronous motor, giving time to .01 sec.
- R-1 Bank of cone heaters, total resistance 5 ohms.
- R-2 Resistor, 1 megohm, 1 watt
- L-1 Filter choke, 112 turns, 5 in. diameter, 13 turns per inch, 16 gauge cotton-covered copper wire on an insulated form.
- C-1 Condenser 0.03 mfd. made from windowglass and copper foil, 39 electrodes each 6 x 7.5 inches
- C-2 Condenser 0.002 mfd, 10,000 volts D. C., mica
- G-1 Analytical gap
- G-2 Auxiliary gap
- T-1 Air core transformer $L_1 = 2$ turns, $L_2 = 20$ turns
- T-2 Neon transformer 110/7500 volts

29. Only slight deviations in the original description were made; a protective resistance was inserted in the circuit of the first transformer, an electric timer was added and some minor variations were made in the smaller parts due to scarcities.

30. The ignition system as described was found to operate well and greatly facilitated rapid determination. At first carbon electrodes were used, but were found to burn and thus change the spacing rapidly. As a substitute, copper electrodes were used and were found to remain at the same distance for many individual determinations, since no appreciable burning of the metal took place.

31. The spray gun used was a Paasche air brush, mfr., model ML-8, (Plate 2) of the type used by artists for creative art and photograph retouching. It was selected for use with the propagation chamber for the following reason: (1) It gave satisfactory results when used on low gas pressures (25 to 30 psi); (2) It had a short liquid passage to the tip; (3) The rate of liquid flow required for the production of a finely atomized spray was small and was more in keeping with the amount of oxygen contained in the propagation chamber; (4) The tip of the air brush was particularly adaptable to the chamber used since the orifice tip could be easily fitted to the brass cap, allowing no atmospheric air to enter and dilute or enrich the carefully regulated gas mixture. Artists' brushes, of different design, were available that equalled the Paasche in spraying ability, but their modification for use in the equipment at hand required adapters of difficult mechanical design. The Paasche brush had controls for air and oil flows but these were set to operate continually at maximum values. The silver needle was further adjusted at M-141 so that the flow, when maintained at the minimum liquid flow adjustment, gave a spray pattern which was conical in shape reaching a diameter of 3" at a point 12 inches from the tip. The spray gun was mounted as shown in Plate 1 with the longitudinal axis in line with the mid-point of the spark gap, thus the spray was directed uniformly with a maximum of oil mist reaching the burning electrodes. It was observed experimentally that this spray gun adjustment produced the most inflammable spray of all those positions tested.

C. Gas Measurement and Control Devices

32. The sole inlet for gas in the propagation chamber is through the tip of the spray gun by way of the rubber tube and metal connector. Although the end of the chamber is open, no appreciable diffusion or dilution with atmospheric air occurs since the air flow through the tube is in excess of 10 liters per min. Some mixing and diffusion undoubtedly occur at the extreme end of the tube as the spray emerges, but none has been noted in the cylinder between the electrodes and the spray gun.

for the determination of the inflammability of an oil. The reservoir burette (W) was filled with the liquid under study. The finger lever on the air brush (M-36) was fastened tightly in the "on" position. Gas tank pressures were then adjusted at the reduction valves for operation at about 25 psi. With the remainder of the system open, the Hoke needle valves at the base of the rotameter tubes (F and BB) were adjusted to produce the desired oxygen-nitrogen mixture. After all adjustments had been made the gas was allowed to flow for about 45 seconds to settle down to a constant flow, and then the flowmeter readings were checked. The burette stopcock was then opened allowing the liquid to spray. After continuously spraying for fifteen seconds, switch S-1 (Plate 5) was connected and the A.C. push button was momentarily pressed. Simultaneously with the striking of the arc, an electric clock was started and the arc was allowed to burn for five seconds, after which time switch S-1 was opened. When the flame propagated from the arc back to the gun, it passed the thermocouple and thus actuated the millivoltmeter. When the millivoltmeter registered 1 mv. or more within five seconds, the test was considered positive, and the oxygen concentration was lowered until the thermocouple potential was less than one millivolt. In this manner the oxygen percentage necessary for combustion could usually be ascertained to $\pm 1\%$ in the lower oxygen percentages and $\pm 2\%$ in the higher ranges. The observation was usually accompanied by a muffled report, the intensity of which increased as the critical amount of oxygen was exceeded.

40. Between each inflammability test, the chamber was allowed to sweep out from one to five minutes depending on the type of oil under study. The electrodes were removed, cleaned and respaced at least once every ten arc discharges. It was found necessary, however, to clean them more often (in the extreme case every time) when consistent results could not be obtained otherwise. This precaution was especially necessary when testing oils which charred readily on the hot electrodes. Benzene was particularly troublesome in this regard.

41. To conserve the pure gases used in the determination, air from the laboratory line was used for sweeping out the chamber during the one to five minutes between determinations. A reduction valve similar to that used on the gas cylinders in Plate 1 was used on the air line to give 25 psi pressure and a flow of ten liters per minute. It was necessary to place a filter of glass wool in the air line to protect the valve and the air brush from contamination due to oil or dust in the system.

42. After a series of inflammability tests on a given oil, it was found necessary to completely clean the apparatus before making tests on a second liquid. The burette, spray gun, flexible connectors and propagation chamber were cleaned by running about twenty ml. of benzene through the assembled equipment. It was often found necessary to completely clean the combustion tube by removing CaN from the propagation chamber and thoroughly swabbing

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C. Gas Measurement and Control Devices

32. The sole inlet for gas in the propagation chamber is through the tip of the spray gun by way of the rubber tube and metal connector. Although the end of the chamber is open, no appreciable diffusion or dilution with atmospheric air occurs since the air flow through the tube is in excess of 10 liters per min. Some mixing and diffusion undoubtedly occur at the extreme end of the tube as the spray emerges, but none has been noted in the cylinder between the electrodes and the spray gun.

33. Initial experiments were conducted with the spray equipment (Plate 1) using air as the propagation atmosphere. Results obtained showed some liquids to be inflammable while others were not, thus dividing the oils into two well-defined classes. No differentiation or evaluation could be made of oils in either class to compare them with other oils in the same class. To make such evaluation possible a mixture of oxygen and nitrogen was used in the cylindrical chamber. By starting with 100% nitrogen and increasing the oxygen concentration in a series of successive experiments, a percentage was reached where the oil in question propagated the flame just enough to give a positive test. By comparing this critical oxygen percentage, a relative inflammability evaluation of the oils with respect to one another was obtained.

34. To obtain this variable gas mixture the apparatus shown in Plate 3 was devised. Oxygen and nitrogen were obtained as compressed gases (2000 psi) in standard cylinders (220 cu.ft. capacity). The tanks were capped with pressure regulating reduction valves, and rubber tubes carried the gases to the metering and mixing apparatus. Rates of flow were measured on glass type Rotameters (Y for oxygen) and (I for nitrogen), consisting of tapered glass tubes containing a carefully constructed float. The float was of such diameter that binding occurred at the lower part of the tapered tube while it allowed more gas to flow as it rose up the tube. The individual gases were then piped to a mixing chamber R where a series of concentric brass cylinders (Plate 4) provided the turbulence necessary to assure proper mixing of the gases. It was necessary to place a pressure gauge at this point in the system to allow the operator to maintain a constant pressure on the spray gun as well as to regulate the rate of flow. Pipe connection was then made to a "T" which carried the spray gun gas connector. An independent connection (Z) with the laboratory air line was made at T so that the exhaust gases produced in each run could be swept out with air, saving the supply of purified gases for actual determinations. Gas flows were controlled by the use of Hoke needle valves as illustrated.

35. The Rotameter type of flowmeter was selected because of its advantages in this type of operation. Ease of adjustment and of reading is of prime importance in this determination and the Rotameter is much better than orifice type meters for this use. In addition Rotameters can withstand the pressures used with safety whereas many other commercially available flowmeters would not have sufficient mechanical strength. The oxygen flowmeter was constructed with a hollow catalin float designed to measure 400 to 8000 ml. per minute over the entire range. The nitrogen flowmeter contained a hollow dural float designed to measure 600 to 12000 ml. per minute over the entire range. A rate of 10 l. per min. at the pressure of 25 psi was selected as the operating flow producing the optimum spray with the oils under study. The flowmeters were carefully

calibrated and a scale was placed on each tube reading directly in percent oxygen in the mixture whose total flow was 10 l. per min. The flow of the gases was regulated at the cylinders (B and FF) and at the Hoke needle valves (F and BB) so that both scales read the desired percentage at 25 psi pressure on gauge N. Flow to the spray gun could be conveniently turned off at S without changing the percentage mixture settings on the other regulating devices.

36. The pressures used in the system, being too great for the laboratory rubber tubing, required the use of a cloth braided, small bore rubber tubing and special threaded metal connectors to fasten the tubing to the gas sources and outlet systems. A legend giving a detailed description of the apparatus shown in Plate 3 is listed in Table 2.

D. Oil Metering Device

37. When a constant pressure and a fixed flow rate are used on the spray gun, the amount of oil passing through the gun and the nature of the spray are dependent upon the amount of liquid which is drawn through the orifice of the spray gun tip by the aspirator effect. Since the oils must pass through a fixed orifice, this quantity in turn will depend upon the viscosity of the oil under consideration. With constant air supply it was noted experimentally that the rate of flow of liquid to the gun varied with the viscosity from 0.5 cc per min. for a heavy mineral oil (SAE #60) to more than 15 cc. per min. for liquids as thin as benzene. This wide range of fuel concentration in the chamber caused some gross discrepancies in inflammability evaluation for many compounds.

38. Apparatus was constructed as shown in Plate 6 for the injection of a fixed quantity of oil per minute independent of the aspirator effect of the spray gun and of the gravitational effect of the liquid column in the burette. The apparatus consisted of a 100 ml. medicinal ground glass syringe mounted horizontally. A hole was placed in the back end of the plunger so that a machined brass nut (1/4" - 20 threads per inch) could be inserted within a closely fitting rubber tube. A slow-speed reversing electrical motor (7 RPM) was placed with its shaft in line with the plunger. A long threaded screw, attached with a connector, was placed inside the brass nut so that rotation of the nut or shaft caused the plunger to move in or out as desired. Connection was made from the end of the syringe to a three-way stopcock connected in turn to the filling burette or the spray gun. The "T" type three-way stopcock was inserted three dimensionally in Plate 1 at a point U in the connector tube.

(b) Experimental Procedure:

39. With the gas measurement and control equipment (Plate 3) connected to the propagation chamber, the apparatus was ready

for the determination of the inflammability of an oil. The reservoir burette (W) was filled with the liquid under study. The finger lever on the air brush (M-36) was fastened tightly in the "on" position. Gas tank pressures were then adjusted at the reduction valves for operation at about 25 psi. With the remainder of the system open, the Hoke needle valves at the base of the rotameter tubes (F and BB) were adjusted to produce the desired oxygen-nitrogen mixture. After all adjustments had been made the gas was allowed to flow for about 45 seconds to settle down to a constant flow, and then the flowmeter readings were checked. The burette stopcock was then opened allowing the liquid to spray. After continuously spraying for fifteen seconds, switch S-1 (Plate 5) was connected and the A.C. push button was momentarily pressed. Simultaneously with the striking of the arc, an electric clock was started and the arc was allowed to burn for five seconds, after which time switch S-1 was opened. When the flame propagated from the arc back to the gun, it passed the thermocouple and thus actuated the millivoltmeter. When the millivoltmeter registered 1 mv. or more within five seconds, the test was considered positive, and the oxygen concentration was lowered until the thermocouple potential was less than one millivolt. In this manner the oxygen percentage necessary for combustion could usually be ascertained to $\pm 1\%$ in the lower oxygen percentages and $\pm 2\%$ in the higher ranges. The observation was usually accompanied by a muffled report, the intensity of which increased as the critical amount of oxygen was exceeded.

40. Between each inflammability test, the chamber was allowed to sweep out from one to five minutes depending on the type of oil under study. The electrodes were removed, cleaned and respaced at least once every ten arc discharges. It was found necessary, however, to clean them more often (in the extreme case every time) when consistent results could not be obtained otherwise. This precaution was especially necessary when testing oils which charred readily on the hot electrodes. Benzene was particularly troublesome in this regard.

41. To conserve the pure gases used in the determination, air from the laboratory line was used for sweeping out the chamber during the one to five minutes between determinations. A reduction valve similar to that used on the gas cylinders in Plate 1 was used on the air line to give 25 psi pressure and a flow of ten liters per minute. It was necessary to place a filter of glass wool in the air line to protect the valve and the air brush from contamination due to oil or dust in the system.

42. After a series of inflammability tests on a given oil, it was found necessary to completely clean the apparatus before making tests on a second liquid. The burette, spray gun, flexible connectors and propagation chamber were cleaned by running about twenty ml. of benzene through the assembled equipment. It was often found necessary to completely clean the combustion tube by removing cap N from the propagation chamber and thoroughly swabbing

the interior with cloths previously dipped in benzene. After re-assembly, the apparatus was blown for thirty minutes with air to remove all traces of benzene used in removing the previous oil. The electrodes were cleaned by removal from the chamber, washing with benzene and polishing to a shiny metal surface with emery paper.

43. With continued use the ends of the electrodes may become pitted and it was necessary to file them after the cleaning operation. When reassembling, the electrodes were properly spaced by screwing them in until electrical contact was made (as observed on a meter or on the electrical timing clock), then the electrodes were turned out to the desired arc spacing (twenty revolutions to the inch). A spacing of 0.10" (two turns) has been found to give satisfactory operation of the chamber so this value was used as a convenient constant gap distance.

44. Operation of the inflammability apparatus utilizing the oil metering device was done in much the same manner as described above. The metering tube was connected into the spray gun inlet (U in Plate 1). The glass syringe was then filled by reversing the motor and drawing the liquid in from the filled burette. After filling the apparatus, the three-way stopcock was turned to allow the liquid to go directly through to a spray gun. In making the inflammability test, the gas mixture was adjusted as before, and allowed to operate for 45 seconds. The motor was then started and the liquid was forced through the orifice tip at the desired rate for fifteen seconds before striking the arc. The oxygen-nitrogen ratio was then varied until the critical value which just propagates the spray back to the orifice under those conditions was reached.

DISCUSSION OF RESULTS

(a) Data Obtained

45. The hydraulic oils which were available in the laboratory were tested in the flame propagation chamber and the results of these tests are reported in Table 3. These results are not intended to be comprehensive, but are merely representative values obtained on several oils and liquids readily available. Studies were made to evaluate the present method.

46. It was noted that the petroleum base oils now in use in naval equipment form inflammable mixtures with as little as 12% oxygen. This is true of the three tested.

O.S. - 1113	Hydraulic Gear Oil, Flash Point 310°F
O.S. - 2943	Hydraulic Gear Oil, (Flat Viscosity) Flash Point 230°F
AN-VVO-366	Aircraft Hydraulic Oil, Flash Point 200°F

The oils are of the low viscosity type and are made from low boiling petroleum base stocks and are thickened by the addition of polymer additives to give the desired viscosity-temperature curves. It is noted that the flash points of these oils are different, but in spite of this difference, no change in the oxygen percentage needed for propagation of the spray was observed.

47. Benzene, when sprayed through the chamber, gave a violent combustion, occasionally with explosive violence, when only 12% oxygen was used. It was necessary, however, to restrict the flow to about 3 cc per minute to obtain this inflammability figure. With high rates of liquid flow, especially the high rate of 15 ml. per minute obtained in an open system, the mixture would not ignite until the oxygen percentage had been increased to over 30%. It has been concluded that the deviation here was caused by an exceedingly rich benzene-air mixture and a shorting of the arc caused by the formation of pyrolytic carbon on the copper electrodes. When this deposit was shaken loose by application of a potential across the electrodes, a violent explosion usually resulted.

48. Cetane (n-hexadecane) likewise gave an inflammable mixture with 12% oxygen even when allowed to flow freely into the spray gun. The cetane used for these tests was about 95% pure and was the commercially available Dupont product used as the diesel fuel standard. The same oxygen percentage was thus obtained on these two liquids in spite of the vastly different results which have been obtained in other inflammability tests.

	<u>Spontaneous Ignition Temp.</u>	<u>Flash Point</u>	<u>Inflammability Oxygen Percentage</u>
Benzene	1250°F	13°F	12%
Cetane	459°F	230°F	12%

It is noted that when a comparison of the inflammability of benzene and cetane is made, benzene is the more inflammable at room temperature in the presence of a free flame while cetane is much more inflammable when the hazard is spontaneous ignition of the oil in a container or on a hot surface. Equal inflammability of the two is observed when sprayed from an orifice onto a hot surface in the presence of a flame.

49. Navy symbol #2135 light lubricating oil gave a spray which propagated flame in the presence of 34% oxygen, but would not give a positive value below this percentage. This oil is

composed of a single petroleum fraction with no viscosity modifier present and corresponds to an SAE 20-30 motor oil. While the viscosity of the 2135 oil was larger than that of some of the hydraulic oils tested (67 c.s. at 100°F as compared with 46.8 c.s. for O.S.-1113 oil), the inflammability was less. Since the O.S.-1113 oil is made from a light base stock thickened with a polybutene polymer, it appears from those results that the inflammability of an oil is more dependent upon the nature of the base stock than upon the viscosity of the finally thickened oil.

50. It is indicated by these inflammability results on petroleum products that the inflammability of the spray of many base stocks is approximately the same. This is presumably true for any oil made from a base stock below the fuel oil or cetane range and possibly for some oils whose base stock is a higher petroleum fraction.

51. Two chlorinated hydrocarbon mixtures were used for tests in the flame propagation chamber. These mixtures were oils made by the Standard Oil Development Company of New Jersey under a naval research contract and were designated as Controller Oil P.S. and Controller Oil CC. Controller Oil PS required 64% oxygen before the spray could be made to strike back and actuate the millivoltmeter attached to the thermocouple to one millivolt. Even at oxygen values higher than 64% the violence of the burning was much less than that of the petroleum oils when fired at 12% oxygen mixture. This inflammability value decreased to 59% when the rate of liquid flow was changed to 3 cc. per minute. Controller oil CC was definitely more inflammable than controller oil PS requiring 34-38% oxygen to produce a mixture propagating a flame. This value was found to rise to 55% when the liquid flow rate was changed from free flow to 1.5 cc. per minute.

52. It was observed when testing these two chlorinated hydrocarbon mixtures that a long sweeping period with air was necessary after each test to remove products formed during the combustion. Running tests in rapid succession using controller oil PS caused an increase in the oxygen percentage necessary to cause spray combustion. Controller oil CC showed the opposite behavior, i.e. when a series of runs was made without sweeping out the air thoroughly in between each run, the percentage oxygen necessary for flame propagation under the conditions specified was decreased.

53. This behavior of the chlorinated hydrocarbon mixtures is interpreted as follows: Controller oil PS after combustion left some volatile halogenated material which acted as a snuffer and required an increased amount of oxygen to burn. Controller CC on the other hand produced the highly inflammable propylene by a scission of the aliphatic side chain. The volatile inflammable gas then remained in the propagation tube along with other products of the

combustion dissolved in the oil on the side walls and made the next test require less oxygen for combustion.

54. The relatively low percentage of oxygen required for the flame propagation of petroleum oils, in contrast with the high percentage mixture required for the chlorinated hydrocarbon oils is in agreement with other observed inflammability properties of these oils.

55. Petroleum base oils when sprayed in the apparatus caused a rapid propagation back to the orifice when the critical oxygen-nitrogen ratio was exceeded only slightly. With these oils, the "strike-back" made such a characteristic sound that the question of a positive or negative test was easily answered and the use of the millivoltmeter and thermocouple was hardly necessary. When chlorinated hydrocarbons were used the determination of "strike-back" was not so definite and recourse to the thermocouple and millivoltmeter was necessary. A voltage rise of 1.0 mv. was selected as the point at which enough heat was produced by the backward movement of the flame to assume a positive test.

56. The cleaning of the electrodes was essential when values were to be checked against previous ones or against one another. The aromatic hydrocarbon compounds tested showed a profound tendency to char and leave a deposit on the hot copper electrodes. This deposit continued to build up until an actual shorting of the arc current across the electrode occurred. The viscous liquids deposited this oily layer on the electrode while some of the pure compounds, i.e. benzene, left a solid carbon deposit. It was found necessary in all of these tests to clean the electrodes and as a maintenance operation the electrodes were removed, cleaned, polished, and replaced after about ten inflammability trials.

57. It should be realized in interpreting the data presented in Table 3 that the values reported are the minimum ones obtained in orienting experiments. Occasionally, an oil gave a negative test at the reported inflammability percentage and this has even occurred at higher oxygen percentages. However, when duplicate determinations were made the lower figures reported could be checked and easily repeated.

(b) Comparison of the Present Method with other Inflammability Tests

A. Flash Point

58. The flash point of a liquid may be considered as the temperature to which the liquid must be heated in order to give off sufficient vapor to form an inflammable mixture with air.

Flash point tests were originally employed, and have been used extensively, to indicate the fire hazard of a large variety of petroleum products. Their use has been extended and they are frequently employed to give information which does not relate to fire hazard. For example, the flash point is sometimes used as a measurement of the volatility of the liquid in question, this being an accepted practice in classifying gasolines.

59. Flash points, determined on petroleum products show a wide range of results. The more volatile gasolines and naphthas flash at temperatures considerably below 0°F. Petroleum ether solvents and some special naphthas have closed cup flash points between 80° and 110°F while the range for the kerosenes is usually from 100° to 160°F. Gas oils and fuel oils generally flash between 150° and 300°F and the majority of lubricating oils have flash points between 275° and 650°F.

60. The flash point of the lower boiling petroleum fractions is of definite value in evaluating the potential fire hazard under conditions where a fire may be started by exposing the vapor, at the correct temperature, with the proper amount of air in the presence of a flame. However, for the hydraulic and lubricating oils, which operate at temperatures lower than that measured as the flash point, the use of this determination as a measurement of the fire hazard is of little value.

61. To illustrate this point, a kerosene having a flash point of 100°F can present a fire hazard in the presence of an open flame when operated in a system which can attain a temperature over 100°F. However, attempts to evaluate the fire hazards of two kerosenes having flash points of 140°F and 160°F for this system operating at a maximum temperature of 100°F gives no information that would show one liquid to be more of a fire hazard than the other. The same analogy can be drawn from the consideration of hydraulic oil of 230°F flash point when used in a system operating at a maximum temperature of 180°F in naval equipment. The evaluation of the fire hazard by means of the flash point is only of value in showing that the flash point is higher than the ordinary operating conditions.

62. This limitation of the flash point test for evaluation of fire hazard has been recognized by the ASTM Committee on Petroleum Products and Lubricants.⁹ The view taken in this report is corroborated by their statement: -- "For a large number of petroleum products, namely the class of lubricating oils, flash point is not determined as an index of fire hazard, but rather for purposes of identification and classification. The interpretation

of figures is not simple and as a general rule it may be said that the flash point of a lubricating oil bears no direct relation to its usefulness. Existing methods for determining the actual usefulness or value in lubricating oils are not satisfactory and in general it may be stated that the flash points in lubricating oil specifications is actually a makeshift to compensate for a large factor of ignorance."

63. Extrapolation of the data on an oil whose inflammability is based on a flash point determination should not be made unless the conditions under which the extrapolated comparison is made are comparable to those under which the flash point was determined. The spontaneous ignition temperature has quite obviously no relation to the flash point, and it is considered equally erroneous to estimate the inflammability toward incendiary missiles or toward the inflammability resulting when a hot exhaust pipe has oil sprayed upon it by a measurement of the flash point of an oil.

B. Fire Point

64. It was observed of petroleum hydraulic fluids that the fire point of the oil occurs only slightly above the flash point, indicating that in the oil flashes occurring in practice, burning readily ensued. A representative O.S.-1113 petroleum oil, which flashed at 310°F had a fire point of 340°F. Once the oil began to burn at this temperature no further application of flame was necessary for complete combustion of the sample.

65. Many halogenated compounds have been observed to fire at a temperature appreciably higher than the flash point. A rough correlation has been observed between compounds having a large difference between flash and fire points and the results of their spray inflammabilities. Compounds having a small difference between flash and fire points usually fired with 12% oxygen in the flame propagation chamber while compounds having a large difference (chlorinated compounds) required oxygen percentages in the range of 45-65 percent. This correlation was observed in a few cases and is being studied further. While no exceptions were noted, too few cases have yet been observed to permit a reliable generalization.

C. Spontaneous Ignition Temperature

66. The inflammabilities of several organic compounds and numerous hydraulic oil mixtures have been studied by measurements of the Spontaneous Ignition Temperatures. There appears to be no simple correlation between flash point and Spontaneous Ignition Temperature since many diametrically opposite values have been obtained, even on petroleum products. For example, benzene has a low flash point of 13°F and cetane has the high

flash point of 230°F, but the Spontaneous Ignition Temperatures of benzene and cetane are 1250°F and 459°F respectively.

67. The spray inflammability of these two liquids is equal since each material ignites and burns readily when sprayed with 12% oxygen. The Spontaneous Ignition Temperature is concluded to be of no more value in predicting the spray or incendiary inflammability of an oil in question than a similar extrapolation of the flash point data to predict the inflammability under these two circumstances.

D. Incendiary Firing Tests

68. A series of firing tests using 50 cal. M-1 incendiary bullets is being conducted in cooperation with the Bureau of Ordnance to determine the inflammability of present and proposed hydraulic oils. Preliminary data obtained indicates that the incendiary inflammability of hydraulic oils is not directly related to their flash points nor their Spontaneous Ignition Temperature. A fair correlation has been obtained between these incendiary firing tests and the laboratory flame propagation method already described here. These tests are still in progress and will be reported when they are completed.

CONCLUSIONS AND RECOMMENDATIONS

69. On the basis of the experimental work and data presented in this report, it is concluded that the inflammability of an oil cannot be evaluated by any one type of inflammability test method. Many times in actual practice, one specific kind of inflammability is a hazard to naval equipment and personnel, and one properly chosen test may be sufficient to measure the inflammability toward this single hazard. However, when the oil in question becomes a danger due to other potentially hazardous conditions, further tests are necessary for proper evaluation each one being specially designed to simulate the actual circumstance. The general fire hazard of an oil under all conditions can then be made only by a measurement of the inflammability under several different circumstances. The methods now available of interest for naval fire hazards are:

- (a) Flash point
- (b) Fire point
- (c) Spontaneous Ignition Temperature
- (d) Flame propagation of the spray
- (e) Incendiary firing tests

70. It is recommended that proposed oils (and their base stocks) be tested by application of the methods mentioned above.

Data obtained from the tests will indicate the degree of non-inflammability to be expected under the several actual hazardous circumstances. This does not imply that all these tests should be applied as a routine procedure, but that the tests be made on the base stocks of the hydraulic oils of different types to indicate the inflammability of fluids made from this type of liquid. Hydraulic fluids can then be selected which have a wide limit of safety under the hazardous conditions to be expected.

71. It is recommended that the method herein described be further applied to the problem of developing liquids to be used as non-inflammable hydraulic fluid base stocks. It is further recommended that the laboratory results so obtained be checked with actual controlled scale fires in naval equipment to ascertain the value of the laboratory methods as inflammability tests for naval hydraulic oils.

BIBLIOGRAPHY

- (1) A.S.T.M. Standards on Petroleum Products and Lubricants, Committee D-2, p. 51 (1942).
- (2) Sortman, Beatty and Heron, Symposium on Petroleum Chemistry, Petroleum Div. A.C.S. Detroit meeting. Sept., 1940, p. 101.
- (3) Confidential Report #ENG-M-51/4529-1, Materiel Center, Army Air Forces, Wright Field, Dayton, Ohio, Jan. 1943.
- (4) Loland and Stanley, Confidential Report #W-1735-4911. Study of Hydraulic Oils, Esso Laboratories, S.O.D. Company (New Jersey) Nov., 1942, p. 55.
- (5) Wiezevick, Whiteley and Turner, Ind. Eng. Chem. 27, 152 (1935).
- (6) Nuckolls, Miscellaneous Hazard Report #2375, Underwriters Laboratories, Nov., 1933, p. 103.
- (7) Fenske, Confidential Report #1369, Div. 11, N.D.R.C. of the O.S.R.D. Hydraulic and Recoil Oils, April, 1943, p. 22.
- (8) Brockman and Hochesgang, Ind. & Eng. Chem. Anal., Ed. 14, 796 (1942).
- (9) Significance of Tests of Petroleum Products. Report of A.S.T.M. Committee D-2, p. 53 (1934).

TABLE 1

Legend of Apparatus Described in Plate 1

- A. Water outlet, brass tube, O.D. $5/16$ ", I.D. $1/4$ ", length $1-1/4$ "
- B. Chamber wall, brass tube, O.D. 3", I.D. $2-7/8$ ", length 15"
- C. Chamber wall, brass tube, O.D. $2-1/2$ ", I.D. $2-3/8$ ", length $13-3/4$ "
- D. Brass collar to hold "E", O.D. $3/4$ ", I.D. $5/8$ ", length $1/2$ ".
- E. Porcelain insulator, O.D. $9/16$ ", I.D. $5/16$ ", length $1-1/2$ "
- F. Copper electrode, D. $5/16$ ", length 6"
- G. Brass nut, $1/2$ x 20, soldered to "Z" as part of electrode support
- H. Brass electrode holder, 2" section of $1/2$ " rod, 20 threads to inch
- I. Brass nut, $1/2$ x 20, soldered to "H"
- J. Cut away view of jacketed chamber
- K. Chromel-alumel thermocouple junction
- L. Porcelain protector for thermocouple leads.
- M. Water inlet, brass tube, O.D. $5/16$ ", I.D. $1/4$ ", length $1-1/4$ "
- N. Brass cap, O.D. $2-7/8$ ", I.D. $2-3/4$ ", length 2", $.235$ " hole $3/4$ " from bottom for spray gun, bushing $1/2$ " from top for thermocouple
- O. Handle for cap, brass rod, D. $3/16$ ", length 6"
- P. Drip hole for waste oil, diameter $1/16$ "
- Q. Spray gun, artists air brush, Paasche type ML-8, self centering silver tip enlarged to 0.71 mm.
- R. Burette clamp
- S. Thermocouple leads, to millivoltmeter
- T. Rubber hose, $1/8$ " x $1/16$ ", braided cloth covered
- U. Metal connector, spray gun part
- V. Resistoflex PVA connector
- W. Burette 25 ml.
- X. Shelf bracket, 4" x 5"
- Y. Brass plate, 1" x $1-1/2$ " x $1/4$ ", threaded to hold "Z"
- Z. Brass rod, $1/4$ " x 4", threaded to "Y", soldered to "G"

TABLE 2

Legend of Apparatus Described in Plate 3

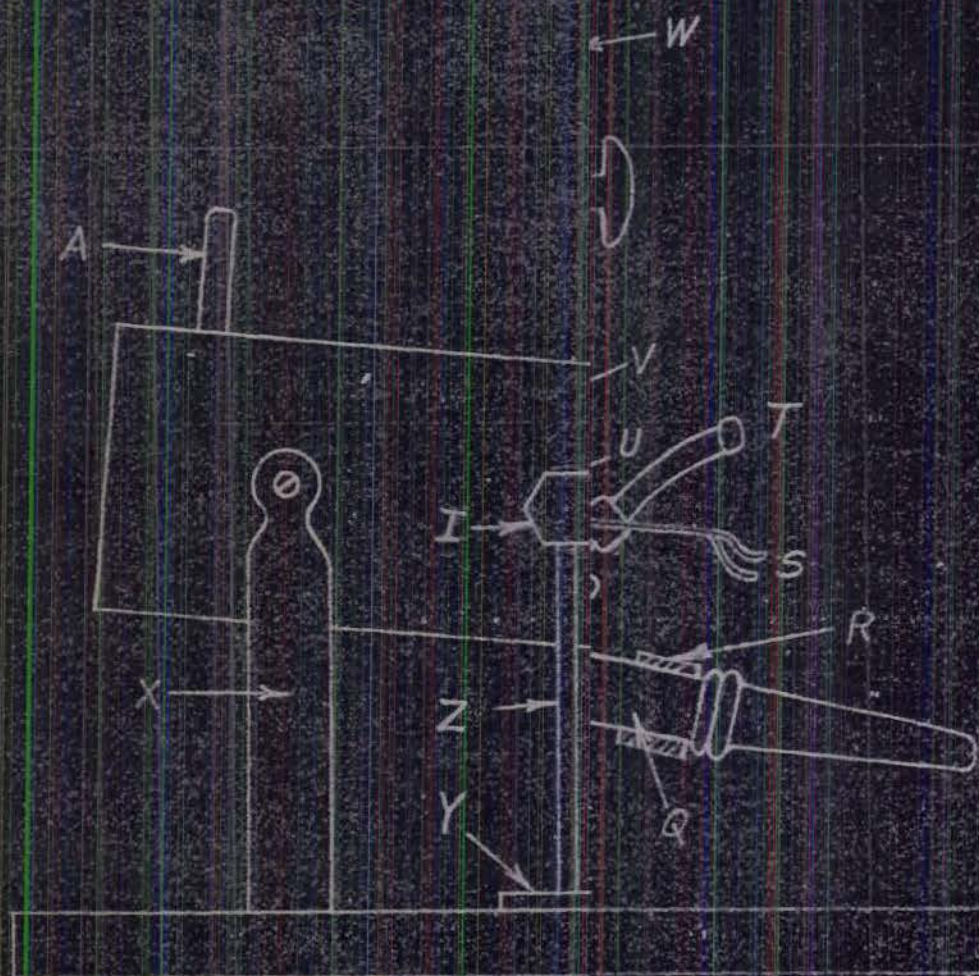
- A. Nitrogen cylinder, 220 cu. ft.
- B. (FF) Pressure regulator
- C. (F,S,U., BB, EE) Hoke needle valve, 1/4"
- D. (H, Z, DD) Rubber hose 1/8 x 1/16, braided cloth covered, fitted with couplings Paasche ports nos. HL240B and HL24S
- E. (V, CC) Elbow pipe fitting, 3/8"
- G. (J, X, AA) Rubber connector 3/8" x 1/8" x 3"
- I. Rotameter type 1-A, with Dural float, Fischer and Porter Co.
- K. (P, Q, W) Elbow pipe fitting 1/4"
- L. (O) Tee pipe fitting, 1/4"
- M. Reducer, 3/8" to 1/4"
- N. Pressure gauge, 0-50 p.s.i.
- R. Mixing chamber (see plate 3-A for detail)
- T. Tee pipe fitting, 3/8"
- Y. Rotameter type 1-A with catalin float, Fischer and Porter Co.
- GG. Oxygen cylinder, 220 cu. ft.

TABLE 3

Inflammability Results on Hydraulic Oils and Fluids Tested

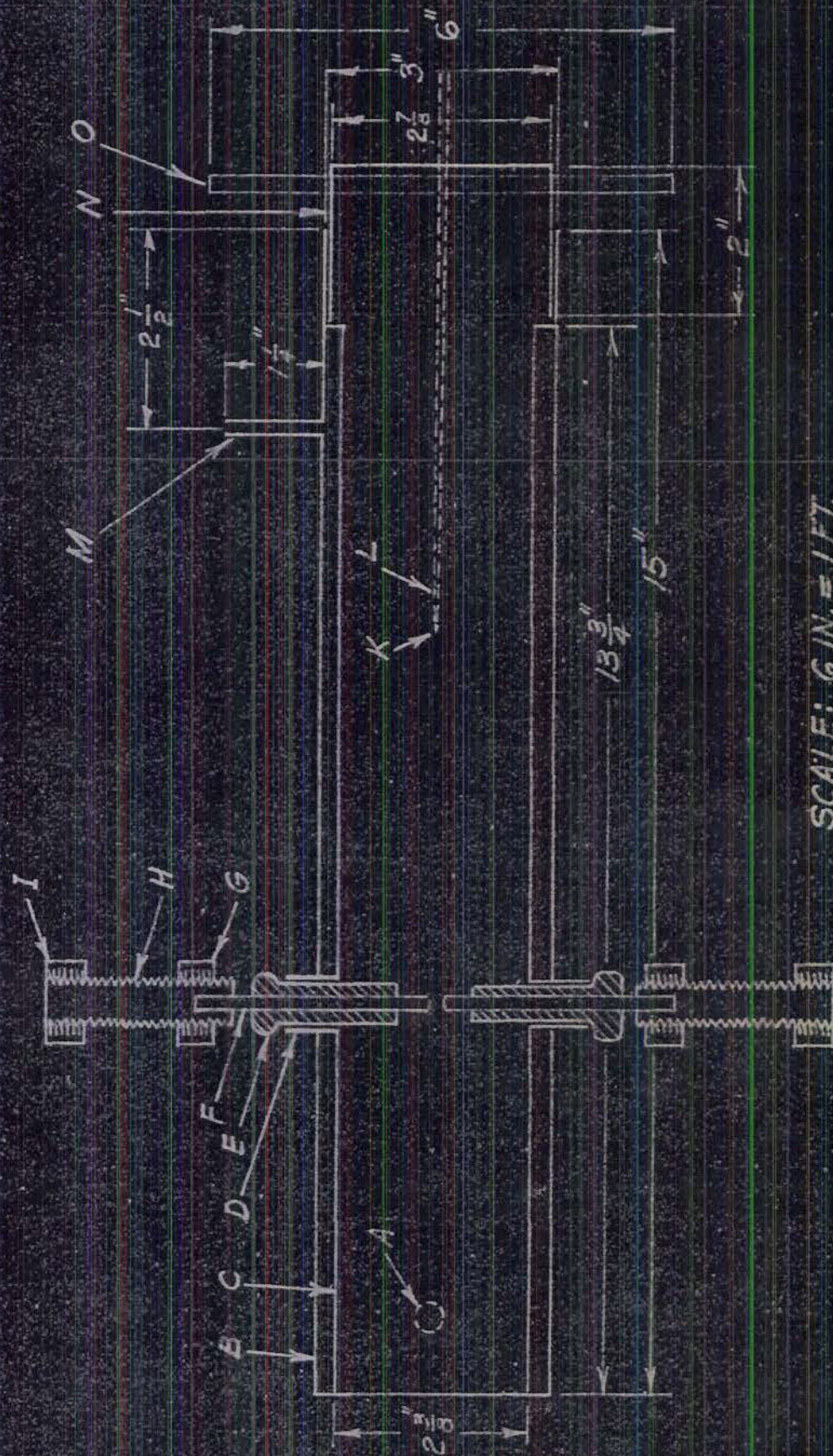
<u>Designation</u>	<u>Description</u>	<u>100°F Viscosity</u>	<u>Flash pt.</u>	<u>Liquid Flow Rate</u>	<u>Inflammability % Oxygen Required</u>
Spec. O.S.-1113	Petroleum Base Oil	41.8 c.s.	310°F	10cc. per min.	12%
"	"	"	"	*6cc	12%
"	"	"	"	5cc	12%
"	"	"	"	3cc	13%
"	"	"	"	1.8cc	14%
Spec. O.S.-2943	Petroleum Base Oil	28.5 c.s.	230°F	*10.4cc	12%
"	"	"	"	10cc	12%
"	"	"	"	5cc	12%
Spec. AN-VV0-366A	Petroleum Base Oil	24.4 c.s.	200°F	10cc	12%
"	"	"	"	*7.3cc	12%
"	"	"	"	5cc	12%
	Benzene (c.p.)	0.62 c.s.	12°F	*17.6cc	30-35%
	"	"	"	3cc	12%
	Cetane (95%)	3.1 c.s.	230°F	*12cc	12%
	"	"	"	5cc	12%
Spec. 2135	Petroleum Lu-67 bricating Oil	67 c.s.	340°F	10cc	34%
"	"	"	"	5cc	36%
"	"	"	"	*1cc	38%
Controller Oil P.S.	Chlorinated Hydrocarbon Base	41.3	none	*3cc	59%
"	"	"	"	10cc	64%
Controller Oil C.C.	Chlorinated Hydrocarbon Base	41.8	350°F	10cc	34%
"	"	"	"	*3cc	38%
"	"	"	"	1.5cc	55%

* Indicates this flow rate was observed when the liquid was allowed to flow freely from the burette through the spray gun during operation.



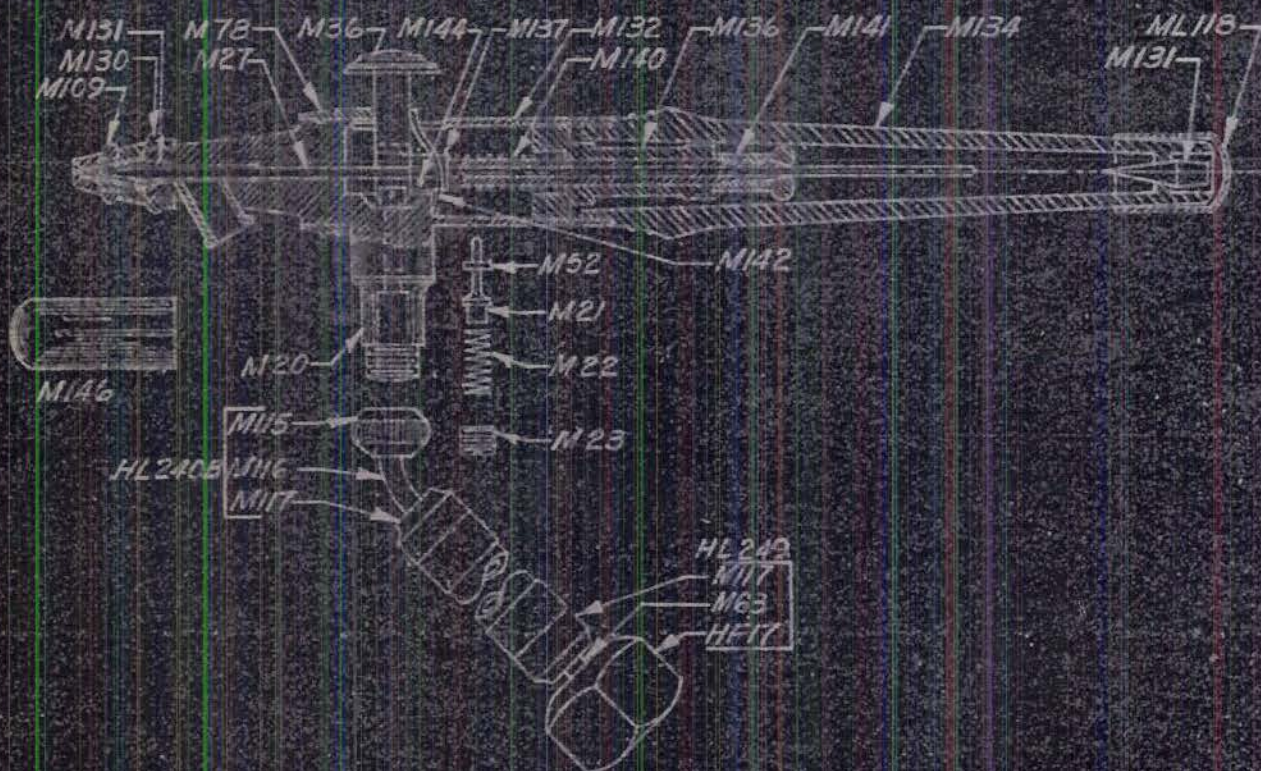
PROPAGMENT

PLATE 1

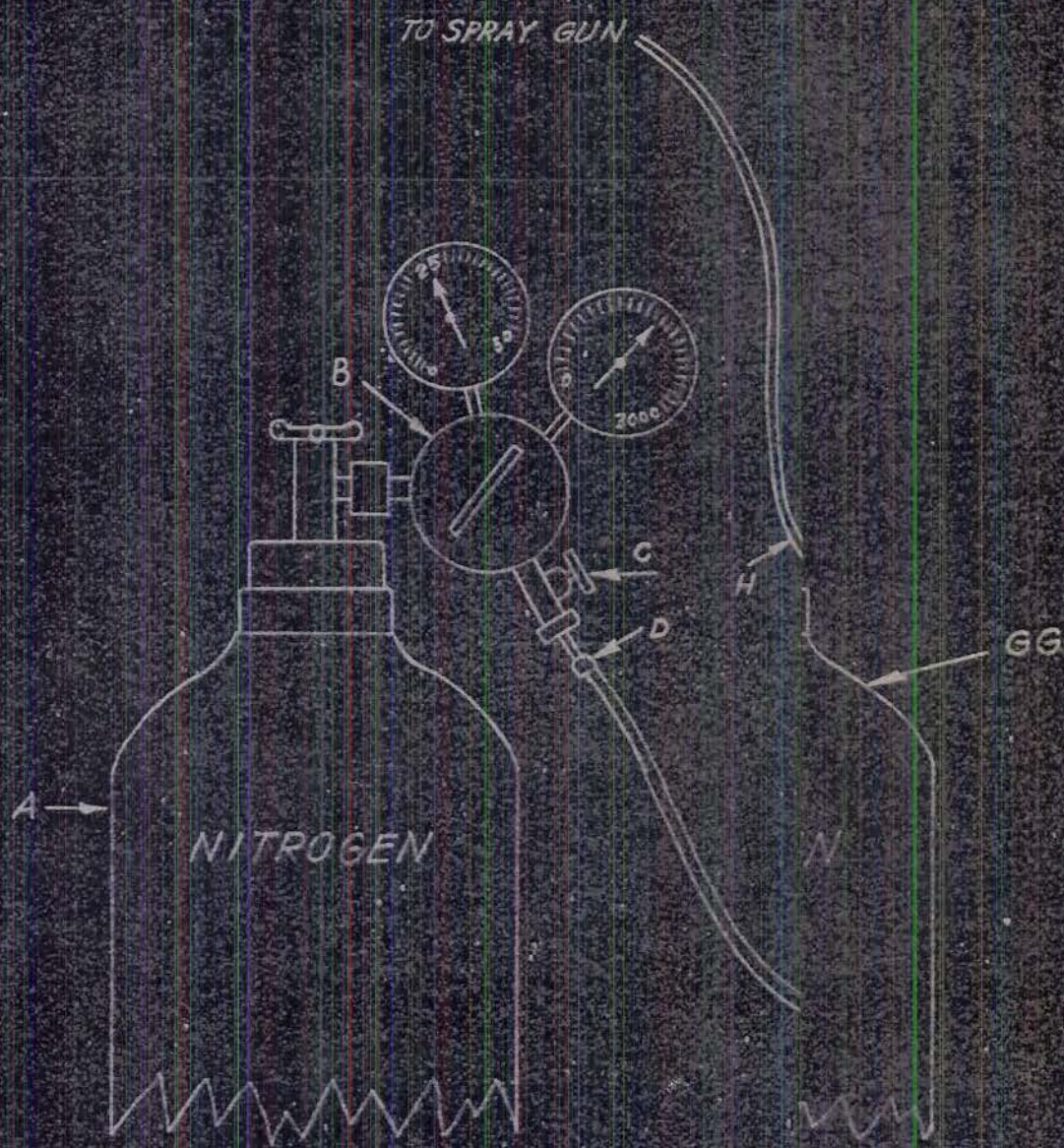


SCALE: 6 IN. = 1 FT.

PROPAGATION CHAMBER
TOP VIEW



SPRAY GUN
ACTUAL SIZE



GAS MEASURE

PLATE 3

MIXING CHAMBER DETAIL



- A-BRASS TUBE $\frac{5}{16}$ " O.D. LEAD IN FROM GAS TANKS
- B-BRASS PLATE $\frac{1}{16}$ " THICK
- C-BRASS TUBE $\frac{1}{2}$ " DIAMETER $6\frac{1}{4}$ " LONG
- D-BRASS TUBE $\frac{15}{16}$ " O.D. $\frac{13}{16}$ " I.D. $6\frac{3}{8}$ " LONG
- E-BRASS PLUG WITH $\frac{7}{16}$ " HOLE DRILLED IN END
- F-BRASS PLATE $\frac{1}{16}$ " THICK
- G-TO MAKE VALVE

