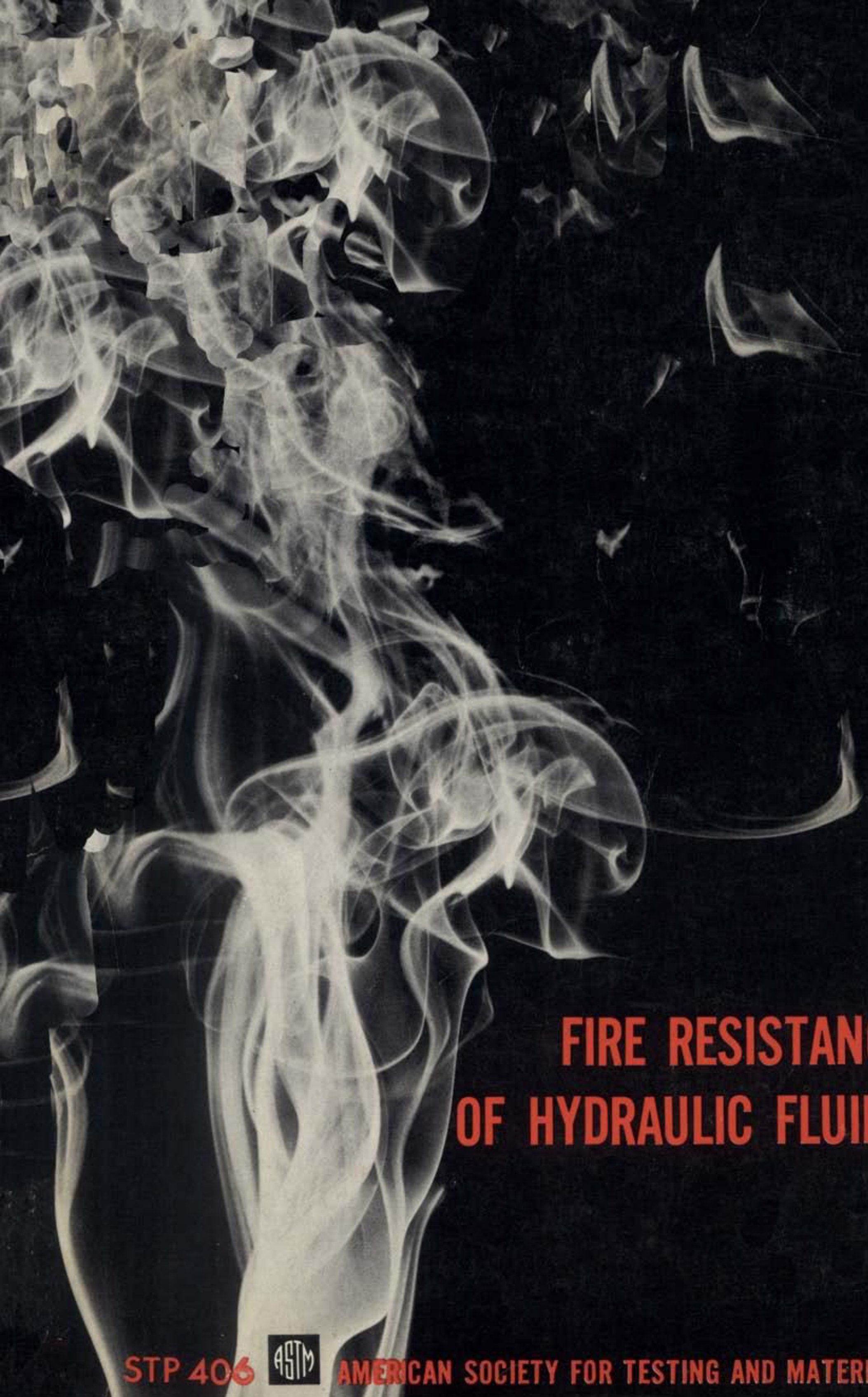

FIRE RESISTANCE OF HYDRAULIC FLUIDS

STP 406

AMERICAN SOCIETY FOR TESTING AND MATERIALS

FIRE RESISTANCE OF HYDRAULIC FLUIDS

A symposium
presented at a meeting of
Committee D-2 on Petroleum Products and Lubricants,
AMERICAN SOCIETY FOR TESTING AND MATERIALS,
and Committee A-6 on Aerospace Fluid Power Technology, SOCIETY OF AUTOMOTIVE ENGINEERS
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FOREWORD

This Symposium on Test Methods for Fire Resistance of Hydraulic Fluids and Lubricants was presented Jan. 26 and 27, 1966 at the ASTM Committee D-2 Week in New Orleans, La. The symposium was sponsored by ASTM Committee D-2 on Petroleum Products and Lubricants and SAE Committee A-6 on Aerospace Fluid Power Technology.

A. R. Lansdown, English Ministry of Aviation, was presiding officer at the first session; R. L. Leslie, Vickers, Inc., presided at the second session; and R. E. Hatton, Monsanto Co., presided at the third session. The Symposium Committee consisted of these three with Dr. Hatton serving as chairman.

During the symposium, a number of motion pictures were shown, which graphically illustrated various fire resistance test methods. These movies pointed out the great difficulty of reducing performance in fire resistance tests to words or to mathematical ratings. The papers include references to most of the movies shown. However, to complete the record, brief descriptions of two movies not so included are given here.

S. P. Polack of the U. S. Bureau of Mines presented a film which demonstrated test methods used in Bureau of Mines Schedule 30, Subchapter E, Part 35 on Fire-Resistant Hydraulic Fluids. Three tests were included: autogenous-ignition temperature test, temperature-pressure spray-ignition test, and test to determine effect of evaporation on flammability (often referred to as the pipe cleaner test). The performances of two fluids — petroleum oil and a water-oil emulsion — were shown in each of the tests. In all cases the emulsion-type fluid showed significantly better fire-resistance properties.

R. E. Hatton of Monsanto Co. presented a movie showing performance of four types of aircraft hydraulic fluids — hydrocarbon, silicate ester-type, phosphate ester, and halogenated polyaryl — in a simulated hot aircraft brake. The fluid was introduced into an aircraft brake unit modified so that it could be heated internally by electric heaters. Fluid was poured into the interior of the brake assembly through a small hole. Burning on contact with the heated brake and flame propagation characteristics were both determined. The temperature was 850 C (1560 F); with fluid flow rates of 500 and 2000 ml/min. The four fluids showed wide differences in fire resistance. The hydrocarbon burned on contact and carried flame from the brake, the silicate-ester burned less vigorously but did propagate flame, the phosphate ester flashed on the drum but did not propagate flame, and the halogenated polyaryl fumed but did not flash or ignite.

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INTRODUCTION

By R. E. Hatton[1]

Interest in and use of fire resistant fluids and lubricants has increased rapidly in recent years. Many questions have arisen concerning such subjects as the meaning of fire resistance, the methods of assessing fire resistance, the relation of test data to actual hazards in use and the interrelations between test methods. The purposes of this symposium were to make available current data and philosophical thought on these subjects through presentation of papers by those active in fire resistant fluid and lubricant development and use. This information should be invaluable to those charged with system design, fluid selection, and study of fire hazards in a wide range of industry - from the depths of the sea to the wide reaches of space.

A major reason for this symposium is the widespread differences of opinion that appear between those active in developing and using fire resistant fluids. Tests for measuring fire resistance have been developed by a number of groups, the majority of tests being designed to simulate actual applications. Relations between such simulative tests and interpretation of data in terms of fundamental combustion phenomena have been largely ignored. However, use of such empirical simulative tests has resulted in fluids and lubricants which have demonstrated reduced fire incidence in actual applications.

For example, aircraft fire resistant hydraulic fluids were developed to meet aircraft industry requirements, described in a specification (AMS 3150) written by representatives of the aircraft industry. Fire tests in this specification were simulative in nature and reflected a philosophy that fire resistance should be determined under simulative conditions and that results from a number of tests must be taken together in order to determine fire resistance. Since all materials will burn under some conditions, and utility requirements limit fluid selection, a degree of fire resistance and not fireproofness or nonflammability is usually obtained. The tests of this specification

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[1] Project manager, Development Dept., Organic Div., Monsanto Co., St. Louis, Mo.

were used to guide the development of fluids which have shown their utility and safety in aircraft use.

It should be noted that there exists a large number of proposed fire resitance tests which have been developed over a number of years and are designed to simulate certain conditions. As new conditions arise, new tests will be developed to simulate these new hazards. Efforts should be devoted to analyzing hazards and reducing them to as simple and fundamental parameters as possible. Standardizing groups and committee activities can provide a real service in helping the user by providing good methods for comparing products and in defining hazards.

This symposium was quite successful and has already generated a considerable amount of discussion. It is the purpose of this Special Technical Publication to record for future study, and as the basis for further discussion, test methods for determining fire resistance and the philisophies, opinions and ideas which led to their development.

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AN APPARATUS FOR STUDYING THE FIRE RESISTANCE OF HYDRAULIC FLUIDS AT ELEVATED PRESSURES

By J. A. Marzani[1]

ABSTRACT: An apparatus has been developed to study the spontaneous ignition phenomenon of hydraulic-type fluids at elevated pressures. Investigations are conducted at selected conditions of temperature, pressure, and reactant concentration. The level of fire resistance is measured in terms of a minimum reaction temperature and a minimum spontaneous ignition temperature for each set of reaction-controlling parameters. In addition, the closed system affords a measure of the energy release associated with the combustion in terms of temperature and pressure rise. Modifications of the basic apparatus can be made to study the influence of scaling and surface effects.

KEY WORDS: fire resistance, fire tests, flammability, hydraulic fluids

NOTE: The opinions expressed in this paper are those of the author and do not necessarily reflect the views of the Navy or the naval service at large.

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[1] Friction and Wear Division, U.S. Navy Marine Engineering Laboratory, Annapolis, Maryland.

An apparatus has been developed at the United States Navy Marine Engineering Laboratory (MEL) in Annapolis for the investigation of combustion phenomena at elevated pressures. The main area of interest lies in hydraulic fluids and their base materials. Some examples are petroleum-base fluids, phosphate esters, phosphonitrilates, silicones, and water-base fluids. Among the many properties required of a good hydraulic fluid, fire resistance is often of chief concern. The stimulus for this concern is the result of several instances of explosions recorded in hydraulic and pneumatic systems. Most of the effort expended on investigating combustion in the past has been done at or near atmospheric pressures. Some notable exceptions to this would be the CFR compression-ignition test (Military Specification MIL-H-19457) which is also performed at MEL, the shock tube work such as that done by Monsanto and others, and the high pressure bomb investigation of Zabetakis et al at the Bureau of Mines. In studying the complex combustion processes of a fluid, it is best to observe the phenomena under conditions such as might be encountered in an actual system.

APPROACHES. Compression ignition testing methods, such as the CFR engine and shock tubes, satisfactorily simulate dynamic system hazards. As an example, explosions often occur in dead-ended pipe sections and pressure gages which are subjected to sudden pressure surges. Combustion reactions of this type are associated with high temperatures and pressures and extremely short ignition delays. However, explosions may also occur spontaneously in a system after exposure to lower temperatures and pressures. Much longer ignition delays are encountered in these situations. To study the ignition and pre-ignition reactions for almost the entire range of ignition delays, a static bomb-type reactor was used. This approach, similar to that of Zabetakis, allows control of the critical reaction parameters of temperature, pressure, and reactant concentration.

PRINCIPLE OF OPERATION. The principle of operation involves subjecting a measured quantity of fluid sample to selected temperature and pressure conditions and monitoring the reaction. For each condition of pressure and sample concentration, there is a certain reactor temperature, called the minimum reaction temperature, below which no evidence of combustion can be detected by the temperature or pressure transducers. When the reactor is at this temperature, the reaction rate is sufficient that the rate of heat generated by the reaction is just greater than the rate of heat loss by the system. There is a small temperature rise, but apparently the depletion of reactants prevents continued acceleration of the reaction. When the reactants are totally consumed, the temperature falls back to the control temperature. As the reactor temperature is increased in subsequent determinations, the reaction rate also increases, in accordance with the theory of Arrhenius. When the reactor temperature is increased to the condition called the minimum self-ignition temperature (MSIT), the reaction rate becomes immeasurably fast. The area of positive ignition at and above the minimum self-ignition temperature presents the greatest danger because the reaction proceeds with explosive force. However, the pre-ignition region below this temperature is also of considerable danger since increasing the quantity of reactants or decreasing the heat losses would allow the slower pre-ignition reactions to propagate into violent reactions.

APPARATUS DETAILS. The apparatus consists essentially of an injection system and a reaction chamber (Fig. 1). The two sections are isolated by a quick-opening needle valve. Means are provided for maintaining the reaction chamber at a pre-selected controlled temperature and for recording pressure and temperature in the apparatus both before and after injection of the sample into the reaction chamber.

INJECTION SYSTEM. The injection system consists of a sample well and a high pressure air storage section. A pressure transducer is located in this section to reduce cleaning and avoid corrosion of this expensive component. This location was shown experimentally to have no discernible effect on either the response time or the magnitude of the pressure signal as compared with a pressure transducer located in the reaction chamber. The total volume of this injection system is approximately 17 cm^3. Without any modification, the sample well will hold up to 1.5 ml of fluid. The remainder of this system is charged with air to a pressure sufficient to inject the sample into the reaction chamber when the injection valve is opened. To date 3000 psig has been the pressure most commonly used.

REACTION CHAMBER. The heavy-wall type 304 stainless steel reaction chamber is approximately 10 in. long and has an inside diameter of 5/16 in. The total volume of this section, including the pressure gage, safety blow-out assembly, and connecting valves and tubing, is about 35 cm^3. The chamber is instrumented with three chromel-alumel thermocouples which are used both to establish the initial conditions and to monitor the reaction. Three separately controlled heaters are used to establish a uniform temperature in this reaction chamber. The two end heaters are necessary to make up the heat losses at the ends of the chamber and maintain a uniform temperature profile throughout the chamber (Fig. 2). The effect of temperature variations in the preheater is less significant because during injection the fluid passes through this area quickly. Temperature variations at the other end are also negligible because this area is generally outside the reaction site. Reactions are usually observed in the areas of thermocouples 1 and 2 (nearest the point of injection), and extend to thermocouple 3 as the injection delay increases. The pressure in this section has been 2000 psig for most of the experiments.

PROCEDURE. With the chamber at the desired temperature and pressure, a measured amount of sample is introduced into the sample well, and the injection system is pressurized. With the recorder operating, the valve is opened and the sample is injected into the chamber. Temperature and pressure traces similar to those in Fig. 3 are recorded on an oscillograph, after proper signal conditioning. Equilibration of pressure takes place in approximately 60 msec after sample injection, in the absence of reaction.

OBSERVATIONS. Any increase of temperature or pressure above the steady-state conditions is taken as evidence that combustion has occurred. The ignition delay is defined, overall, as the time lapse between injection and the maximum temperature attained during the run. This delay definition approaches that generally accepted for explosive reactions in which the delay is measured from injection to the first rapid rise or "kick" in the recorded temperature or pressure signals. The equality in definitions arises from the fact that in very fast reaction rates associated with explosive reactions, the time from injection to the first "kick" or to the peak signal is essentially the same.

DISCUSSION. In each case where rapid transients occur, such as injection and ignition, the thermocouple response will invariably lag the pressure transducer response. At conditions of high temperatures and short ignition delays, the error introduced by thermocouple lag and the non-homogeneity of the reaction as seen at the different thermocouple locations can be significant. This error can be eliminated by observing the pressure signal for these conditions. With long delays which exemplify slow reaction rates, the pressure rise and pressure-rise rate are too small to detect accurately; temperature indications, therefore, are used in this borderline area. The response of the thermocouple is sufficient to follow these slower reactions with no significant error. In addition, since the thermocouple is located in the reaction zone, it is very sensitive to small changes which result from the slow reactions.

Fig. 4 shows the corresponding pressure and temperature traces, side by side, for a range of reactor starting temperature (T_n) for one pressure and one sample size. Although these traces are typical, not all fluids react in such an orderly manner. In particular, the effect of temperature on the pressure rise is greatly influenced by the sample size and the concentration gradients present at the time of ignition. This may result in a succession of low pressure peaks at the higher temperatures and the greatest pressure peaks occurring at the middle or lower end of the temperature range.

Some fluids produce double ignitions, even at long ignition delays. Others produce self-ignitions after delays in excess of 100 sec without giving any evidence of pre-ignition. However, a most consistent relationship does exist between temperature and ignition delay. Fig. 5 shows a plot of temperature versus ignition delay at one pressure and sample size for two fluids, a triaryl phosphate and a fluorinated phosphonitrilate (tetrameric octafluoroamyl phosphonitrilate). It is interesting to note that both fluids reach a horizontal asymptote in approximately 20 sec, showing a limit for positive ignitions (where temperature and pressure "kick" occur) called a minimum self-ignition temperature (MSIT). A test only 7 F lower than this level produced no evidence of combustion for the phosphonitrilate. As may be seen in Fig. 6, the triaryl phosphate continued to show exothermic reactions at conditions as much as 120 F lower. This second horizontal asymptote would represent a minimum reaction temperature. It therefore appears that for the phosphonitrilate the minimum self-ignition temperature and the minimum reaction temperature are approximately equal. For some fluids these two critical temperatures can be considerably affected by the reactant concentration. Higher concentrations generally lead to lower values. This is probably because of the higher energy released from reactions involving larger amounts of material. The sample concentration has more effect on minimum self-ignition temperature than on the minimum reaction temperature.

The concentration of reactants (sample size) also affects the delay period before ignition (or reaction). The effect of sample size on ignition delay resembles Fig. 7. Other factors being equal, the delay varies inversely with the temperature. The sample size appears more critical at the lower temperatures with a gradual shift of the optimum concentration for minimum ignition delay toward the rich limits. Care must be taken in studying a fluid to be certain that the concentration used is sufficient to react at temperatures near the minimum reaction temperature, T_6. For example, if a constant stoichiometric volume were used, the apparent minimum reaction temperature would be between T_2 and T_3, a value possibly too far from T_6 to have significant meaning.

It has been mentioned that pressure is an important parameter in defining the fire resistance of a fluid. Fig. 8 shows the detrimental effect of pressure on the fire resistance of a triaryl phosphate hydraulic fluid and the experimental fluorinated phosphonitrilate. Ignition values obtained at atmospheric pressure, using ASTM D-2155-63T test procedure, are compared to those values obtained in the high pressure reactor at 2400 psig. The explosion hazard is increased not only by the reduced fire resistance of the fluid at pressure but also by the inability of the system to contain the pressure increase from the combustion reactions. The magnitude of this pressure rise depends of course on the amount of reactants present, the reaction rate, and the geometry of the system.

In the combustion apparatus, the pressure rise is a measure of the energy released. For the more rapid reactions which go to completion, this value can be compared directly with the heat of combustion. This has been shown in experiments with hexane. In the case of commercial hydraulic fluids, such a comparison is usually complicated by the physical and chemical complexities of the reactions. However, a relative rating on the reactivity of fluids can be obtained.

Fig. 9 shows the effect of pressure on the ignition delay and the MSIT of a triaryl phosphate hydraulic fluid in the combustion apparatus. It should be noted from the figure that the temperature required for an ignition delay of 4 sec is lowered approximately 80 F, and the MSIT of the fluid is lowered approximately 45 F by increasing the equilibrium pressure from 1700 to 3300 psig.

A similar apparatus is used to investigate the influence of larger reaction chamber diameters. The apparatus modifications for this purpose are primarily for safe adaptation to diameters of 1 and 2 in. In general, the reactions in larger diameter reactors are more complete and more violent. Increasing the diameter may result in an apparent increase or decrease in the fire resistance, depending upon the fluid, for comparable pressures and sample concentrations. The study into the effect of diameter on the ignition level is continuing. The larger chamber has the advantage of using liners which enable safe studies into the surface effects of different metals.

SUMMARY. The preceding discussion has centered primarily upon an apparatus and the presentation and analysis of its data. It is a new tool and much is still being learned. Improvements can still be made, but it does afford insight now into the combustion phenomena under conditions where they occur. Work is proceeding in two directions simultaneously. Fluids are being studied in a screening process on a relative basis to establish which are more promising. The more fire-resistant fluids are then looked at in greater detail. In the other direction research is being conducted into the mechanisms and effects of the controlling parameters. Such conditions as temperature, pressure, sample concentration, ignition delay, system geometry, and surface catalysis are being studied.

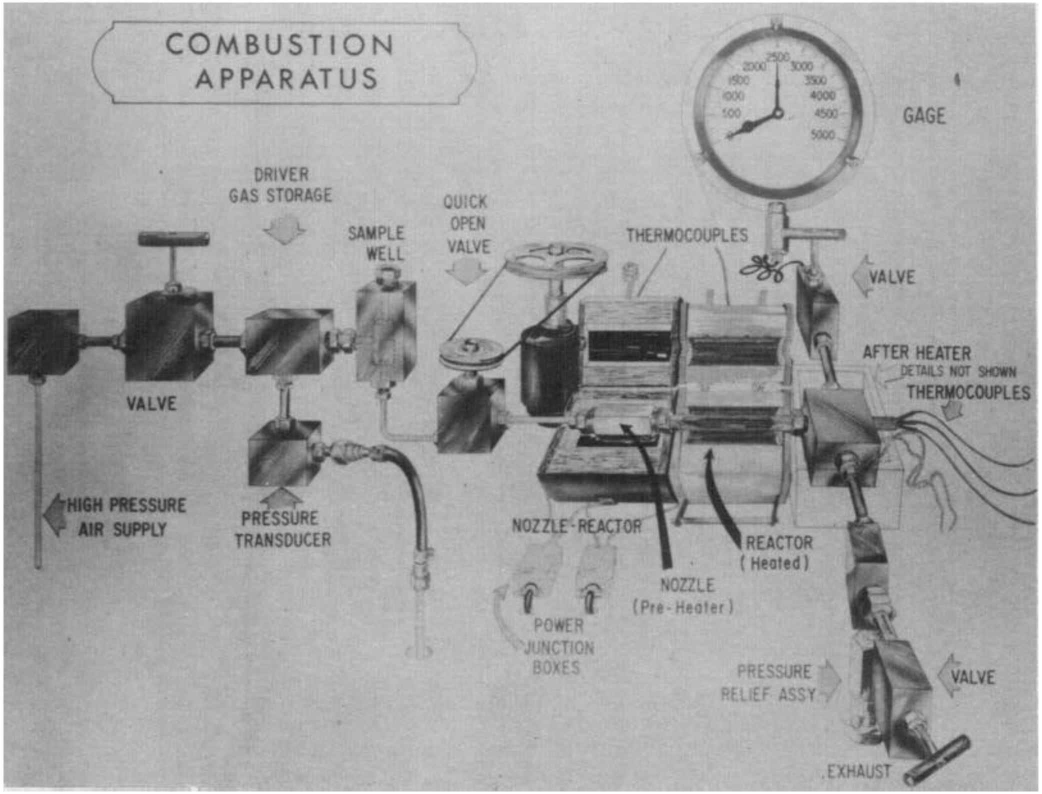


Fig. 1--Combustion Apparatus.

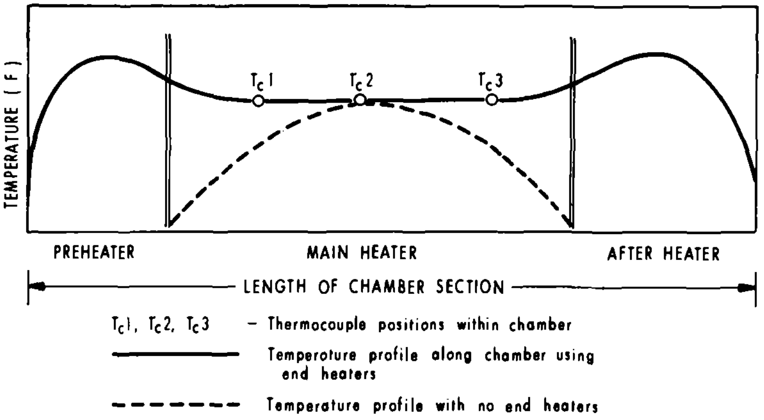


Fig. 2-- Reactor Temperature Profile.

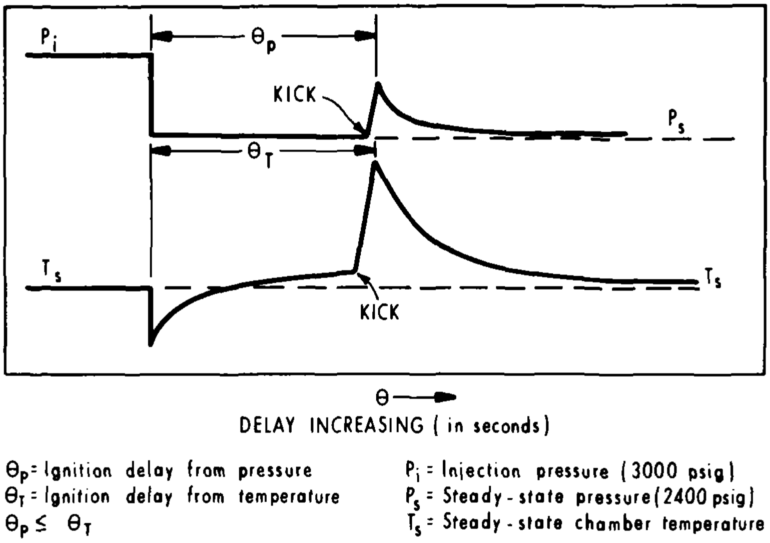
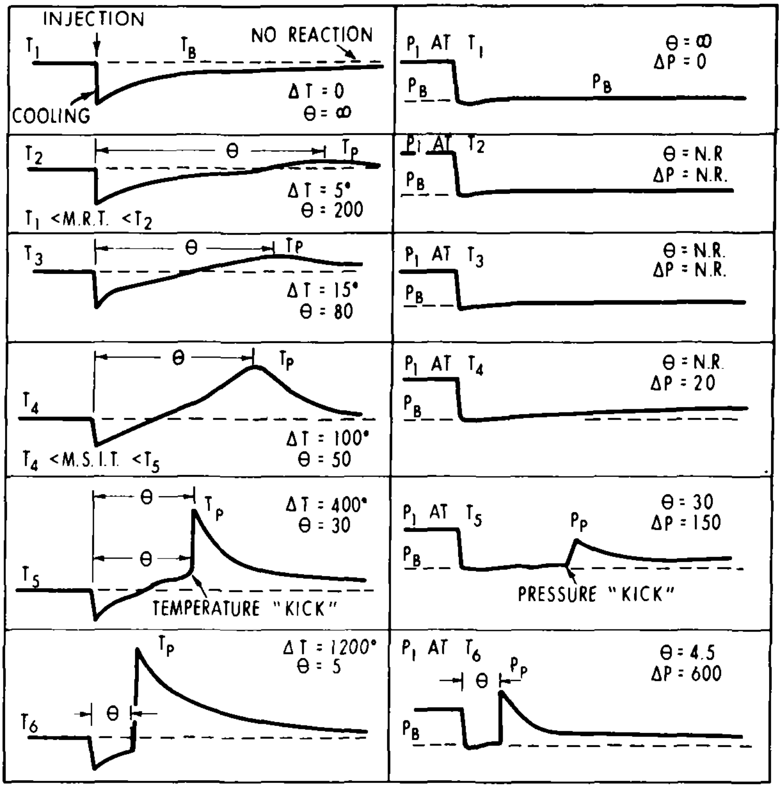


Fig. 3--Typical Trace.



Legend

T_B - Reactor base temperature
T_P - Peak temperature
$\Delta T = T_P - T_B$
$T_P = T_N (N = 1, 2, \cdots 6)$
$T_N < T_N + 1$
P_I - Injection pressure
P_B - Base pressure (equilibrium)
P_P - Peak pressure
$\Delta P = P_P - P_B$
θ - Ignition or reaction delay
T - °F
P - Psig
θ - Seconds
N.R. - Not readable

NOTE: Not to scale. All numbers are arbitrary.

Fig. 4--Typical Family of Temperature and Pressure Traces.

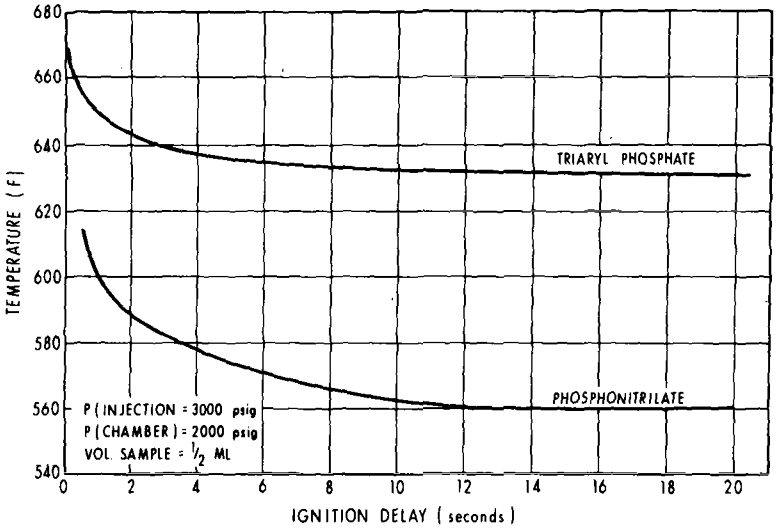


Fig. 5--Relative Fire Resistant Levels at 2400 PSIG.

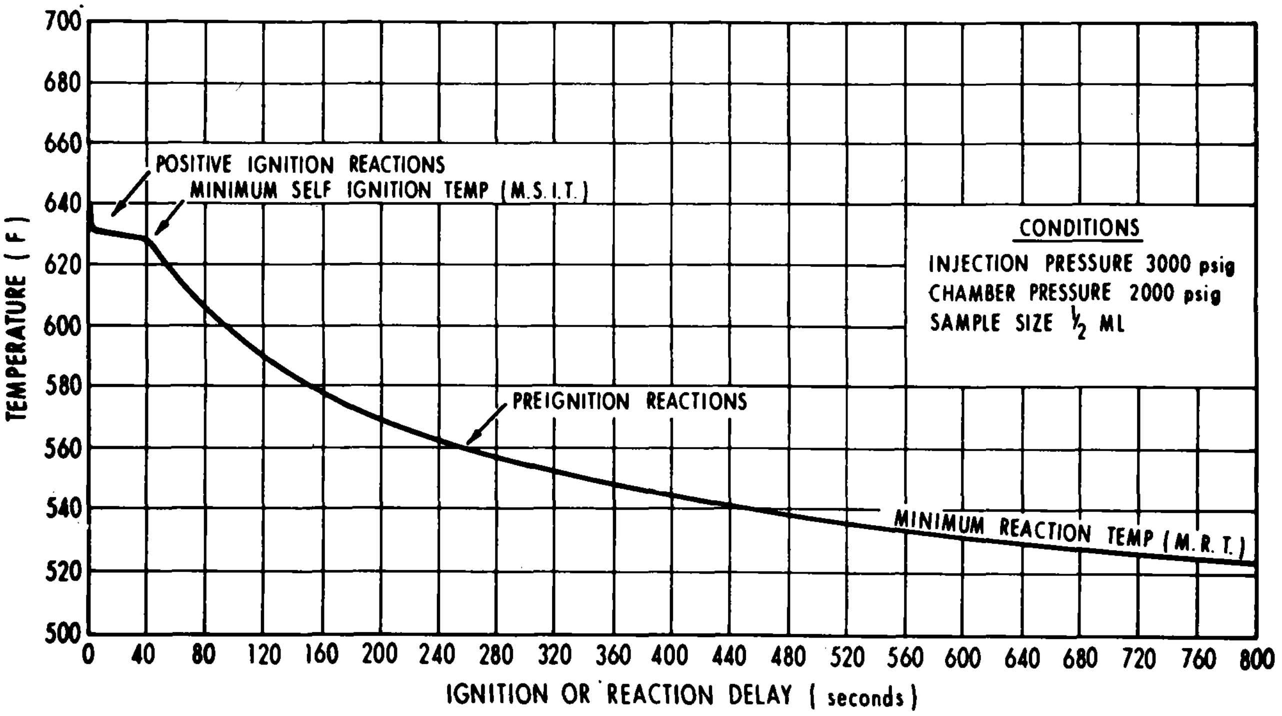


Fig. 6--Ignition Profile of MIL-H-19457(Type I) Tri-Aryl Phosphate.

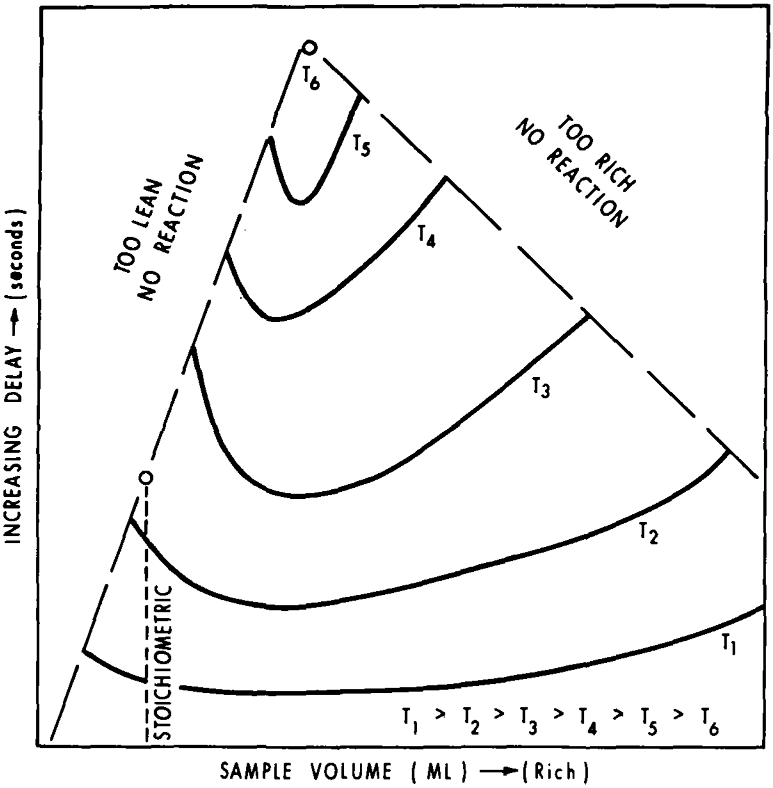


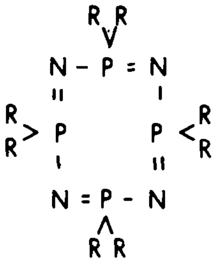
Fig. 7--Sample Size vs Ignition or Reaction Delay at Constant Pressure.

FLUID	TEST METHOD		
	‡ ASTM	* HIGH PRESSURE REACTOR	
	A. I. T. (F)	M. S. I. T. (F)	M.R.T. (F)
A	1040	630	515
B	900	560	560

LEGEND -

A ⟹ Mil H 19457 (TYPE I) TRIARYL PHOSPHATE

B ⟹



Where:
R $^{-}OCH_2(CF_2)_4H$

‡ Run conditions of atmospheric pressure
* Run conditions of 1/2 ml sample and 2400 psig

Fig. 8--Effect of Pressure on Ignition Levels.

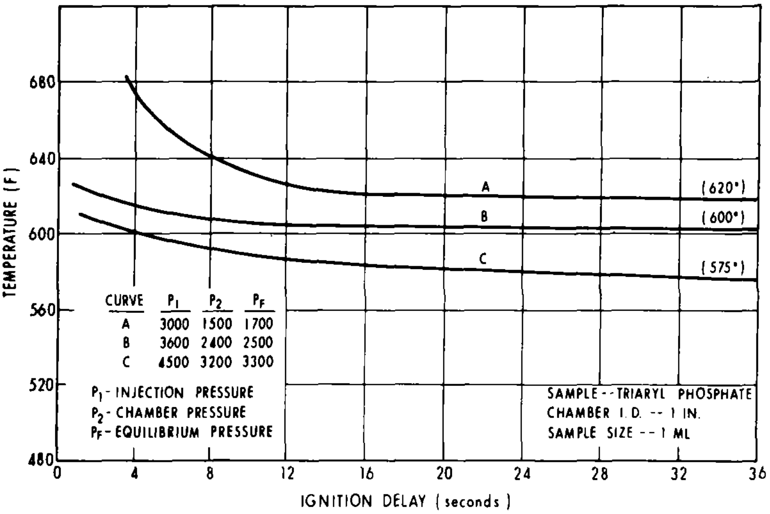


Fig. 9--Effect of Pressure on Fire Resistance (SIT).

HYDRAULIC FLUID CLOSED COMPARTMENT FIRE TESTS

By K. A. Kander[1]

ABSTRACT: A Closed Compartment Fire Test for evaluating fire resistance of hydraulic fluids in advanced supersonic aircraft is discussed. This test simulates leakage in proximity to heated aircraft skin at temperatures up to 550°F, both with and without simultaneous electrical arcing. The response to an electrical arc (time to initiate fire), size of fire, intensity of fire, flame propagation, and duration of fire after removal of arc are observed during each test. Simulative flammability tests of this type are required, since conventional hot manifold drip and spray tests do not always predict fluid flammability within the confines of a heated, closed compartment.

KEY WORDS: fire resistance, flammability, hydraulic fluids, fire tests, spray flammability test

The supersonic transport presents new problems for the hydraulics engineer, not the least of which is fluid leakage in a heated, closed compartment with electrical arcing as a possible source of ignition. The Federal Aviation Agency emphasizes the importance of considering such fire hazards in the following statement from its Supersonic Transport Request Proposal:

"The design shall incorporate features to minimize the possibility of inflight and ground fires or explosions and shall provide means to detect and control these hazards should they occur. Factors to be considered are high-temperature induced environment, auto-ignition, lightning, electrostatic potential, inerting, location and character of potential fire zones, detection and extinguishing system requirements."

Although leakage can be reduced through the extensive use of permanent plumbing connections, improved reconnectable fittings, and manifolding components, leakage possibilities still exist; hence, serious consideration must be given to flammability in the selection of an SST hydraulic fluid.

The flash, fire, and auto-ignition temperatures of a fluid, and the results of spray and drip hot-manifold tests do not necessarily predict the fire hazard resulting from leakage in a

[1] Airplane Division, The Boeing Company, Renton, Washington.

closed compartment. To compare the flammability of candidate fluids under this condition, The Boeing Company devised the closed-compartment fire test. For proprietary reasons, this paper does not discuss specific test results.

Test Setup and Procedures

A series of tests was conducted to determine the fire resistance of SST candidate hydraulic fluids under realistic fire hazard conditions. A hot-manifold drip test and a hot-manifold spray test simulated fluid leakage in proximity to heated surfaces such as brakes or hot ducts. The closed-compartment spray test discussed in this paper simulated the leakage of hot fluid in a heated compartment in the presence of intermittent electrical arcing.

The test setup is shown as a schematic diagram in Fig. 1. A titanium duct 12 in. in diameter by 12 in. long, containing an electrode centered along the axis of the duct, is placed in a 36 ft^3 oven together with a stainless steel tubing coil and a 60 deg, 1 gal/hr fuel nozzle. A distance of 9 in. from the nozzle to the entrance of the duct produces a well-dispersed spray at 1000 psi. A solenoid valve, located in the supply line between an accumulator and the oven, is used to control spray-time intervals. The oven top is ajar approximately 1/4 in. during testing, and a 3/4 in. drain hole in the oven floor is open to provide explosion relief and to permit some air to enter after the initial buildup and release of pressure.

Figure 2 is a photograph of the oven, and Fig. 3 is a photograph of the installation inside the oven with the electrode and duct displaced from their normal positions for photographic purposes.

Closed-compartment spray tests of each candidate fluid are conducted at sea level ambient pressure with fluid and oven stabilized temperatures of both 450° and 550° F. The test sequence is as follows:

1. Spray for 2 sec without arc.
2. If no ignition occurs, resume spraying and simultaneously introduce electrical arc of approximately 1/2 sec duration to observe response to arc.
3. Ventilate chamber and stabilize at desired temperature, then initiate simultaneous spray and electrical arc until ignition occurs. After ignition, shut off electrical arc and continue spray until observations of fire size, intensity, propagation, and sustaining characteristics are made.
4. Ventilate chamber and stabilize at desired temperature, then initiate spray. At time intervals selected to produce various concentrations of atomized fluid in chamber, introduce arc to investigate explosive characteristics.

During each test, color motion pictures are taken through the oven window and the following data are obtained:

- o Response to arc (time to initiate fire)
- o Size of fire
- o Intensity of fire including explosive characteristics
- o Propagation of flame from arc to nozzle
- o Time of burning after removal of arc

A method of evaluating such data is discussed later under "Evaluating Test Results."

Since this is a comparative rather than an absolute test, and since data are obtained by visual means, at least one repetition of the entire test for each fluid is recommended. Where erratic, non-repeatable data are obtained, additional testing is required to obtain a reasonable level of confidence. Of 17 fluids tested to date, only two required more than two complete tests to reach this level of confidence.

A more closely controlled means of obtaining the explosive characteristics of fluids is now under investigation with the present test equipment. Spray characteristics, nozzle orifice size, distance from nozzle to arc, and measured spray times before introduction of the arc will be varied to account for differences in fluid density and fluid viscosity as well as the fuel/air ratios required for accelerated burning.

Selection of Test Parameters

Reproducing all of the anticipated fire hazard conditions associated with hydraulic fluid in a supersonic transport would be prohibitive in terms of cost and time. To provide comparative data on fluid flammability in a closed compartment at a reasonable cost and in the time available, the following test parameters were established:

o Volume of Test Chamber - 36 ft^3

This volume is representative of an equipment compartment. A chamber of this size was readily available, together with incorporated temperature controls.

o Ambient Pressure - Sea Level

The presence of the maximum amount of oxygen is a conservative approach. However, ignition in the presence of an arc can occur at any altitude, and during rapid descent, the structure will retain heat. In the event of a structural failure, with resultant venting of a compartment, pressure buildup due to ram air could approximate that of sea level.

o Static Air (No Convection)

Static air conditions were maintained to evaluate explosive hazards caused by a buildup of vapors.

o Stabilized Temperature Levels 450° and 550°F

These temperature levels bracket the skin temperatures of a Mach 2.7 supersonic transport and provide fluid selection information for higher speed aircraft.

o Nozzle Configuration 60 deg Spray Cone, 1 gal/hr Fuel Nozzle

This nozzle provides a wide dispersal of finely-divided particles for evaluating explosive hazards.

- Nozzle Pressure - 1000 psi

A nozzle pressure of 1000 psi was selected to obtain maximum dispersal and to eliminate any possible increase in spontaneous ignition temperatures that might occur at lower pressures as noted in a previous WADC-Bureau of Mines Test Report (1). No discernable difference in ignition characteristics was noted when Boeing tests were originally conducted at 200 psi.

- Distance Nozzle to Arc - 9 In.

This distance was selected by trial to give a well-dispersed spray into the titanium duct.

- Duct Material - Titanium

The duct material on which the spray impinges is of titanium, since this is the metal chosen for the SST structure.

- Time Intervals for Spraying and Arcing

The time intervals described in the test procedures were selected by trial at the start of the program to obtain a wide variety of flammability data and to investigate explosive hazards.

Evaluating Test Results

A fire resistance value for each fluid tested in the closed-compartment fire test may be obtained by utilizing the test data and by making certain assumptions in assigning values to each criterion.

A method of deriving a weighted fire resistance factor (K_c) for the closed compartment test is as follows:

a. Response to Arc:
   Immediate (< 0.5 sec) = 15
   Delayed (> 0.5 sec) = 0
b. Size of Fire:
   Confined (Does Not Envelop Duct) = 0
   Envelops Duct = 15
c. Intensity of Resultant Fire:
   Explosive = 40
   Flickering = 0
d. Fire Propagation:
   Back to Nozzle = 15
   Localized at End of Duct = 0
e. Burning after Removal of Arc:
   > 5 sec = 15
   0 sec = 0

The above values assigned are upper and lower limits for each test; intermediate values may be obtained by observation during tests.

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(1) WADC Technical Report No. 52-35, Supplement 4, Dated January 1956, "Research on the Flammability Characteristics of Aircraft Fuels," by M. G. Zabetakis of U.S. Bureau of Mines.

$Kc = \Sigma\ a, b, c, d, e$

The lower the factor, the higher the fire resistance. This approach is primarily a tool for comparing fluids and cannot in itself be used as an absolute measure of fire resistance. A fluid may be rejected for any single unacceptable property or performance characteristic, even though its calculated fire resistance value is good. For example, a fluid that ignites at 550°F without an arc or explodes when finally ignited would be rejected for use in a supersonic transport.

Seventeen fluids have been subjected to closed-compartment fire tests to date. Some general observations may be made:

o All fluids ignited in the presence of an arc.

o There was a large variation in flame propagation and intensity between fluids.

o The only variation obtained in spraying at 200 and 1000 psi was the flame size due primarily to the difference in expelled fluid volume for a given time.

The results of other tests such as flash, fire, autoignition, and hot manifold tests also performed by Boeing to assess fire resistance do not necessarily correlate with the results of the closed-compartment fire test. As an example, the following table lists the relative grouping of 3 fluids in the above tests, with 17 fluids tested.

RELATIVE RATING - 17 FLUIDS TESTED

Fluid	Flash Point Deg F	Fire Point Deg F	Auto-Ignition Temperature	Fire Resistance In Spray Hot Manifold Test	Fire Resistance In Drip Hot Manifold Test	Fire Resistance In Closed Compartment Test
A	Low	Low	Low	Good	Poor	Extremely Poor
B	Low	Low	Low	Good	Poor	Fair
C	Low	Medium	Medium	Good	Medium	Good

Conclusions

o Tests of the type described in this paper are necessary to predict fluid flammability in a closed compartment.

o The closed-compartment fire test, as now constituted, depends upon visual observation rather than precise instrumentation. However, data sufficient to compare the flammability of fluids are obtained. Other tests, such as hot-manifold drip and spray tests, must be used to evaluate flammability under other aircraft operating conditions.

o There is a need for more basic research in fluid flammability with closely controlled test parameters and accurate measurements of results. Such research efforts could be performed in a smaller chamber and could therefore more accurately control test parameters such as fuel/air ratios, spray dispersal, spray pressures, etc. Studies of this nature would relate variations in test parameters with measured flammability.

o To our knowledge, no test program will predict fluid flammability under all operating conditions with absolute accuracy. However, simulative laboratory flammability tests such as the closed-compartment fire test are required in the selection of a suitable hydraulic fluid.

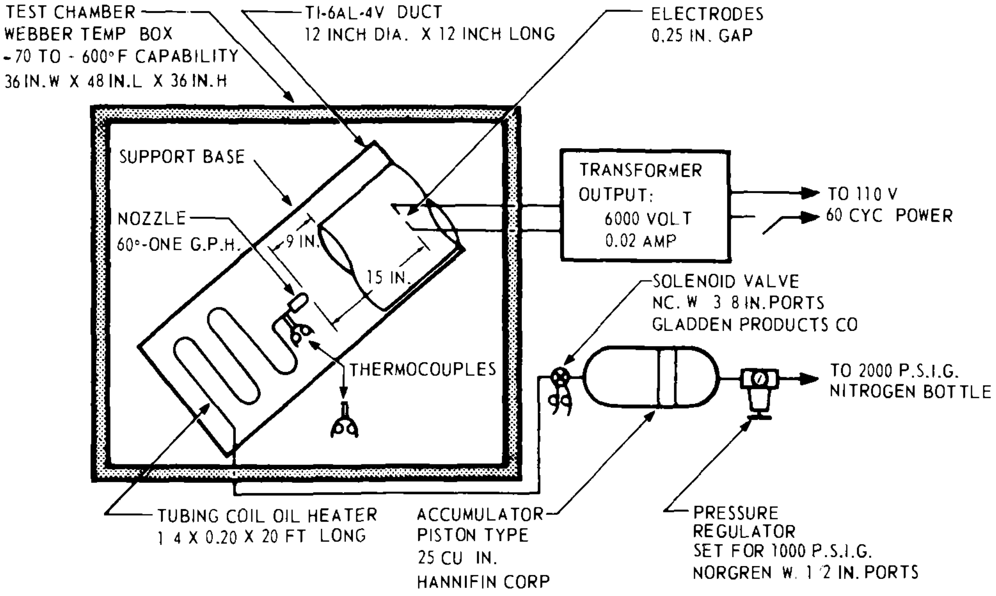

Fig. 1 SCHEMATIC DIAGRAM OF TEST SETUP.

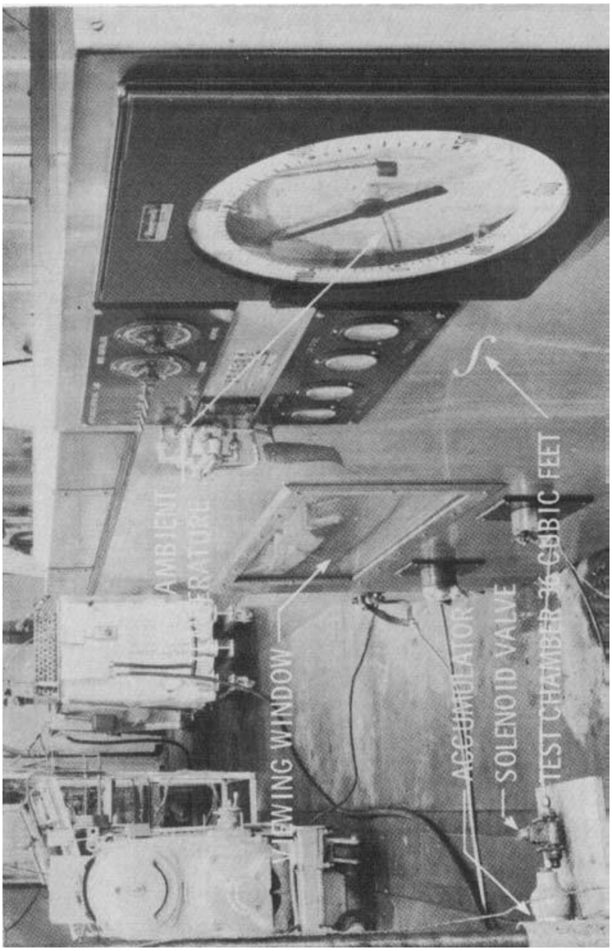

Fig. 2 OVEN EXTERIOR.

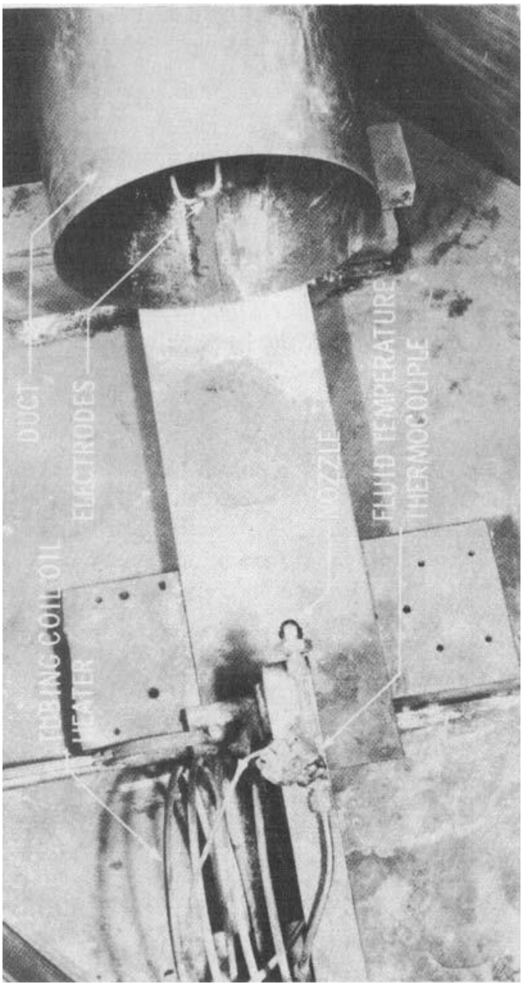

Fig. 3 INSTALLATION INSIDE OVEN.

A SIMPLIFIED SPRAY-FLAMMABILITY TEST FOR HYDRAULIC FLUIDS

By H. H. Rowand, Jr.[1] and L. B. Sargent, Jr.[2]

ABSTRACT: Hydraulic lines are known to rupture permitting a stream or spray of hydraulic fluid to encounter the flame of burning fuels or the surface of hot or molten metal. This is a prevalent cause of fires in many industries. High-pressure spray-flammability tests simulate this type of industrial hazard and are able to distinguish the difference in fire resistance among the various kinds of hydraulic fluids.

The available high-pressure spray tests are inconvenient to use, however, since they usually must be done outdoors where they are subject to the vagaries of the weather. If done indoors, the tests must be performed in specially assembled fireproof areas. In addition, relatively large volumes of test fluids are needed, and the high-pressure pump, motor, and auxiliary equipment can become costly.

A low-pressure spray-flammability test that can be conducted safely in a conventional laboratory hood is described. The equipment required is simple and inexpensive and only limited amounts of fluid and fuel are needed. The flame from a laboratory gas-air burner serves as the source of ignition. The data obtained correlate very well with those from a conventional high-pressure spray-flammability apparatus. Results from both kinds of tests evaluating the several types of fire-resistant hydraulic

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[1] Research Engineer, Lubricants Div.,Alcoa Research Labs., Aluminum Co. of America, New Kensington,Pa.

[2] Chief, Lubricants Div., Alcoa Research Labs., Aluminum Co. of America, New Kensington, Pa.

fluids are presented in tabular and pictorial forms.

A variety of test conditions is not available with the new method described, but the conditions are chosen so that they simulate practical industrial situations and provide correlation with other flammability tests at a sufficient confidence level.

KEY WORDS: hydraulic fluids, fire resistance, flammability, ignition, fire tests, flame

A prevalent cause of industrial fires is a rupture in a pressurized hydraulic line where the escaping fluid contacts a flame or a hot surface. An obvious solution to this problem is to use a hydraulic fluid that will not ignite under those circumstances. Like many things, however, "flammability" is a relative term and must be measured in order to assess its value. Many kinds of fire-resistant fluids of varying degrees of flammability have reached the market place in recent years, and it has become necessary to test them in order to ascertain their value to industry.

There are many requirements for hydraulic fluids, but this paper is concerned only with their ability, when in the form of a spray, to resist ignition upon contact with a flame. Several high- and low-pressure tests for evaluating spray flammability have been used and described[3-6]. These tests are discriminating and useful and have served a definite purpose in helping to supply fluids of satisfactory fire resistance to industry.

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[3] SAE Specification, AMS-3150B.

[4] U. S. Government Specification, MIL-F-7100.

[5] U. S. Federal Test Method 791 (Tentative Standard 6052T).

[6] H. H. Rowand, Jr., "A Spray Flammability Test for Hydraulic Fluids," Symposium on Hydraulic Fluids, ASTM STP 267, Am. Soc. Testing Mats., 1959, p. 50.

These referenced tests, however, are inconvenient to use since they must be performed outdoors or in specially assembled indoor fireproof areas. If done outdoors, they are subject to the uncertainty of the weather and the smoke and fumes can annoy neighbors. In addition, relatively large volumes of test fluids are needed, and the equipment that is necessary can be bulky and costly. Because of the testing conditions, these methods can be dangerous to personnel and buildings.

These factors, and our need for a relatively safe, quick, and convenient indoor test for evaluating fire resistance of hydraulic fluids, caused a search to be made for such a method, which culminated in the test described in this paper. The conditions of the test allow it to be performed in a laboratory hood.

Apparatus

The heart of this new, simplified, spray-flammability test is an airless paint spray gun[7] (Fig. 1), which is a simple, inexpensive mechanism that pumps the test fluid onto a disk rotating at 15,000 rpm. The disk propels the fluid in the form of small droplets through a regulated slot in the side of the gun. A well-defined, flat, atomized spray pattern is provided in which overspray is reduced to a minimum, since air and high pressures are not involved. The only utility supply needed for the gun is a 115v ac electric current.

The atomized spray produced by this gun simulates the type of discharge which is produced when a small rupture occurs in a pressurized hydraulic line. The adjustments needed to produce this spray are simple and more convenient than those needed to produce a

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[7] Model 2300 manufactured by Electro Engineering Products Co., Inc., Chicago, Ill., or Rogers Rotary manufactured by Napco, Inc., Cleveland, Ohio.

suitable spray pattern in the ordinary high-pressure flammability tests.

The source of ignition is a glass blower's torch, using a natural gas-air mixture adjusted to provide a "lazy" yellow flame. Any other source of this kind of flame can be used in place of the glass blower's torch.

This apparatus should be set up in a laboratory hood with a normal draft to carry away any smoke or fumes generated during the test.

Procedure

The flame is positioned in front of the gun slot opening at a distance of 4 in. so that the spray hits the flame about 1 in. away from the end of the burner. The gate opening on the gun is adjusted to 1/4 in. and the volume adjustment is slightly opened. About 500 ml of the test fluid are preheated to 150 F and then poured into the gun reservoir (only about 25 ml of fluid are actually used per test).

The burner is ignited and the gun turned on. Almost immediately an assessment of the degree of flammability of the test fluid is obtained.

The tests described here were carried out in a metal laboratory hood (5 ft wide by 3 ft deep by 3 ft high) with a conventional exhaust system. Excess fluid that does not burn or vaporize impinges on a metal sheet and drains into a metal pan, from which it may be disposed. At times, the flame of burning hydraulic fluid will extend to this metal sheet and cause the fluid adhering to it to burn.

Results

The degree of flammability of a fluid under the conditions of this test can best be described by the following terms:

1. Violent fire - where the entire spray pattern ignites and burns vigorously.
2. Spasmodic fire - where the fluid ignites sporadically and each ignition extinguishes itself almost immediately.
3. Fire at torch - where the fluid burns in the area of the flame only.
4. No fire - absolutely no indication of ignition.

Fig. 1 illustrates the apparatus and the type of flame used.

Fig. 2 illustrates the violent fire obtained when a mineral oil hydraulic fluid of about 300 SSU/100 F is tested. Fluids of this type ignite immediately and burn vigorously throughout the spray pattern, as shown.

Certain synthetic nonaqueous fluids of the phosphate ester type produce a "fire at torch" result which is illustrated by Fig. 3.

The water-in-oil (invert) emulsions ignite almost as readily as do mineral oils and produce fires of about the same degree of violence (Fig. 4). At the other extreme, no fires have been obtained with a number of water-glycol hydraulic fluids (Fig. 5).

The spasmodic fire is illustrated in Fig. 6 and is difficult to depict in a still photograph because of the fleeting and unpredictable ignitions. This type of ignition is observed with certain fluids such as phosphate esters, as shown in Table 1.

The results obtained with the authors' test on the reference fluids distributed by Committee N, Section VI, of ASTM Committee D-2, are shown in Table 2. These results correlate well with those from other fire-resistance tests and from our knowledge of the composition of the fluids.

The atomized spray emitted by the new apparatus has made it possible to show differences in the flammability characteristics of fluids which could not be detected with high-velocity, high-density, straight-stream sprays. Because of the small volume of test fluid used in this new tool, the smoke and fumes produced are considerably smaller in volume than those produced in the referenced tests.

Previous to the development of this test, fire resistance of hydraulic fluids was evaluated in these laboratories by a much more sophisticated, outdoor, spray-flammability test method described by Rowand[6]. Correlation between the previous method and the present simpler one is good, as can be inferred from the results shown in Table 1. The selected illustrative results cover the several types of hydraulic fluids being marketed.

Discussion

Combustible fluids can be ignited by several different methods. The test described considers only the ignition of a fluid spray precipitated by a burning fuel. While the results obtained are indicative of the fire-resistance level of the fluid evaluated, they should not be considered as a final rating of the fluids because other sources of ignition, such as an electric arc, probably would rate them differently.

The test is a simplified one, capable of considering only limited variation in the several parameters involved, and cannot detect subtle differences in fire-resistance of fluids. It has a useful purpose, however, as a "go - no go" type of test and has correlated very well with experience. It will separate the "safe" fluids from the questionable ones for those users desiring a high margin of safety.

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[6] H. H. Rowand, Jr., "A Spray Flammability Test for Hydraulic Fluids," Symposium on Hydraulic Fluids, ASTM STP 267, Am. Soc. Testing Mats., 1959, p. 50.

No consideration is given to other aspects of hydraulic fluids under conditions of potential ignition, such as the generation of fumes or smoke, as the test is concerned only with the question of flame ignition.

Conclusion

A simplified spray-flammability test which can be conducted in a conventional laboratory hood is described. It uses a small amount of test fluid, a commonly available flame burner, and a simple and economical spray generating device. Results from this test on a variety of commercial hydraulic fluids are presented and show good correlation with results on the same fluids from a more sophisticated, high-pressure spray-flammability test method.

Because of the limited variation of test variables which can be examined by this method, it cannot detect subtle differences in fire resistance of fluids, nor can it evaluate the fire resistance of fluids under a number of test conditions. It can discriminate among commercially available fluids and detect those that present potential fire hazards and those that can be considered safe in industrial usage where potential hydraulic line ruptures allow the escaping fluid spray to contact an open flame.

Table 1--Fire resistance of hydraulic fluids as measured by spray-flammability tests.

		Flammability	
Fluid	Type	Low Pressure Spray[a]	High Pressure Spray[b]
A	Mineral oil	Violent fire	Violent fire
B-1	Phosphate ester base	Fire at torch	Fire at torch
B-2	Phosphate ester base	Fire at torch	Fire at torch
B-3	Phosphate ester base and mineral oil	Violent fire	Violent fire
B-4	Phosphate ester	Spasmodic fire	Spasmodic fire
B-5	Phosphate ester	Spasmodic fire	Spasmodic fire
C-1	Water-glycol	No fire	No fire
C-2	Water-glycol	No fire	No fire
D	Invert emulsion	Violent fire	Violent fire

[a] Present method - Rowand and Sargent.

[b] Prior method - Rowand[6].

Table 2 --Flammability results on reference fluids from Committee N, Section VI, ASTM Committee D-2, with the simplified spray test.

Fluid	Results
1-A-1	Violent fire
2-B-2	No fire
3-C-1	Spasmodic fire
4-D-2	Fire at torch
5-D-1	Fire at torch
6-B-3	Violent fire

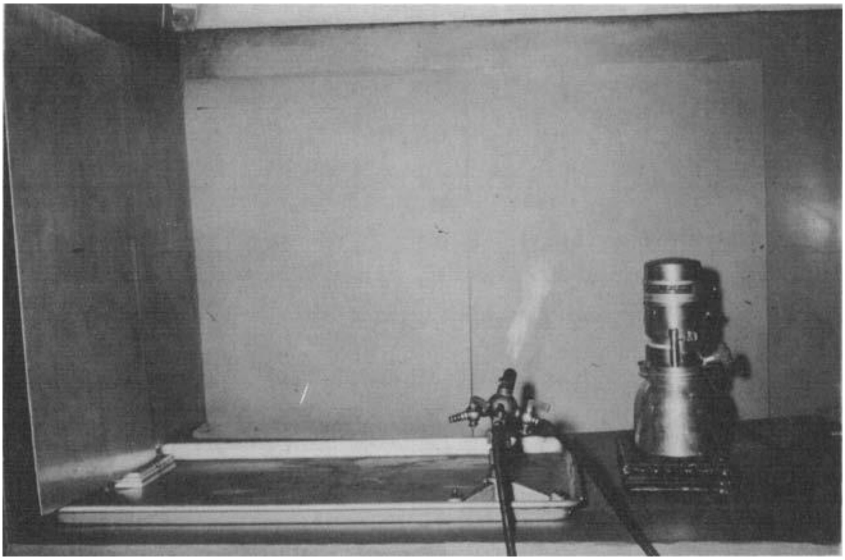

Fig. 1 -- Spray Flammability Test Apparatus.

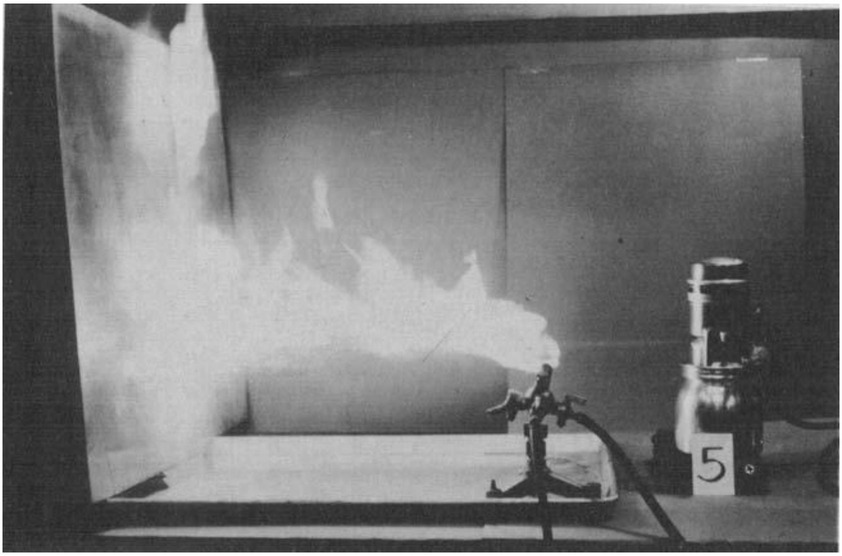

Fig. 2 -- Violent Fire - Mineral Oil.

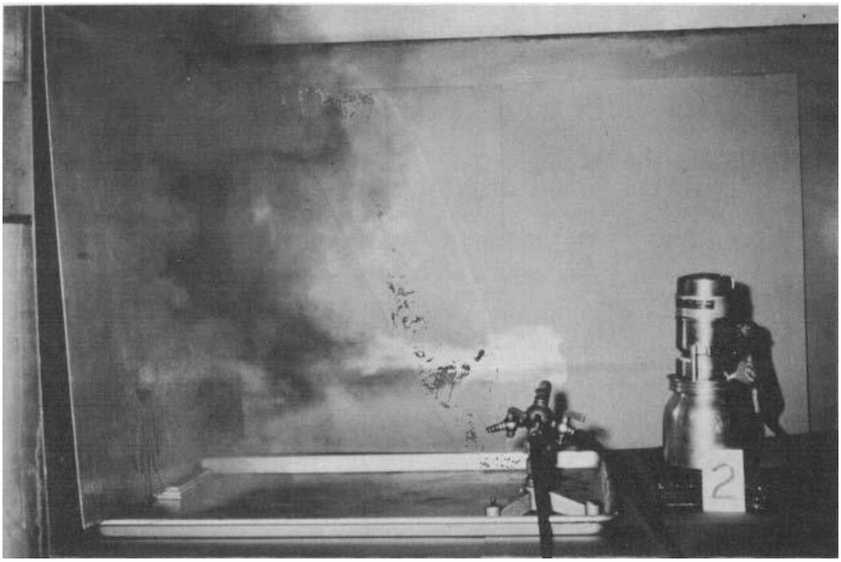

Fig. 3 -- Fire at Torch - Phosphate Ester Base.

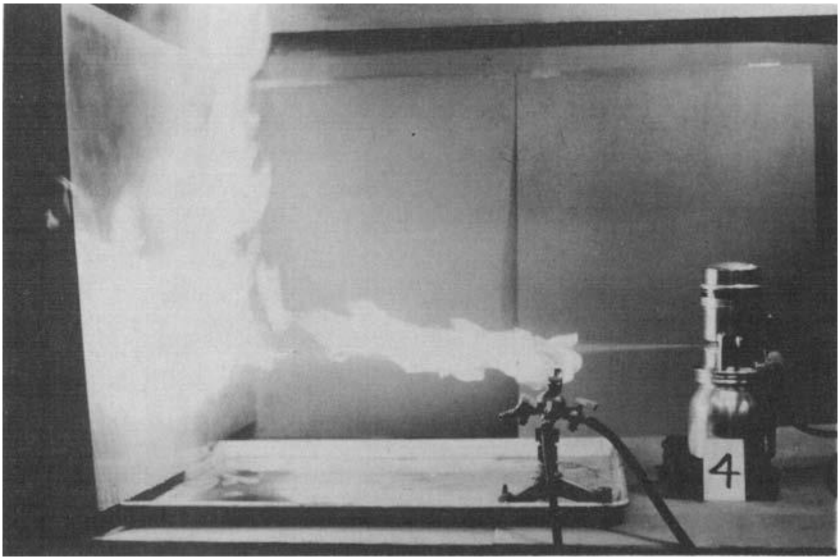

Fig. 4 -- Violent Fire - Invert Emulsion.

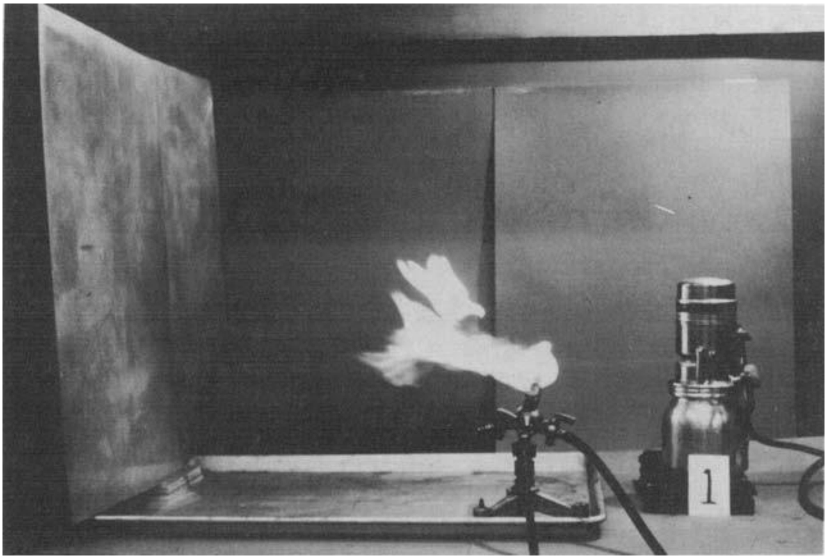

Fig. 5 -- No Fire - Water-Glycol .

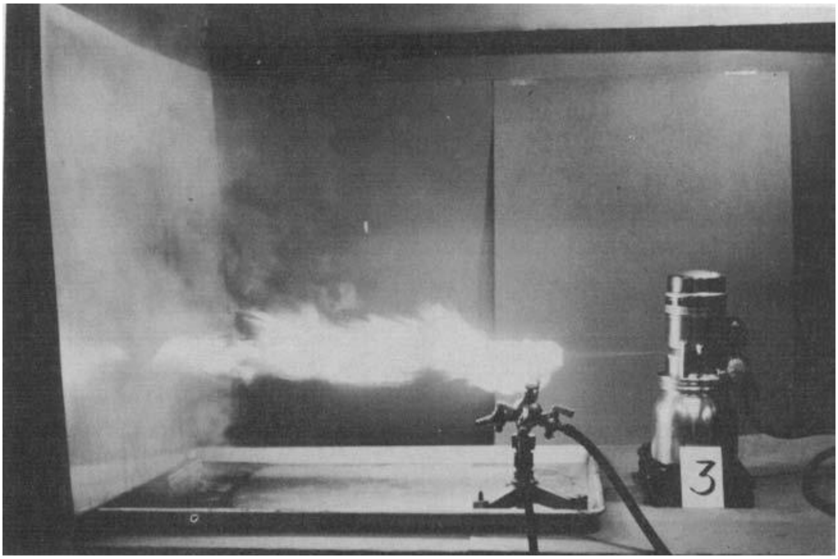

Fig. 6 -- Spasmodic Fire - Phosphate Ester.

COMMENTS ON AIRCRAFT FLUID FIRE HAZARD ASSESSMENT

By R. S. McCord[1]

ABSTRACT: Tests for assessing the fire hazard from hydraulic fluids, coolants and lubricants in aircraft have been evaluated in light of the new hot wing compartment condition to be encountered in future supersonic transport aircraft (SST's). Existing flammability tests for subsonic aircraft report inception of visible flame while SST's require tests reporting excessive temperature, or pressure rise as the evidence of flammability. The large number of environment variables affecting flammability prevent certain extrapolation of a fluid's hazard rating from a test procedure to actual conditions of use. Flammability is thought to be more a condition of environment than a material property.

KEY WORDS: fire resistance, flammability, hydraulic fluids, supersonic transports, fire tests

Our work to date at the Douglas Aircraft Company in assessing the probable hazard associated with the use of various functional fluids in supersonic aircraft might be gravely misleading if presented in an incomplete state at this time. Our objective was and still is to produce aircraft hydraulic systems, both subsonic and trans-sonic, which will have zero total fire hazard. In connection with the forthcoming supersonic transports (SST's), we first examined performance of the various subsonic hydraulic systems and the properties of their fluids.

The meaning of the subsonic fluid flammability test methods of ASTM, of SAE specification AMS 3150C, and the MIL-F-7100 incendiary gun fire test method, together with the performance records in both these laboratory tests and in actual service of all past fluids were then re-examined. The hydraulic fluids included the original castor oil base types, the old AAF 3580 petroleum oil, MIL-H-5605 petroleum oils, the MIL-H-8446 synthetic siloxane high temperature fluid, MIL-F-7083 water base fluids used as late as the Korean War, and the present commercial jet aircraft, phosphate ester type hydraulic fluids.

[1] Project Engineer, Douglas Aircraft Co., Santa Monica, Calif.

For the last two fluid types, which were developed in the 1940's using the test procedures just listed and were the only products making any claim to fire resistance, no fires were found in service. Apparently for the types of ignition we have in subsonic aircraft, these early fire test methods are adequate, perhaps more than adequate.

The next step was to define what additional ignition sources would exist in future trans-sonic aircraft designs and add these new test requirements to those used previously in developing fire-safe hydraulic fluids. Immediately it was found that the Mach 3 design of commercial supersonic transport aircraft probably would be the most severe case in terms of need for a fire-resistant hydraulic fluid. Aircraft designs for speeds higher than Mach 3 might actually have a lower total fire hazard. Total fire hazard, which we consider to be the product of a fluid's flammability rating, times the degree of exposure to ignition sources, times the value of the mission under consideration, was highest for the Mach 3 design. Beyond that, the Mach 7 and Mach 15 aircraft dwelt within the earth's atmosphere such a short time per flight that their temperature and ignition problems actually decreased and the total fire hazard was no worse than the Mach 3 case.

It was also found that the principal difference between the old subsonic design case and the Mach 3 case was the possibility of accidental hydraulic sprays or mists occurring within hot, relatively unventilated compartments such as in the leading edge of a wing and then reacting to produce a temperature and pressure rise. Normal ambient temperatures in the wing would be in the 450 to 550F range. Fluid temperature probably would be 450F. Catalytic surfaces available to initiate combustion reactions certainly would include titanium, cobalt, and high chromium and nickel alloys. Ventilation rate would be very small but not zero. During a rapid descent the high temperatures might exist simultaneously with almost sea level air pressure. There was uncertainty whether designers could completely exclude the possibility of open electric arcs occurring simultaneously.

Hence, we considered the worst case where arcs did occur. Also it was clear that as the designed operating temperature in aircraft wings rose, perhaps eventually reaching the condition where a wing would be designed to operate normally in a glowing red condition, the reporting of visual flame as a criterion of flammability becomes less and less meaningful. Since our new Mach 3 supersonic transport's wings can not "see" but can react to higher temperature or pressure or to a corrosive atmosphere we changed our thinking and used only a sudden rise of pressure or temperature above the ambient; or evidence of corrosive attack on the structure by the fluid's decomposition products as the indicators of a fluid's ignition in a new SST hot compartment fluid flammability test.

Study of physical and chemical properties of proposed Mach 3 hydraulic fluids failed to disclose any obvious property which would correlate with ignition or non-ignition under these SST hot compartment conditions. Furthermore, simulated Mach 3 hot wing compartment fluid flammability tests of available products in which the heated fluid was sprayed into a hot 4 1/2 in. diameter by 36 in. long titanium tube both with and without an electric arc, failed to correlate either with known fluid properties or with the older subsonic flammability tests on the same fluids. Disconcertingly, some fluids which did not pass the older tests passed these supposedly more severe supersonic hot compartment conditions. Our work so far has added five new variables: fluid quantity, dwell time, geometry of the compartment, ambient pressure, and atmospheric motion to our test envelope without really clarifying anything. It is apparent that in any one portion of this test envelope of multiple variables, a fluid might pass, yet fail catastrophically if only slightly different conditions of environment were tried. In the face of this information, we could only conclude that fluid flammability was more a property or result of the environment tested than it was an innate property of the fluid.

Flammability apparently is a derived product of environment and material rather than a pure property of material alone. Reconsideration of the older fluid flammability tests tends to confirm this. The total available energy in these older tests was actually low enough that many anomalies in flammability were unwittingly obscured. We know now that all our subsonic fire-resistant aircraft hydraulic fluids can sometimes be made to burn under some non-use simulating conditions. For example, even water can be oxidized or "burned" to hydrogen peroxide if we were to push its use as a safe functional fluid into some exotic applications. Obviously, our present phosphate ester commercial aircraft hydraulic fluids serve us safely today only because our subsonic aircraft designs never really tax the fluid's chemical structure. Equally obvious is that at the higher SST energy levels, present subsonic fire-resistant fluids will be overtaxed and unsafe and that a new chemical class of fluids will be needed to maintain the present margin of safety.

We know there are useful differences between chemically different classes of fluids even in this unquantitative area of flammability assessment. To find these differences and put them to use in the form of 450F fire resistant fluids, we are funding further study of the fundamentals of unwanted combustion. Adoption of any operating commercial SST hydraulic fluid, before solutions for the remaining problems have been found, will require severe compromise in either the aircraft's performance or its total flammability hazard rating.

First, we must know all the variables which influence flammability of fluids in the case of the SST hot compartment. The full range of each variable possible in the SST must be explored.

Otherwise a designer adopting a fluid as safe within his set of SST environmental variables and their ranges, and believing the resulting total flammability hazard rating for that fluid, might actually be perilously close to complete reversal of his hazard rating because of some undiscovered variable or a variable range that actually extends beyond his assumptions. It was the discovery of the pronounced effect upon our tests of variables formerly thought to be of little concern, expecially the marked lowering of ignition temperature caused by slight air movement, which prompted me to delay a formal paper on techniques of exact assessment of fluid flammability.

The second pitfall to be avoided in future work is the negative test, the laborious, detailed, simulative flammability test which when finally done tells only that we are safe in one little area of operation and gives no clue of what may happen outside that set of assumed conditions. If flammability is a material property we should be able to obtain positive flammability design properties useable much as are density, viscosity, volatility, and freezing point. Such data should enable us to outline the entire envelope of safe operating conditions rather than speculate about the few unrelated points in our environment which flammability tests now disclose.

Solution of these last two problems would greatly simplify the task of both the aircraft system designer and the fluid synthesizer.

To summarize, we can say that of the fluid flammability tests now in use:

1. All are different.
2. Each is indicative of something.
3. Some contradict others.
4. None, by itself, is clearly sufficient for SST design.
5. When all are taken together they cast doubt on the validity of any extrapolation from one set of environmental conditions to any other even slightly different set of conditions. They make us realize how much more our present term "fluid flammability" is related to the selected technique of testing rather than being an innate fluid property useful for design purposes.

ASSESSMENT OF THE FLAMMABILITY OF AIRCRAFT FLUIDS

By J. A. Macdonald[1]

REFERENCE: J. A. Macdonald, "Assessment of the Flammability of Aircraft Fluids," Fire Resistance of Hydraulic Fluids, ASTM STP 406, Am. Soc. Testing Mats., 1966.

ABSTRACT: The methods adopted for assessing the fire resistance of aircraft fluids have been critically reviewed. Most attention has been devoted to an examination of methods of assessing the likelihood of spontaneous ignition of fluids under the most severe conditions that may be encountered in flight. The present closed vessel and hot manifold tests are inadequate and may be misleading. Some other tests, for example the wick, spark ignition of spray, and CAA flammability reference scale, are criticized as being too narrow in their scope.

It is suggested that adequate basic information on the flammability of fluids would be obtained by improved versions of two simple tests:

1. Flash point (lower and upper).
2. Minimum spontaneous ignition temperature in a larger isothermal enclosed apparatus.

Where the application involves ignition on the outside of a hot body in cooler surroundings, the effects of environmental factors are so strong that each case should be treated individually, though some guidance can be provided by tests on a 6 in. hot pipe or flat plate.

KEY WORDS: fire resistance, flammability, fire tests, flash point, spontaneous ignition, hydraulic fluids

The fuels available for aircraft propulsion all exhibit ignition characteristics which can make them hazardous under certain conditions. The same appears to apply to lubricating oils at the moment, but hydraulic fluids have not been restricted to natural minerals, and recent developments have

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[1] Mechanical Engineering Dept., Royal Aircraft Establishment, Farnborough, Hampshire, England.

led to the appearance on the market of a number of competitive fluids for which a claim is made that in addition to their good functional properties they are "fire resistant."

The assessment of "fire resistance" has always presented a problem, since the conditions under which a fluid fire can occur in aircraft cover a wide spectrum varying from 'wick' pool or spray fires ignited by flame or spark to spontaneous ignition of vapor by heated surfaces. It is therefore desirable that standardized tests be employed which realistically represent the worst conditions that may be met in flight. Most attention is given to ignition by heated surfaces, which is becoming increasingly important owing to the large areas of such surfaces presented by jet engines at all aircraft speeds and by the structure at supersonic speeds.

In the following paper the results of spontaneous ignition tests on a number of aircraft fluids are presented together with comments on the significance of the test techniques. Suggestions are also made about the basic tests of all kinds considered essential to assess the relative merits of fluids from the point of view of their safety in aircraft.

SPONTANEOUS IGNITION TEST TECHNIQUES

The subject of ignition by heated surfaces has occupied the attention of investigators for many years. It has been shown that ignition depends on many factors such as the nature of the surface; its shape, size, and orientation; and the composition of the combustible air mixture. In the following paragraphs an attempt is made to assess the significance of some physical variables on ignition, partly to illustrate the very limited application of particular configurations as a standard for comparing fluid flammability and, probably more important, to illustrate to aircraft designers how some physical conditions lead to increased ignition temperatures.

Uniformly Heated Enclosures

Ignition Temperature -- Tests have been made in uniformly heated vessels varying in volume from 125 cm^3(1) to 7.5 ft^3(2). The smaller vessel is used in the ASTM Method of Test for Autogenous Ignition Temperatures of Petroleum Products (D286-58T), whereas the larger vessel, made of steel, was used by Boeing to determine experimentally the effect of vessel size on ignition. Recent work (3) at the Royal Aircraft Establishment using kerosine fuel in stainless steel vessels varying in diameter from 1 to 18 in. showed that the ignition temperature varied with the vessel size as shown in Fig. 1. This result has some significance in the design of a standard test, since, if the temperatures are to be regarded as limiting values for design purposes (rather than as merely comparative values), then it is necessary for the test vessel to be as large as the largest heated space expected in the aircraft. In any case, it seems unwise to use a vessel smaller than about 6 in. diameter, since the inhibiting action of the walls then becomes significant. A point which has not been established is whether all combustibles would be affected equally by vessel size.

Table 1 shows the ignition temperatures and their associated conditions of test for a number of fluids in air at atmospheric pressure. The interpretation of these data presents some difficulty largely because the criterion of ignition is not always defined and presumably varies from test to test, as may also the fuel concentration. There is a need to establish a common standard in any test such as determining for each fluid the optimum concentration for ignition (as is done in Ref. 1) and adopting a reliable criterion for ignition such as a minimum ignition pressure rise of, say, 1 psi. A visual observation of flame is regarded as unreliable since ignition pressure rises of several psi are given by cool flames which cannot be readily seen.

Ignition Delay — A characteristic of the spontaneous ignition process is the delay period required for preflame reactions prior to an explosion. This is a factor of some importance in evaluating the risk of ignition in hot ventilated regions in aircraft which contain combustible vapor. Work on this aspect using kerosine fuel yielded the results shown in Fig. 2. Results for hydraulic fluids in enclosures of uniform temperature are scarce, but what evidence there is indicates that although the ignition temperatures of the synthetic fluids are higher than those for mineral oils the delays vary in a random fashion at their respective ignition thresholds. Ventilating air may therefore be of little value in preventing the ignition of some synthetic fluids. However, since this factor can be significant in the design of systems to avoid explosions, the minimum values found should be reported for temperatures near the ignition threshold.

NON-UNIFORMLY HEATED CONDITIONS

Flammable fluids may leak into a region where the temperature is not uniform - such as a compartment containing a hot duct or pipe which is at a significantly different temperature from that of the enclosure walls. The likelihood of ignition in such a case will depend on the temperature to which the mixture is heated by the duct and the time the gas takes to traverse the hot boundary layer and adjacent zones of recirculation in relation to the ignition delay period.

Natural convection and ventilation processes predominate in these cases. In the following notes the results of tests made in an apparatus (see Fig. 3) simulating the leakage of fluids into an unventilated region containing hot pipes of 6, 3, and 1½ in. diameter are discussed. The fluids employed in this study are kerosine (Avtur), DTD 585, chlorinated silicone, and phosphate ester. The tests with the first two were done with whole vapor and liquid fuel, whereas liquid tests only were made with chlorinated silicone and phosphate ester because of their low vapor pressure and the onset of fluid decomposition below ignition temperature. The fluids tested are commented on and compared in the following paragraphs.

Kerosine

The results of tests with vapor and liquid are shown as threshold lines on Figs. 4 and 5, respectively, for the three pipes. Ignition temperature shows a strong dependence on pipe diameter particularly at low sphere temperatures with liquid injection. The pipe temperatures required to give ignition increase with reduction of sphere temperature about equally for either mode of fuel injection with the 6 in. pipe, but to a much greater extent with liquid injection on the two smaller pipes. The sudden steepening of the curves for liquid injection at temperatures about 350 to 400C is believed to be due to a reduction in fuel concentration caused by the onset of film boiling on the pipe. Values for the 6 in. pipe appear to correspond with those given by flat plates (4) at similar environmental temperatures. With liquid injection, ignition pressure rises increase to 40 psi at about 15C above the ignition threshold, whereas with vapor injection there is a slower rise to about 30 psi. In cases where the ignition is below 400C the pressure rise tends to decrease markedly at about 450C, and care is required not to mistake this for the minimum spontaneous ignition temperature (SIT).

An analysis of the heat transfer rate from the pipes (see Fig. 6) to the vapor air mixture for the ignition threshold temperatures as shown in Fig. 4 appears to be reasonably independent of pipe size, and since the delay values at the minimum ignition temperature are of the same order, about 15 sec, the total heat input to mixture for each pipe is reasonably constant. This indicates that a basis for the correlation of such work may lie in the heat transfer relationships rather than the thermal or chain branching theories with which so far we have been unable to correlate the results obtained in these tests.

DTD 585

The ignition thresholds are shown in Figs. 7 and 8 for vapor and liquid injection, respectively. In this case liquid injection gives slightly lower ignition temperatures except for the 3 and $1\frac{1}{2}$ in. pipes at the lowest sphere temperatures. Again, the differences are much greater for the 3 and $1\frac{1}{2}$ in. pipes than for the 6 in. one. DTD 585 liquid is generally more ignitable than kerosine liquid, but the reverse is true when vapor is applied below the pipes.

The pressure rises for this fluid are similar to those given by kerosine. The delay results are also similar to those for kerosine but generally yield slightly lower values.

Chlorinated Silicone

The ignition thresholds for the 3 and 6 in. pipes are shown in Fig. 9. The pipe ignition temperature is reasonably independent of the sphere temperature over a fairly wide range, a characteristic not exhibited by other fluids. Tests made by Rolls Royce (5) using chlorinated silicone sprayed on to a flat plate show agreement over the environment temperature range covered by the present work on the 6 in. pipe.

The pressure rises are slightly less than shown for kerosine and DTD 585, but the delay values are of comparable magnitude. A feature noticed is that the ignition from the 3 in. pipe yielded pressure rises much less than from the 6 in. pipe and less than those obtained from kerosine and DTD 585 on the 3 in. pipe.

Phosphate Ester

Figure 10 shows the ignition boundaries for the three pipes. The ignition temperature varies appreciably with the sphere temperature. The explosions are less violent than for the other fluids, giving pressure rises up to about 15 psi, and the delays are very long varying from about two minutes to over ten minutes.

Comments on Hot Pipe Tests

A comparison of the behavior of the fluids tested on hot pipes in a cooler 18 in. sphere (Figs. 11 and 12) under static conditions indicates that the order of merit of the four fluids is changed significantly by a change in pipe size or sphere temperature. Other physical factors which can affect the likelihood of ignition in the presence of hot pipes are the dimensions and shape of the enclosure, the fuel concentration, which depends on its boiling characteristics and the rate and method of application, and the ignition delay (particularly if there is a ventilating flow).

It therefore seems that such tests, to be of value, must be done at conditions that fairly closely simulate those under which the fluids will be used. If a general comparison of fluids outside hot surfaces is required to rate fluids in order of merit, this should be done on 6 in. pipes (which give ignition temperature close to the minimum attainable under these conditions) at the environmental temperature of interest and covering a range of fluid application rates to obtain the minimum ignition temperature.

HOT MANIFOLD TEST TO SPECIFICATION AMS 3150C

There is no agreed international method of investigating conditions under which spontaneous ignition of aircraft hydraulic fluids can occur. The British Ministry of Aviation Test Specification (DTD 5526 - draft) calls for the use of a closed uniformly heated container, whereas the Americans rely on either closed vessel tests (ASTM Method D286-58T or D2155-63T) or, to an increasing extent on the exposed manifold test AMS 3150C. The closed vessel tests are generally fairly closely comparable, but the hot manifold rig has been reported to give much higher values.

A series of tests using the hot manifold rig was therefore initiated to investigate its reliability and the values it gives for kerosine, DTD 585, chlorinated silicone, and phosphate ester. The manifold temperature and rate of fluid application were varied widely from the suggested values in AMS 3150C in order to ensure that minimum SIT values were obtained. The results of these experiments are shown in Fig. 13 and show that, apart from kerosine, the ignition temperature is little affected by the rate of flow of fluid.

The temperatures indicated by a thermocouple mounted in the recommended position on the side of the tube are about 30C lower than those at the top, the cooling effect of the fluids on the thin walled tube was very great (up to 400C), and ignition delay times vary randomly over a range of 10 to 300 sec, mainly due to the effect of external drafts in this exposed arrangement. The ignition temperatures, for some fluids, are much higher than would be obtained on a 6 in. tube or flat plate and very much higher than are given by closed vessels. Consequently, this apparatus is regarded as having very limited validity as a standard means of assessing the risk of spontaneous ignition generally in an aircraft. It would seem better to rely on closed vessel tests as a general criterion and to treat other arrangements on their merits as individual cases, since the liability to ignition can be affected by a number of factors, for example, size, temperature distribution of the heated surface and its surroundings, pressure, fluid concentration, and degree of ventilation.

SUGGESTED TESTS FOR FLAMMABILITY

Flash Point (closed)

This test is simple, reproducible, and should give a good indication under controlled conditions of the fluid temperature at which sufficient vapor is given off to be ignited and thus of the fire risk at sea level in the presence of an ignition source. It may be argued that this test does not give a sufficient indication of the persistence of a fire and that a "fire point" test would be more appropriate as indicating not only the ignitability but persistence of a flame without further external agency. This argument is not strictly valid because (a) 'fire point' is generally recognized as being much less reproducible than 'flash point', (b) the presence of a flame in practice would ensure the continuance of vapor, and (c) the proposed tests on flammability range (see below) would indicate the ease of burning rich concentrations of vapor. The quoted range of temperature between 'flash point' and 'fire point' is reasonably constant for most of the fluids so far tested. In the case of spray ignitions, the ease of ignition will depend on the heat supplied from an external source to vaporize the droplets - hence ignition depends on droplet size, temperature, and size of flame.

Range of Flammability

The flash point test relates to one concentration of air to fuel and is therefore connected with a particular temperature. Ignitions can occur over a fairly wide range of fuel vapor concentrations depending on the fluid employed. Therefore, it is desirable to obtain the range of concentration which is ignitable. This will involve obtaining vapors at temperatures above the 'flash point' condition until the "upper flash point" temperature is obtained. This condition may be difficult to obtain with some synthetic fluids which readily decompose at temperatures near the flash point, however some indication of this risk would still be obtained even under these limited conditions. There may be some point in recording pressure rise as an indication of the presence of flame rather than relying on visual observation. The present (Pensky Martens) standard apparatus could be employed for the above tests.

Minimum Spontaneous Ignition Temperature

This test condition is becoming increasingly important in high speed aircraft in regions where the environmental temperature may be high with possible higher internal temperatures, particularly in the region of engines or hot ducts.

The minimum SIT is known to depend on vessel size, vessel material, and fuel concentration. It is recommended on the basis of our experience that the minimum SIT be established under the following conditions:

1. A uniformly heated stainless steel cylinder 12 in. in diameter, 12 in. long should be used.
2. Fuel concentration should be varied over a range sufficient to obtain optimum quantity for each fluid.
3. A criterion of ignition should be established. We suggest that an ignition pressure rise of 1 psi would be suitable provided that it occurs at a rate exceeding 1 psi/sec. In addition to the above, the ignition delay should be recorded at the minimum SIT and 10 and 20C above the minimum.

Since lower ignition temperatures will be obtained with this apparatus compared with the present standard apparatus (ASTM D286 or DTD 5526 equipment), the criterion of low flammability will need to be fixed at a lower level than at present, so as to allow those fluids to pass which give an appreciable improvement in safety.

COMMENTS ON OTHER FLAMMABILITY TESTS

The following tests (in addition to those already discussed) are used by various authorities. Some comments are made on their advantages and disadvantages.

Wick Test

A bunsen burner is applied to a protruding portion of a wick of glass wool soaked in a small bath of fluid. The ease of ignition and persistence of flame after removal of bunsen flame are noted. This appears to be a useful test for indicating the possible spread of a fire, but if a flame is present due to some other combustible it is likely to continue to burn and therefore keep alight even the less flammable fluids. The degree of risk in a situation of this nature will depend to a large extent on the temperature of the fluid and degree of ventilation. A more realistic test would be the 'flash point' of the fluid, which is not dependent on diffusion criteria or rate of heat transfer to the fluid under test. The wick test gives very little indication of the relative merits of the fluids, particularly of those which show no persistence of flame when the bunsen burner is removed.

Spark Ignition of Fluid Spray

The test fluid is pumped through a diesel injector and nozzle to form a cone of fine mist of approximately 20 deg included angle. The electrodes are placed 6 in. from the injector nozzle on the fringe of the mist cone. This test is most difficult to control; ignition depends on droplet size, and spark position is very critical, both having a profound effect on ignitability. The correct conditions for one fluid may be considerably different for another fluid, since reproducibility of droplet size is difficult. In particular, the droplet size will vary from one fluid to another for a given nozzle size due to changes in fluid viscosity and other physical properties.

Flammability Reference Scale

This test was originated by the U.S. Civil Aeronautics Authority. A measured quantity of a mixture of the test fluid and an inhibitor or "snuffing agent" is sprayed through an oxy-acetylene flame from a jet at high pressure. The length of the flame, which is produced along the stream of fluid, is measured at the instant of its separation from the ignition source. A proportional reduction in this flame length is obtained by increasing the inhibitor content until a point is reached where a small change in mixture composition produces a large change in flame length. The percentage inhibitor required to produce this critical condition is taken as a measure of the flammability of the fluid under test.

It is difficult to visualize this test as a measure of the ignitability of the fluid, it is rather a measure of the ease by which a fire could be extinguished by an inhibiting agent under the specified condition of test.

CONCLUSIONS

After consideration of the tests which are commonly employed for assessing the flammability of fluids, I consider that little useful information is given by the 'wick' test, spark ignition of fluid spray, the flammability reference scale, and the hot manifold test, but adequate basic information would be given by the following tests:

1. flash point (lower and upper limits) .
2. minimum SIT in an improved standard test apparatus and using better defined criteria.

ACKNOWLEDGMENT

I am grateful to my colleagues at the Royal Aircraft Establishment for their valuable assistance in preparing this paper and to the Ministry of Aviation for permission to publish it.

LIST OF REFERENCES

(1) 1965 Book of ASTM Standards, Part 17 D286-58T, American Society for Testing and Materials.

(2) D. D. Kurtovich and G. E. Hays, "Spontaneous ignition and supersonic flight," Society of Automotive Engineers, Paper 431C, October 1961.

(3) J. A. Macdonald and R. G. White, "Spontaneous ignition of kerosine (AVTUR) fuel vapour: The effect of vessel size," R.A.E. Technical Report No. 65138, July 1965.

(4) G. Beardsley, "Investigation of spontaneous ignition temperature of kerosine," Rolls Royce Ltd., England, Ref. DMB/Gdl 20 PW, April 1964.

(5) G. Beardsley, "Investigation of Spontaneous ignition temperature of DTD 585 and Silcodyne H hydraulic fluids." Rolls Royce Ltd., England, Ref. DMB/Gdl 20/HC, September 1964.

(6) J. M. Kuchta, A. Bartkowiak and M. G. Zabetakis, "Autoignition of Hydrocarbon jet fuel." R. I. Bureau of Mines report of investigations 6654.

(7) H. E. Berlec and M. G. Zabetakis, "Compressor and related explosions," I. C. Bureau of Mines information circular 8187, 1963.

Table 1--Data on spontaneous ignition of fluids in uniformly heated enclosures.

Vessel Type and Capacity	Source of Information	Ignition Temperature of Fluids Tested, deg C			
		Kerosine	DTD 585 mineral (MIL 5606)	Chlorinated Silicone	Phosphate Ester
Pyrex 150 cc	ASTM Method D286 Chemical Inspectorate M.O.A.	228	225	480	593
Steel Cylinder 6" dia. 21" long[a]	Chemical Inspectorate M.O.A. D.T.D. 5526	247	240	410	490
Pyrex Flask 200 cc	Ref. 6 U.S. Bureau of Mines	232(JP6)	-	-	-
Pyrex Flask 2000 cc	"	227(JP6)	-	-	-
Stainless Steel 2540 cc	"	228(JP6)	-	-	-
Stainless Steel 460 cc	"	238(JP6)	Ref. 7 353 (mineral fluid)	-	Ref. 7 559
Stainless Steel 1800 cc (4" diameter)	R.A.E. Ref. 3	226	-	-	-

[a] NOTE: One cc of fluid is used in this test and probably gives too weak a concentration for hydrocarbon fuels to yield the minimum ignition temperature, whereas Ref. 1 calls for the optimum fuel quantity.

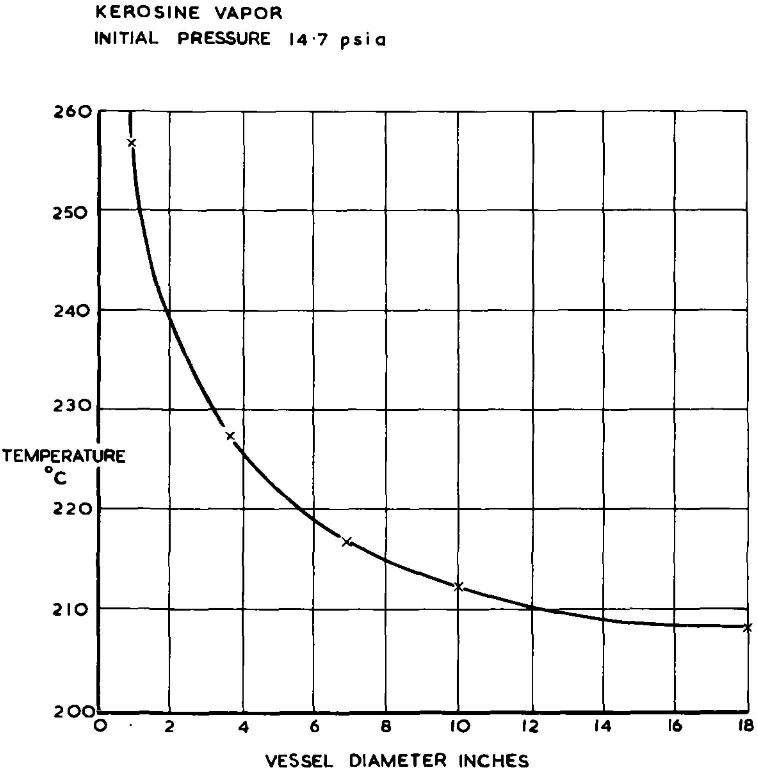

Fig. 1 The effect of size on the minimum spontaneous ignition temperature given by uniformly heated vessels.

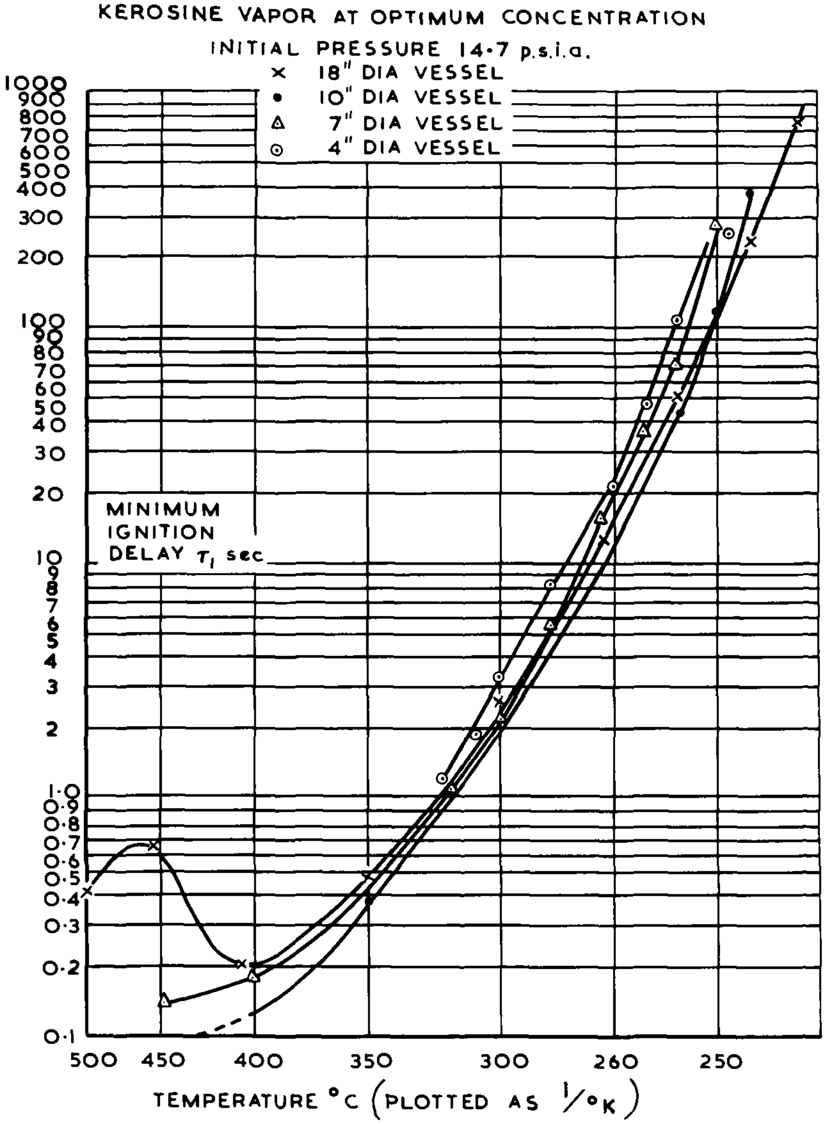

Fig. 2 The effect of temperature on ignition delay time in uniformly heated vessels.

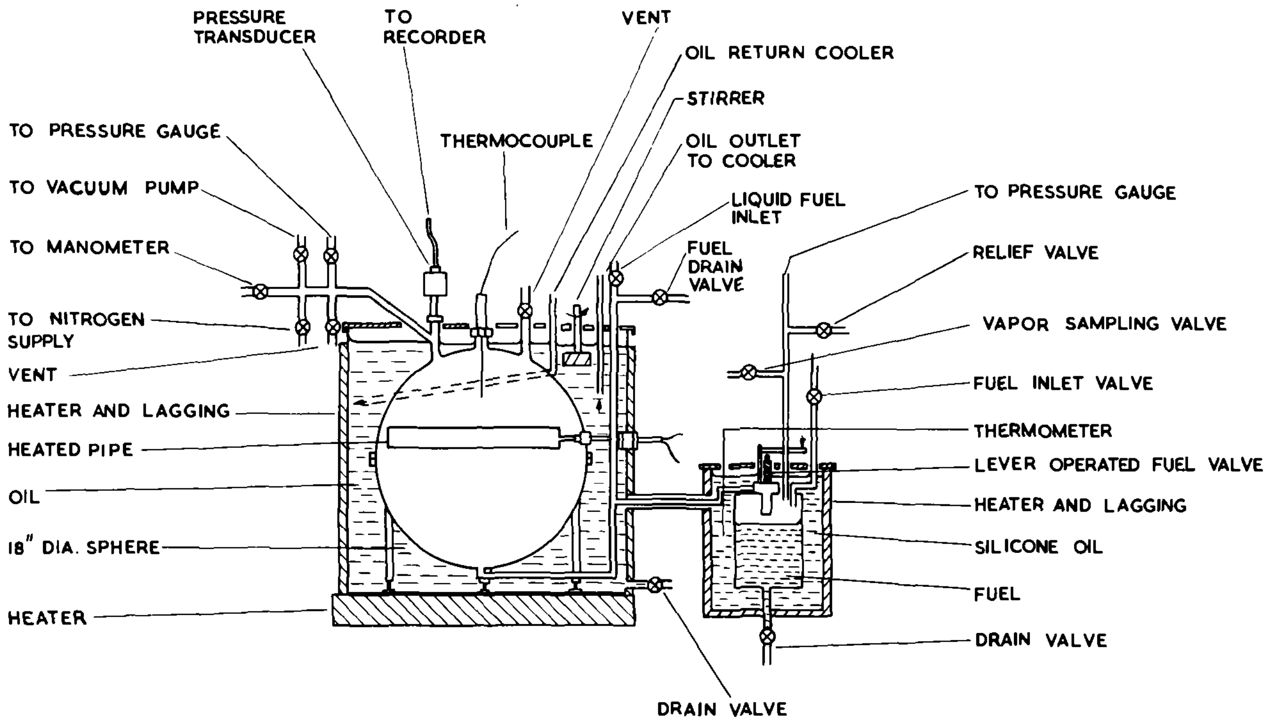

Fig. 3 Apparatus for determining the spontaneous ignition temperature of fluids by hot pipes.

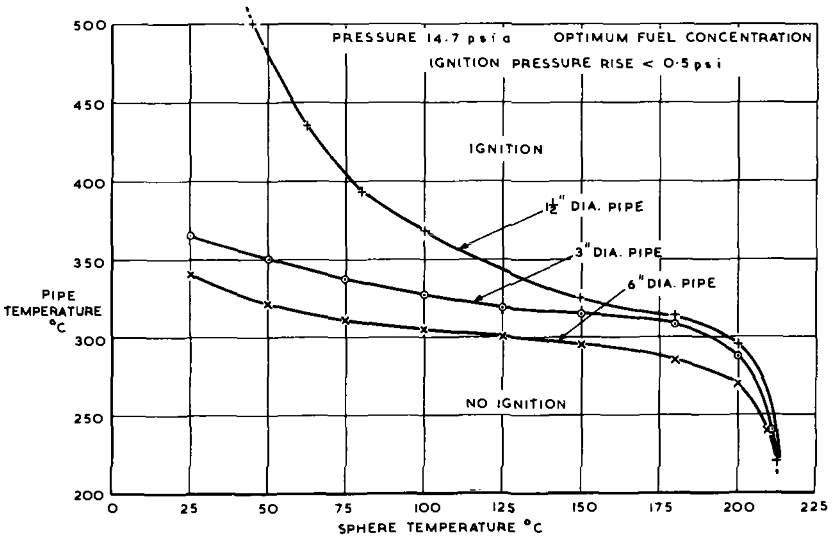

Fig. 4 Ignition boundaries for Avtur vapor on a hot pipe in an 18 in. sphere.

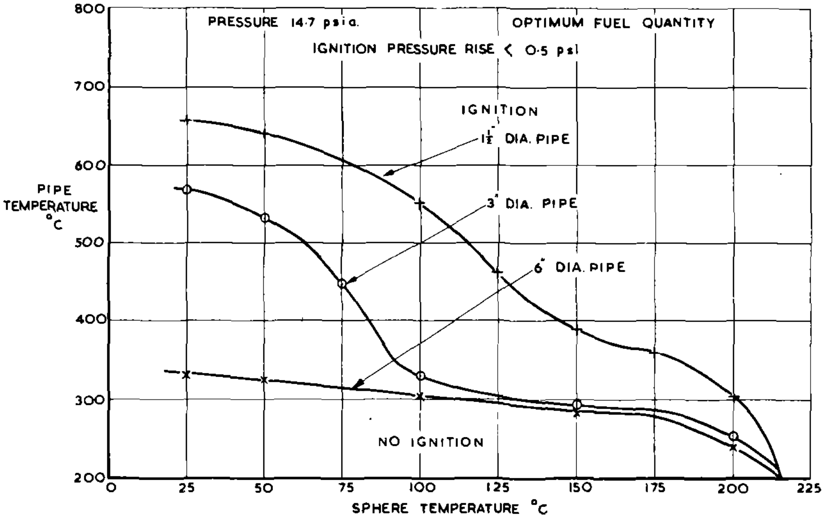

Fig. 5 Ignition boundaries for Avtur liquid on a hot pipe in an 18 in. sphere.

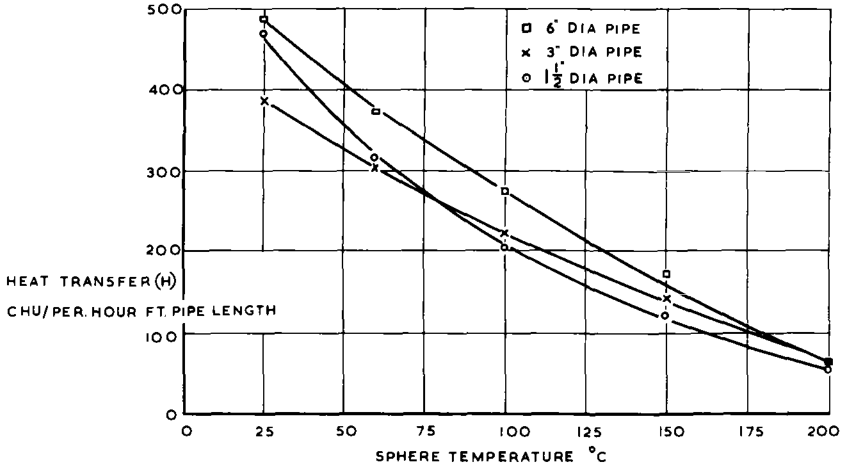

Fig. 6 Heat transfer from pipes at ignition threshold.

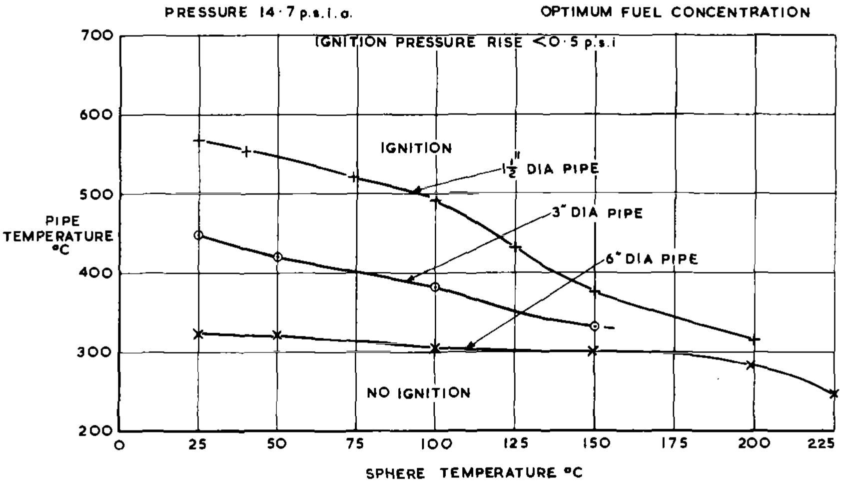

Fig. 7 Ignition boundaries for DTD 585 vapor on a hot pipe in an 18 in. sphere.

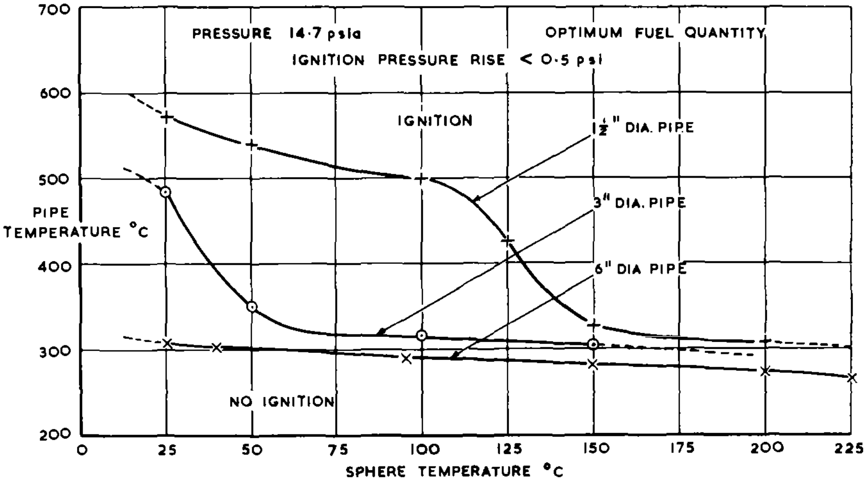

Fig. 8 Ignition boundaries for DTD 585 liquid on a hot pipe in an 18 in. sphere.

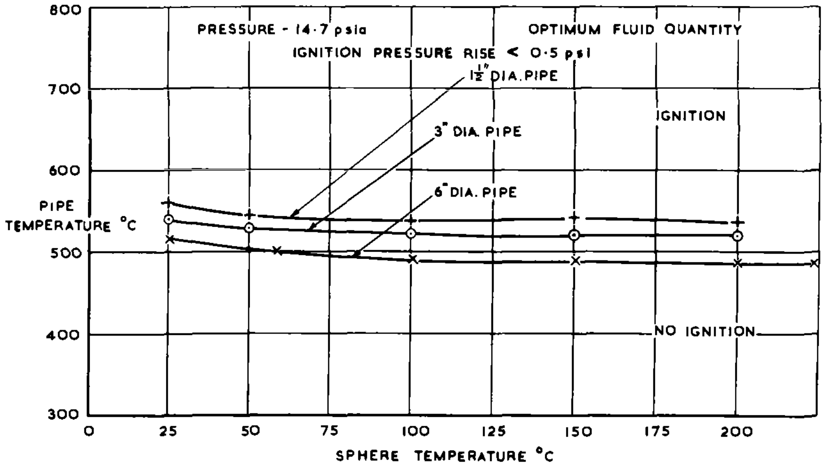

Fig. 9 Ignition boundaries for chlorinated silicone liquid ''
on a hot pipe in an 18 in. sphere.

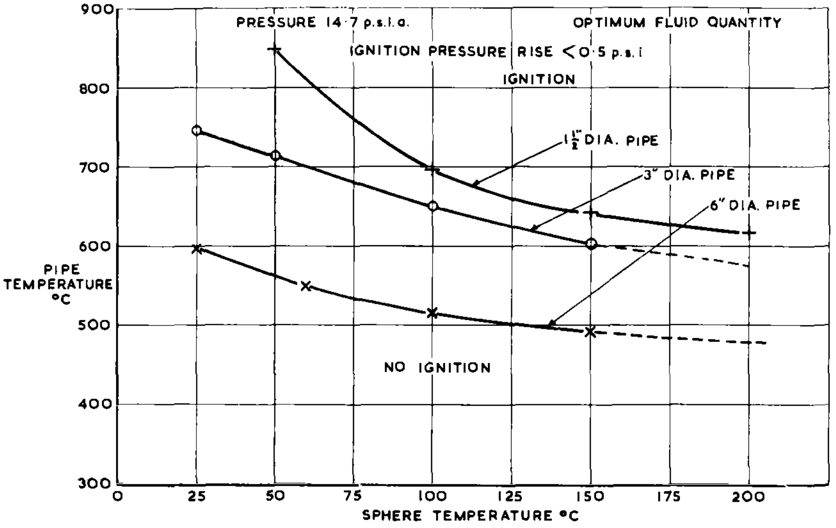

Fig. 10 Ignition boundaries for phosphate ester liquid on
a hot pipe in an 18 in. sphere.

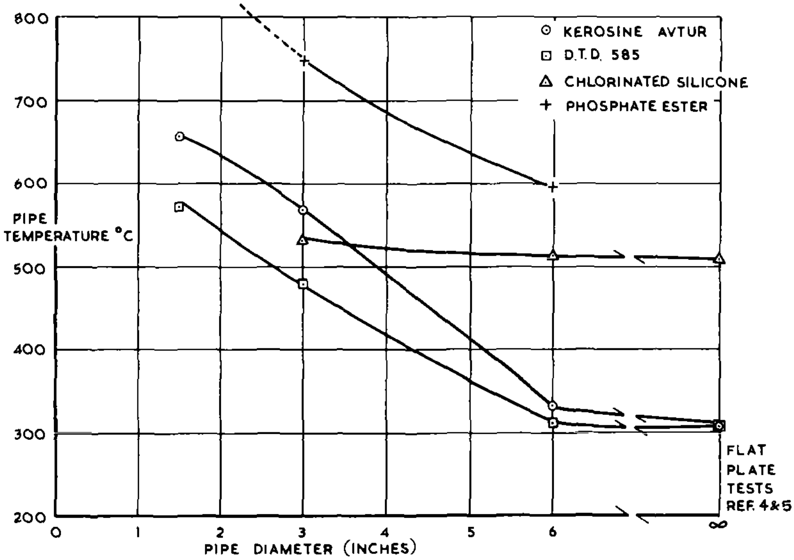

Fig. 11 Ignition boundaries (sphere temperature 25 C)

liquid dropped on to pipe.

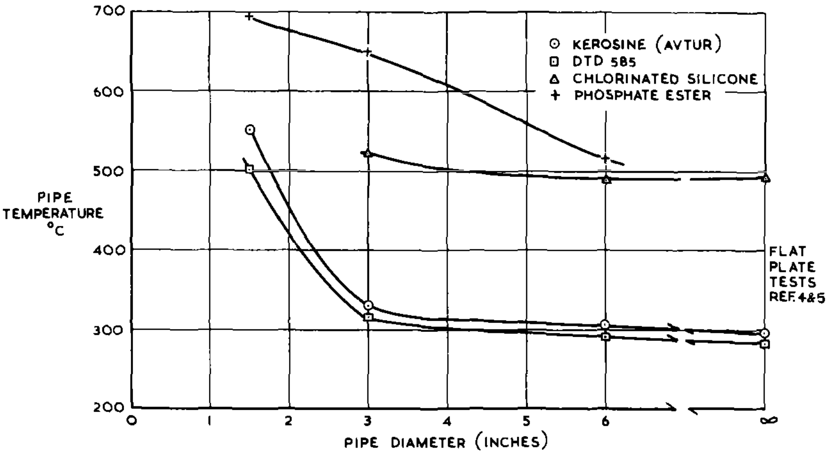

Fig. 12 Ignition boundaries (sphere temperature 100 C)

liquid dropped on to pipe.

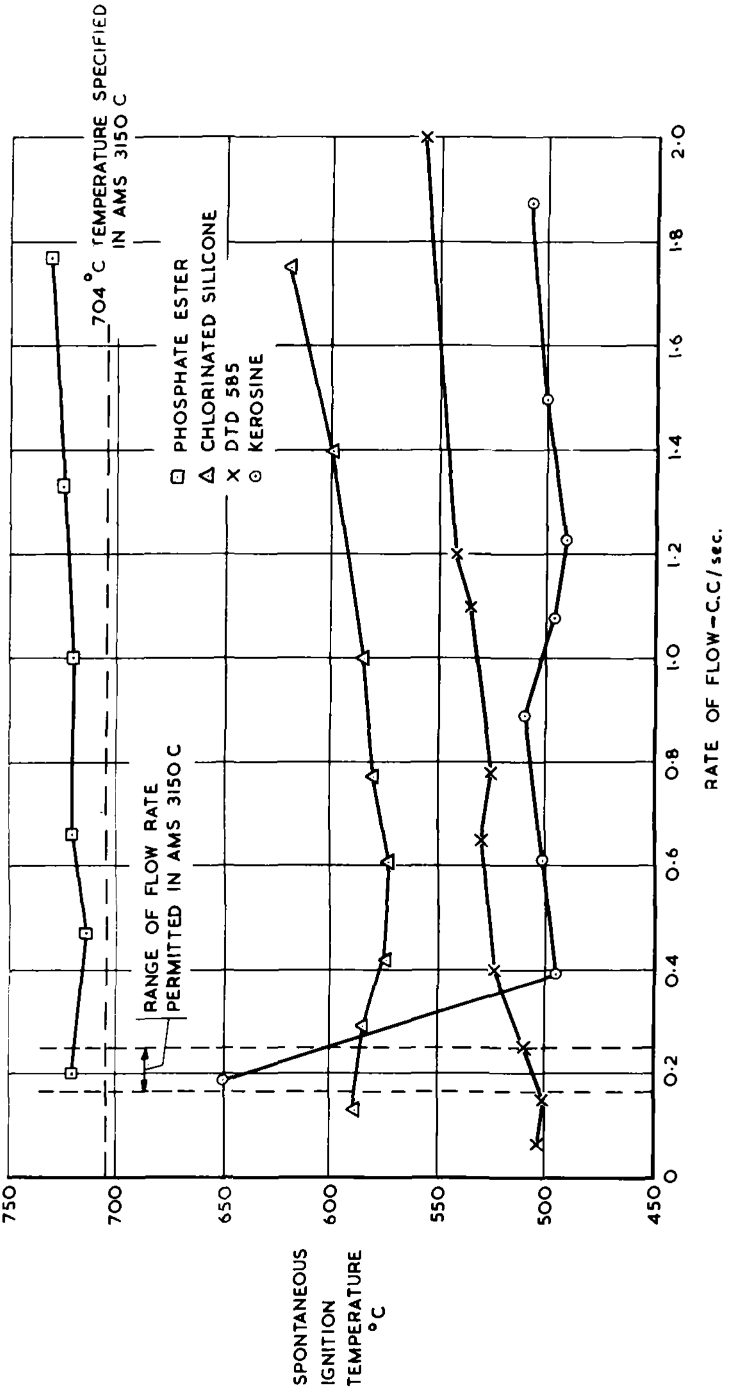

Fig. 13 Spontaneous ignition temperatures for various fluids using the hot manifold test rig AMS 3150 C.

THE IGNITION OF FLAMMABLE FLUIDS BY HOT SURFACES

By D. G. Goodall[1] and R. Ingle[2]

REFERENCE: D. G. Goodall and R. Ingle, "The Ignition of Flammable Fluids by Hot Surfaces," Fire Resistance of Hydraulic Fluids, ASTM STP 406, Am. Soc. Testing Mats., 1966.

ABSTRACT: The full-scale fire testing of aircraft power plants has suggested that laboratory closed-vessel spontaneous ignition tests give results which are pessimistic for many practical applications. The results of a number of test programs involving specific configurations of hot and cool surfaces have indicated that, for a particular fluid, it might be possible to establish a thermal correlation which would enable the risk of ignition to be calculated at the design stage.

Tests have been carried out on two rigs, both incorporating a hot bottom plate surface onto which the test fluid was sprayed, the side walls and top plate being somewhat cooler. The first rig was a static rig in which top and bottom plate temperatures and the gap between them was varied. The effects of mixture strength, atomization, and wall materials were studied for kerosine, and the ignition temperatures for various fluids have been determined for a limited number of configurations.

The second rig consisted of a small wind tunnel in which the effect of air velocities up to 10 ft/sec and altitudes up to 20,000 ft were investigated. It is shown that air velocities up to 3 ft/sec have a large effect on spontaneous ignition temperatures.

A correlation based on gas temperature and distance from the nearest wall shows good agreement for the static and wind tunnel rigs and for closed-vessel tests of other investigations. An outline of the future program of work is given.

KEY WORDS: spontaneous ignition, fire resistance, fire tests, flammability, hydraulic fluids, fuels, lubricants, kerosine, hot plate test, wind tunnel test

[1] Superior numbers refer to footnotes at the end of this paper.

In recent years, much attention has been given to spontaneous ignition problems because of the high skin temperatures of aircraft operating at high Mach numbers. The problem is not really new, however, since for many years aircraft engines have been operating with carcass temperatures above the laboratory closed-vessel spontaneous ignition temperatures for fuels and lubricants, and installation engineers have collected a good deal of ad hoc data to justify the safety of particular power plants. Fig.1 indicates the main risk areas for a typical subsonic by-pass jet installation.

Much of the data relating to aircraft power plants has indicated that freedom from spontaneous ignition is possible with individual surfaces as much as 400 F above the spontaneous ignition temperatures indicated by the laboratory closed-vessel tests. For example, it is generally accepted that spontaneous ignition of kerosine/air mixture is possible down to about 410 F, but we have found that, under certain practical operating conditions, kerosine can be sprayed on surfaces at 750 F and even 900 F without ignition taking place.

As a result of evidence of this kind, it was decided to investigate spontaneous ignition as a thermodynamic problem to see whether it might be possible to predict the limiting conditions for particular configurations on the basis of heat transfer calculations. It is appreciated that spontaneous ignition involves chemical problems of great complexity, but from the point of view of practical engineering, it is usually sufficient to know the maximum safe temperature for any particular configuration of hot and cool surfaces. Consequently, by limiting the number of variables involved and by concentrating on the determination of an envelope curve for each flammable fluid within which ignitions were not obtained, it was hoped to obtain results sufficiently consistent to be suitable for analysis by standard heat transfer methods and which might then be extrapolated and checked against data obtained on representative power plant configurations.

The ultimate objective for our work has been to enable spontaneous ignition risks to be calculated in the design stages of a new installation with a similar degree of confidence to that with which surface temperatures can be estimated. Most of the testing has been carried out using kerosine as the flammable fluid, to measure the effects of some simple changes in geometry, as well as changes in type of spray, mixture strength, altitude, and air velocity.

The conclusions presented are based on the results for kerosine, but sufficient work has been carried out on other fluids, such as ester-base lubricants, mineral base and silicone base hydraulic fluids, to give some idea of the application of the conclusions to these fluids.

Once again it must be emphasized that this paper describes an engineer's approach to the problem of spontaneous ignition. No attempt has been made to obtain values of absolute significance, but rather to derive a theory capable of predicting the effects of changes of environmental conditions once reliable laboratory data are available for a particular fluid under a particular known set of conditions.

Test Equipment and Procedure

General

So far, the investigation has been limited to two simple configurations, the first representing an enclosed, stagnant volume with a hot lower surface and rather cooler top surface and side walls, while the other represented a somewhat similar configuration with a small ventilating airflow passing through it. In each case the ignition risk was assumed to arise as a result of leakage of flammable fluid from a point in the upper surface. Thus, these first configurations have given the simplest possible simulation of ignition in the space between the upper surface of an aero engine and the adjacent cowling, due to a dribble of flammable fluid from a pipe running across the top of the engine. The effect of variation in gap between hot and cool surfaces has been investigated on a static hot plate rig, and the effect of variation in ventilating air velocity with a fixed gap has been investigated on a small wind tunnel with a heated floor section.

Description of Static Hot Plate Rig

The main features of the static hot plate rig are shown in Fig.2 and a photograph of the rig itself is shown in Fig.3. The rig provided an enclosed volume within which the flammable fluid under test was injected downward onto a heated bottom plate surface. Top and bottom plate temperatures could be controlled independently, and the side wall temperatures, which were not directly controlled, were measured by thermocouples embedded in them.

Considerable time was spent in developing this rig, to eliminate shortcomings shown up by inconsistent results in the early tests. In its final form, as shown in Figs.2 and 3, it consisted of an electrically heated bottom plate 11.5 by 7.75 in. made (for most of the tests) from 1 in. thick stainless steel, separated from an air-jacketed top plate by means of interchangeable side walls or sandwich pieces, which enabled the gap between top and bottom plates to be varied between 1 and 6 in. in increments of 1 in. These parts, which were made of mild steel, were lagged externally with asbestos, as were the exposed edges of the bottom plate. The top plate could be either heated or cooled by passing hot or cold air through the jacket. Surface temperature measurements were obtained from 5 thermocouples welded 0.040 in. below the surface of the bottom plate, 4 thermocouples welded flush with the under surface of the top plate, and 1 thermocouple in the side and rear walls of the sandwich pieces. All the thermocouples were of the chromel alumel type.

Provision for injecting the test fluid onto the bottom plate was by means of a hand-operated syringe, discharging through an air blast cooled stainless steel tube terminating in an 0.030 in. diameter orifice. Six disk-type pressure relief valves were situated around the base of the enclosed box to relieve any pressure build-up within the box, and, as will be seen from Fig.3, the whole rig was screened to reduce drafts to a minimum. Ignition was observed visually through mica windows in the lowest sandwich piece.

Extensive temperature measurements were made during the commissioning of the rig, and it was established that the temperature of the bottom plate could be varied over the range 390 to 930 F by varying the heating current, with a scatter in thermocouple readings of less than ±9 F and that the top plate temperature could be varied, with a similar degree of accuracy, over a range of 75 to 660 F by passing cold or hot air across its upper surface.

With the fuel injector nozzle replaced by a thermocouple probe, air temperatures were recorded at intervals of $\frac{1}{2}$ in. from the center of the bottom plate surface for gaps of 1, 3, and 6 in. with bottom plate temperatures of 392, 572, and 752 F and top plate temperatures of 122, 302, and 572 F.

For the determination of each spontaneous ignition limit a minimum of 10 repeat tests was carried out to confirm non-ignition, and in some cases as many as 20 to 30 checks were made to establish one point.

Test Procedure for Static Hot Plate Rig

The sandwich pieces were arranged to give a particular gap, the bottom plate temperature was fixed at a predetermined value, and the top plate temperature was allowed to settle to that produced by radiation from the bottom plate. Because of its large heat capacity, the rig was allowed to soak for approximately $1\frac{1}{2}$ hr to ensure stabilized temperatures throughout. A predetermined quantity of fuel was then injected and a period of 3 min allowed for ignition to occur. During this period, smoke and vapor could be observed coming slowly from the rig vents, but the occurrence of a hot flame resulted in an unmistakable explosiion, varying from a weak 'puff' to a sharp explosion depending on conditions. Cool flames (detected by the characteristic white exhaust fumes) which did not proceed to thermal ignition, were ignored.

If ignition occurred, the time lag between injecting the fuel and ignition was recorded and the top plate temperature progressively reduced in steps of 36 F until no ignition occurred (after at least 10 repeat tests). The top plate temperature was then increased in 9 F steps until ignition occurred at least once in 10 attempts. If, in the initial tests, no ignition occurred, this procedure was reversed in order to determine the highest top plate temperature giving no ignition in at least 10 successive tests. The box was purged by an air blast between successive tests.

Description of Wind Tunnel Rig

The wind tunnel rig, which was used to investigate the effects of varying air velocity and pressure with a fixed geometrical configuration, is shown diagrammatically in Fig.4 and a photograph of it in Fig.5. The tunnel was made from 3/16 in. thick nickel-chromium alloy, the working section being 5 ft 6 in. long, $6\frac{7}{8}$ in. wide, and $3\frac{7}{8}$ in. deep, and the last 18 in. of the tunnel floor being electrically heated to provide the 'hot surface' for ignition purposes. Temperature measurement was again by means of chromel alumel thermocouples, 3 of which were welded 0.040 in. above the tunnel top surface and 6 below the surface of the heated section of the tunnel floor. The heated section was enclosed in insulating firebricks to reduce heat losses and ensure reasonably uniform duct temperatures.

Since consistency of injection was felt to be more critical on this rig, the flammable fluid was supplied by an electrically driven pump through an electronically timed water-cooled fuel injector, incorporating an 0.039 in. diameter orifice located above the upstream end of the heated section. The electric heaters were capable of heating the tunnel floor to 1472 F, with a maximum scatter of ±9 F on the thermocouple readings, and radiation from the floor raised the tunnel roof temperature to a maximum of 1112 F. Maximum tunnel air velocities of just over 10 ft/sec were obtained under sea level conditions, the air being supplied at approximately room temperature.

By fitting an exhaust ejector to the tunnel and operating a variable restriction at the tunnel inlet, it was possible to obtain a similar range of air velocities at tunnel air pressures corresponding to altitudes up to 20,000 ft.

Test Procedure for Wind Tunnel Rig

The test procedure was generally similar to that for the static rig, the rig being allowed to soak at temperature for 2 hr before carrying out tests with progressively increasing airflows. The minimum airflow to suppress ignition was obtained to the nearest 0.1 ft/sec on the basis of no ignition in at least 10 tests. Ignitions were again determined visually, a typical example being shown in Fig.6.

The hot surfaces of both the static and the wind tunnel rig were cleaned periodically, and sufficient repeat tests were carried out to ensure that false results were not being obtained due to surface deposits.

Test Results

Fluids Tested

The following fluids were tested in the course of the program. In order to limit the duration of the program only kerosine was subjected to the full series of tests, the remainder were subjected to specific tests as described later.

Kerosine	-	D.Eng.R.D.2494
Wide-cut gasoline	-	D.Eng.R.D.2486
Ester-base lubricating oil	-	MIL/L/7808D
Mineral-base hydraulic fluid	-	DTD.585
Silicone-base hydraulic fluid	-	DTD.900/4725

Results Obtained on Static Hot Plate Rig

Fig.7 shows a typical set of results for the static hot plate rig and illustrates the determination of the limiting curve of top plate temperature versus bottom plate temperature for non-ignition of 1 cm^3 of kerosine injected with a 2 in. gap between top and bottom plates. This curve has been included to give an idea of the amount of testing carried out to determine each such curve by showing the cases where further tests were considered necessary because ignition occurred after a number of non-ignitions and also to show the relative small scatter for the boundary conditions. On the assumption that visual determination of ignition gives a satisfactory indication of any reaction liable to cause a fire, we consider the accuracy of our spontaneous ignition temperature determinations to be within ±9 F.

Figs.8, 9, and 10 show the spontaneous ignition boundaries determined by this method for aviation kerosine to Specification D.Eng.R.D.2494 (JP.1), wide-cut gasoline to Specification D.Eng.R.D.2486 (JP.4), an ester-base lubricating oil to Specification MIL/L/7808D, a mineral-base hydraulic fluid to Specification DTD.585, and a low-flammability silicone-base hydraulic fluid to Specification DTD.900/4725 when injecting through the 0.030 in. diameter orifice at a pressure of approximately 20 psig with gaps of 1, 3 and 6 in., respectively, between the top and bottom plates of the rig. Once again the curves are plotted to show the highest top plate temperature which could be achieved without causing ignition for each value of bottom plate temperature, and thus the region to the left of each curve represents the safe region from the spontaneous ignition point of view.

Since the tests were intended to investigate ignition on the bottom plate, it should be noted that very little work was carried out with the top plate the hotter of the two since, as will be discussed later, it is intended to include this in a later program.

In these tests, the volume of mixture injected was varied in proportion to the enclosed volume, to give a nominal mixture strength of three times the stoichiometric value, since other work had shown this ratio to give the lowest ignition temperature. It was, of course, appreciated that the actual mixture strength would vary widely within the rig, particularly when the ignition delay times were short, and in order to obtain some idea of the change in ignition temperature which would result from a change in nominal mixture strength, further tests were carried out in which the volume of fluid injected was held constant so that the nominal mixture strength was inversely proportional to the enclosed volume. Fig.11 shows the effect (for aviation kerosine) of mixture strength for a 3-in. gap, the effect being generally similar for other gaps. These tests showed that the effect of mixture strength on ignition temperature was very small with rich mixtures of two to eight times stoichiometric but confirmed that the minimum value occurred with mixture between three and four times stoichiometric.

Another variable investigated on the static hot plate rig was fluid atomization. Most of the work was carried out with a fixed gap of 3 in., and the results for kerosine are summarized in Fig.12, while those for the ester-base turbine engine lubricating oil and the mineral hydraulic fluid are shown in Fig.13. Unfortunately, no corresponding data have yet been obtained for the silicone-base hydraulic fluid. The 0.030 in. diameter orifice had been found to give a plain jet of liquid during the injection period, and to obtain finer atomization it was replaced by a swirl atomizer incorporating a 0.016 in. diameter metering orifice. This atomizer was also arranged to spray downward onto the bottom plate, and, although no attempt was made to obtain a quantitative measure of the degree of atomization obtained, the 'minimum atomization' was arbitrarily defined as being that given by 30 psi pressure difference and 'maximum atomization' was that given by 200 psi pressure difference across the jet.

All the fluids tested showed significant changes in spontaneous ignition temperature with degrees of atomization, although comparison of Figs.12 and 13 shows that the relationship is not a simple one, since it was reversed when comparing the ester-base lubricant with the other two fluids.

The lower bottom plate spontaneous ignition temperatures (Fig.13) obtained with a low degree of atomization with kerosine is thought to be due partly to the scattering of small droplets over a larger area of the bottom plate, thus giving higher heat transfer rates into the liquid fuel, and partly to a better mixing of air and fuel vapor at the bottom plate surface. However, with a high degree of atomization, ignition temperatures are higher than those for the liquid jet and the low atomization spray which is due to a reduction of penetration to the surface of the bottom plate. This has two effects: (1) the mixture strength in the boundary layer at the bottom plate will be reduced, and (2) the heat required to evaporate the fuel in the air is derived from the air instead of the bottom plate. The latter effect will cause steepened temperature profiles near the bottom plate and thus call for a higher bottom plate temperature to give ignition.

Referring once again to Fig.13, this shows that for the ester-base oil, a higher degree of atomization invariably resulted in a lower ignition temperature. This is thought to be merely due to the resultant increase in the number of small droplets obtained for this heavier and more viscous fluid giving a higher rate of heat transfer and evaporation from the surface of the bottom plate, there being little evaporation in the air due to the very low vapor pressure.

The physical properties of the mineral-base hydraulic oil DTD.585 are nearer to those of kerosine, and the results obtained show trends similar to those for kerosine. It must be pointed out that all our analytical work has been based on the lowest spontaneous ignition temperatures measured, these in the case of kerosine, being with 'minimum atomization'.

Finally, some comparative tests were made using kerosine at three times stoichiometric concentration with a 3 in. gap and the 'minimum atomization' condition, to compare ignition temperatures for different bottom plate surfaces. The results of these tests are shown in Fig.14. From this, it will be seen that the effect of the hot surface material was surprisingly small, the painted aluminium plates giving ignition temperatures slightly (16 F) lower than those for stainless steel, and the anodized and machined aluminium plates giving slightly (22 F) higher temperatures than stainless steel.

Results Obtained on Wind Tunnel Rig

For the tests on the wind tunnel rig it was decided to inject a fixed quantity of kerosine (3.6 to 3.8 cm^3) in all cases, in order to limit the number of variables involved, and since the results on the static rig had already indicated that, for rich mixtures, mixture strength was unlikely to be critical. Different degrees of fuel atomization were obtained by injecting through an 0.039 in. diameter orifice at pressures of 50, 500, and 1000 psig, the corresponding periods of injection being 1.1, 0.4, and 0.28 sec, respectively, to give the same total injection quantity. Fig.15 shows the results obtained at sea level for the three injection pressures, the ignition boundary being expressed in terms of the minimum air velocity to prevent ignition for any given temperature of the heated floor section so that the area to the left of the curve constitutes the 'safe' region. It will be seen that large increases in spontaneous ignition temperature were obtained with small increases in airflow, especially at the lower end of the range.

Similar tests were carried out with the tunnel internal pressure reduced to simulate altitudes up to 20,000 ft, and a typical set of results for three heated floor section temperatures of 1100, 1277, and 1436 F, with an injection pressure of 500 psig is given in Fig.16. This graph illustrates how the effect of air velocity in inhibiting spontaneous ignition becomes even greater with increasing altitude and shows a limiting altitude for each temperature above which ignition is not possible even without airflow.

Fig.17 is a carpet graph relating all the results obtained with 500 psig injection pressure, including those for sea level conditions already given in Fig.15. In the interests of simplicity, no attempt has been made to include individual test points on this carpet plot, nor are the corresponding results for the tests where injection was at 50 and 1000 psig included, since these do not differ greatly in form and are not necessary for the purpose of our main argument.

Discussion

Examination of the results presented in the paper leads one to a number of interesting conclusions:-

As expected, large differences in spontaneous ignition temperatures were obtained for the different fluids tested, varying from a minimum of 482 F for kerosine, with hydraulic fluid DTD.585 approximately 9 F higher, up to approximately 750 F for the silicone-base hydraulic fluid DTD.900/4725 in the static rig with equal top and bottom plate temperatures (Figs.8 to 10).

In the second place, after allowing for the difference in chemical effect associated with the use of different fluids, the results have once more confirmed the extreme sensitivity of spontaneous ignition to environmental conditions, but at the same time have shown that the ignition boundary may be determined with quite a high degree of repeatability provided the conditions are sufficiently closely controlled (Fig.7). This conclusion we have found somewhat surprising in view of the generally non-homogeneous temperature conditions in our test rigs and also in view of the deliberate lack of preheating of the injected flammable fluids. Such factors as mixture strength and wall temperature, for example, were deliberately arranged to be far from uniform, the latter giving rise to freely circulating convective airflows. All these factors are appropriate to practical engineering applications but would usually be carefully eliminated by a conscientious laboratory worker, so that the repeatability of our results has been encouraging.

Thirdly, as has already been pointed out, the tests have indicated that, for fluid injected in liquid form with nominal concentrations richer than stoichiometric, nominal mixture strength does not have an important effect on the spontaneous ignition limits determined (Fig.11). It is presumed that this is because, with this technique, local variations of mixture strength near the point of impingement of droplets on the hot surface almost cover the range from zero (pure air) to infinity (pure liquid) and thus provide sufficient mixture at the appropriate concentration for the minimum spontaneous ignition temperature. It may be remarked here that in all cases the heat capacity of the hot surface onto which the fluid was injected was considered to be large relative to that of the injected fluid, so that the minimum amount of chilling of the hot surface took place. Thus, the results obtained should represent the minimum temperatures for spontaneous ignition appropriate to each configuration, and many practical cases involving continuous fluid leakage would require somewhat higher temperatures to give ignition due to local chilling of the hot surface.

Somewhat surprisingly, except in the case of the silicone-base hydraulic fluid, deposits on the hot surfaces were light and gave little change in the spontaneous ignition temperature. Also, the tests on different plate surfaces and surface finishes in the static rig showed that these introduced a scatter of the order of only ±18 F as compared with a machined stainless steel plate, an aluminium painted surface giving the lowest temperature and an anodized light alloy plate the highest.

The results are considered to have shown, therefore, that when assessing the risk of spontaneous ignition in a given engineering application, the important parameters to be considered are the type of flammable fluid which may be present together with the normal heat transfer factors of size and shape of enclosure, surface temperatures, and air pressure and velocity.

So far, time has permitted an analysis along these lines to be carried out only on the data obtained for kerosine at 14.7 psia. It is hoped ultimately to demonstrate that the theory applies equally to other fluids and ambient pressures, but more test evidence will be necessary to enable this to be done.

Many previous workers have, of course, discussed the problem of spontaneous ignition from a thermal viewpoint. It has been pointed out by Jost [3], for example, that the lower limit for spontaneous ignition of a mixture can be considered as being that temperature at which the rate of heat output from the chemical reaction begins to increase more rapidly than the rate of extraction of heat by the surroundings.

It was considered that, for the static rig, radiation was likely to be the major mode of heat transmission, and calculations were, therefore, carried out to determine the net radiant heat flow between the walls of the enclosure and a small spherical volume of mixture near the center of the bottom plate surface to compare the equilibrium gas temperatures near the hot plate (assumed to be the temperature at which the radiant heat flow was zero for the limiting conditions with various configurations).

The results of these calculations are shown in Fig.18 for kerosine with a 3 in. gap between top and bottom plate surfaces. Similar curves are included in Fig.18 for the radiation heat balance at the center of spherical vessels 10 and 18 in.

diameter, respectively, as used in recent tests at the Royal Aircraft Establishment [4], assuming the gas temperature to be varied while the wall temperatures are held constant at values corresponding to the lowest spontaneous ignition temperatures observed.

For the static hot plate rig, the calculations have been carried out for an element of gas 0.1 in. from the surface of the hot plate, and it will be seen that over almost all of the range of conditions a radiant heat balance was obtained at a gas temperature of 470 F, but as the top and bottom plate temperatures became similar the gas temperature at balance rose to 485 F. The radiant heat balance at the center of 18 and 10 in. spheres occurs at temperatures of 406 and 414 F, respectively, showing that some scale or shape factor must be used to correlate more closely the spontaneous ignition temperature of homogeneous enclosures and enclosures with differential wall temperatures on the basis of zero radiant heat flow. However, a rough correlation exists in that, at ignition boundaries, the gas temperature for zero radiant heat balance lies between 406 and 482 F for rich kerosine/air mixture over the range of enclosure sizes and shapes investigated, including the heated spheres, while for the hot plate rig alone the correlation is much closer.

In other words, this work suggested that a rich kerosine/air mixture would ignite in an enclosure comprising a large heat source once a small volume of the mixture had been heated to a minimum temperature of the order of 406 to 482 F. The fact that the enclosure incorporated a bottom surface much hotter than this would not necessarily cause ignition provided other walls of the enclosure were sufficiently cool to give a radiant heat balance for the mixture close to the hot surface at a mixture temperature below the range given above.

While this correlation may prove to be satisfactory for certain practical applications where, under more or less stagnant air conditions, flammable fluid drips down on to a hot surface, further consideration shows that it has some obvious shortcomings. The most important of these is that it ignores heat transfer by conduction and convection, which are important in certain configurations, such as when the upper surface is the hotter of the two and when airflow is present as in the wind tunnel rig.

The explanation for the scale effect reported by several workers probably lies in the inhibiting action of walls of the vessel, as described by Cullis, Fish and Gibson [5]. This results in a need to modify the simple heat balance theory to include the proposition that, while there is for each mixture a fundamental spontaneous ignition temperature, this would only be achieved in the center of an infinitely large container, and, due to the effect of the walls in terminating the chain reactions essential for ignition to proceed, this fundamental temperature must be somewhat higher if the distance of the point of ignition from a wall is reduced. This theory offers an explanation of the higher spontaneous ignition temperatures reported for smaller diameter spherical or cylindrical vessels and also for hot surfaces with steep temperature gradients close to their surface.

As the following analysis shows, using this theory, it is possible in principle, at least, to predict the spontaneous ignition temperatures for a wide variety of configurations by plotting isotherms adjacent to each hot surface and comparing the temperature at any distance from the wall with that required to overcome the inhibiting effect caused by the chain-breaking action of the wall itself.

Analysis

Assumptions

Let us first assume that there is a temperature at which chemical reaction occurs at a sufficiently fast rate to produce an explosion at an infinite distance from a wall under the best mixture strength for the chosen constituents. This temperature is given the symbol T_o and is reckoned to be independent of pressure over a limited range of pressures.

Secondly, let us assume that near a wall of a given material the wall has an inhibiting effect on ignition such that

$$T_g = T_o + f \text{ (distance from the wall)} \qquad (1)$$

Where T_g = gas temperature at ignition point in deg F, and
f = some function to be determined.

This implies that, with homogeneous mixtures and temperatures, ignition will occur at the greatest possible distance from the wall.

For non-homogeneous temperature distributions, a comparison of the ignition temperatures determined from (1) with a curve of gas temperature versus distance from the wall will determine whether an explosion risk obtains.

Investigation of Rig Results

Homogeneous Temperature Rigs

A recently published report by the Royal Aircraft Establishment, Farnborough [4] gives results reproduced in Fig.19 of a very extensive series of tests on stainless steel cylindrical and spherical vessels ranging from 1 to 18 in. diameter using kerosine fuel D.Eng.R.D.2494 (JP.1) injected into air in vapor form.

Replotting these results against $r^{-\frac{1}{2}}$ in Fig.20 (where r is the distance from the nearest wall in inches) gave the following approximate formula

$T_g = T_o + k\,r^{-\frac{1}{2}}$ .. .. (2)

Where T_o = basic spontaneous ignition temperature at an infinite distance from any wall (deg F),

T_g = spontaneous ignition temperature at distance r from the nearest wall (deg F), and

k = a constant.

It was found that, over the pressure range 6 to 14.7 psia, T_o remained constant at 380 F, while k = 82.5 at 14.7 psia and increased with reduction in pressure.

No quantitative analysis has yet been attempted of the pressure effects either in these closed vessel tests or on the Rolls-Royce wind tunnel rig (Fig.17). However, both sets of results indicate that ignition temperatures increase with altitude, thus confirming the trend reported by other workers [3].

Rolls-Royce Static Hot Plate Rig

As already mentioned, this rig featured a box-type enclosure with a solid bottom plate at temperature with, in general, cooler top plate and side walls. Kerosine fuel to Specification D.Eng.R.D.2494 (JP.1) was injected at 50 to 59 F. All tests were carried out at atmospheric pressure and all results are uncorrected for barometric pressure.

The temperature profile for the air within the enclosure was measured at the vertical centerline for various combinations of top and bottom plate temperatures, and from these measurements a typical series of temperature profiles was estimated near the center of the hot bottom plate. These profiles are plotted against $r^{-\frac{1}{2}}$ in Fig.21. The curves show that convection currents within the chamber gave quite sharp temperature gradients near the bottom plate. The boundary of the ignition temperature profiles shown in Fig.22 is given by the formula:-

$T_g = 390 + 22\,r^{-\frac{1}{2}}$ .. .. (3)

Where r is the distance from the bottom plate in inches.

This form shows remarkable agreement with basic ignition temperature T_o given in Eq (2), but the constant k = 22 is approximately one quarter of that for the closed chamber results.

Rolls-Royce Wind Tunnel Rig

In this rig the working section $1\frac{1}{2}$ ft long was preceded by a calming section 4 ft long, but flow straighteners were not fitted. Calculation of temperature profiles at the end of the working section showed that for most of the tests the flow was turbulent with a laminar sub-layer and that ignition must have been occurring in the sub-layer, since the temperature gradient was very steep and the temperature had fallen to 400 F or below at the sub-layer boundary with wall temperatures up to 1472 F.

Of course, with longer heated surfaces the laminar sub-layer temperature gradient will become less steep, the temperature at the sub-layer boundary will increase, and ignitions will occur at lower gas temperatures farther away from the wall.

In comparing the results from the wind tunnel rig with those for the static hot plate rig, it is important to bear in mind that on the static rig, ignition delay times of up to 3 min were allowed. In the case of the wind tunnel rig, however, the maximum delay times corresponded to the transit times for the mixture passing over the hot plate, which in turn depended on the velocity of the laminar sub-layer. Thus the ignitions obtained were generally associated with short delay times.

Fig.23 shows curves of ignition temperature versus delay time obtained on the Rolls-Royce static and wind tunnel rigs as well as a curve from R.A.E. Report [4]. This latter curve clearly shows a negative temperature coefficient region (between 698 and 878 F) where there is believed to be a change in the mechanism of chemical reaction, also described by Cullis et al [5], and this region is within the temperature range covered by the wind tunnel tests. Fig 23 also shows the transit time of the elemental layer at the chosen ignition point in the wind tunnel rig which is generally similar but higher than that for the closed vessel tests. This discrepancy is believed to be the time required to heat and vaporize the liquid fuel. The similarity between the two curves indicates that transit time also has a controlling or limiting effect on ignitions.

Two curves of ignition gas temperatures are plotted against $r^{-\frac{1}{2}}$ in Fig.24, the first being that at chosen ignition points and corresponding to the transit times shown in Fig.23 and the second showing ignition gas temperatures at transit times chosen arbitrarily as one second longer than the delay times for the closed vessel tests. The boundary curve has the formula:

$$T_g = 383 + 36\, r^{-\frac{1}{2}} \quad .. \quad .. \quad .. \quad .. \quad (4)$$

again agreeing with the basic ignition temperature (T_0) from the previous results, but with k only approaching half the value of k = 82.5 for the closed vessel tests.

In general it should be realized that an increase in heated surface length allows ignition to take place at a lower temperature due to long transit times and at the same time generates higher mixture temperatures at greater distances from the walls. With a very long heated surface we could expect ignition temperatures approaching those of a closed cylindrical vessel of similar diameter to that of the duct. However, for practical engine installations most hot surfaces are relatively short in the direction of airflow, and here, we would expect that the short transit times indicate any possible ignition point to be of high temperature very close to the wall which, of course, implies a high wall temperature to give ignition.

No explanation has been found for the term $r^{-\frac{1}{2}}$, but as this is a boundary of a range of temperature/distance relations it may have to remain an empirical term.

To summarize, we would expect that the basic ignition temperature (T_O) can be derived directly from laboratory closed-vessel tests over a range of vessel sizes. The choice of a limiting value of k is more difficult since we have shown that k varies from 22 in the static hot plate rig, through 36 in the wind tunnel rig, to 82.5 in closed-vessel tests for kerosine fuel. However, it is believed that the low value of k for the static hot plate rig is due to the wrong choice of position for measurement of the temperature profile. It is to be expected that ignition was occurring nearer the edge of the hot plate where circulation currents were less active and therefore the temperature gradients less steep. Further measurements of temperature profiles are to be carried out in the near future to confirm this.

Fig.25 shows the three ignition temperature boundary lines plotted against $r^{-\frac{1}{2}}$ for comparison purposes, and it is recommended that for practical applications the constant k used should be taken for the present as one third of the k derived from laboratory tests with closed vessels, that is, for kerosine $k \approx 28$, and later work will be aimed at defining these constants more closely.

Conclusion

The results of this work have shown quite clearly that the risk of spontaneous ignition in any given application is determined by the temperature of a critical volume of mixture rather than by a hot surface temperature and that the surface temperature is a controlling factor only in so far as it affects the temperature of any flammable mixture. Thus it is possible to justify current aircraft practice in which it is considered safe to operate with local surfaces much hotter than closed-vessel ignition tests would indicate. Of course, where all the surfaces of the enclosure are at high temperatures as in supersonic aircraft spaces or where a ventilation airflow temperature is high, spontaneous ignition will be possible at temperatures approaching those of closed vessels of similar size. However, it may be possible to raise the ignition limit by consideration of transit time in the above cases, but any stagnant regions will constitute an ignition hazard at temperatures near those for closed vessels.

Calculations of mixture temperature gradients adjacent to hot surfaces may yet prove too complicated to be practicable, particularly where incidental or deliberate airflows are passing over an irregularly shaped surface such as that formed by a flanged pipe joint.

Future Work

It is obviously necessary to carry out additional tests, to check the effects of additional configurations, involving heated side walls and upper surfaces, as well as to do tests with airflows over longer heated surfaces and with hot airflows. This work is to be put in hand shortly, and it is hoped to obtain further confirmation of the tentative theory which has been put forward.

Acknowledgments

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Footnotes

1. Development Engineer, Fuel System and Fire Precautions Dept., Rolls-Royce Limited, Derby, England.

2. Section Leader, Fire Precautions Dept., Rolls-Royce Limited, Derby, England.

3. W. Jost and H. O. Croft, Explosion and Combustion Processes in Gases, McGraw-Hill Book Co. Inc., New York, N.Y., 1946.

4. J. A. MacDonald and R. G. White, 'Spontaneous Ignition of Kerosine Fuel Vapour : the Effect of Vessel Size', Technical Report No. 65138, Royal Aircraft Establishment, Ministry of Aviation, Farnborough, Hants., England, 1965.

5. C. F. Cullis, A. Fish and J. F. Gibson, 'The Oxidation of Hydrocarbons : Studies of Spontaneous Ignition in Small Vessels', Proceedings of the Royal Society 1965, Series A, Vol. 284, No. 1396.

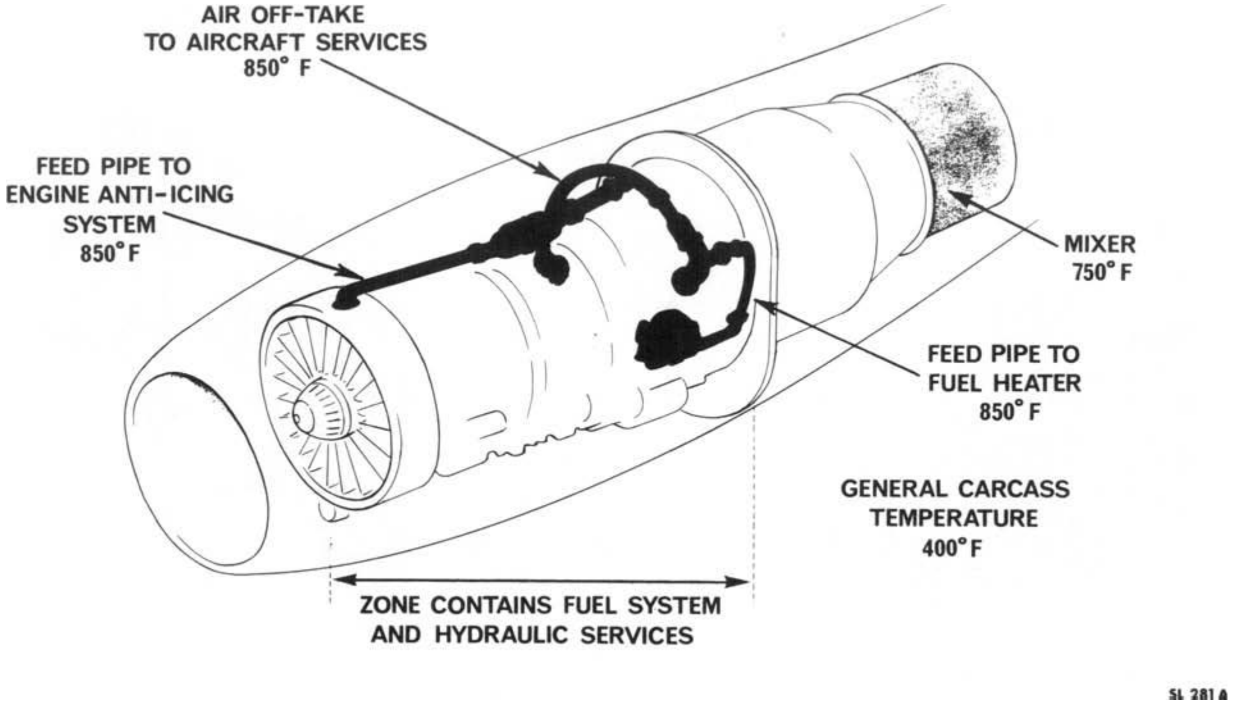

Figure 1. Typical by-pass engine surface temperatures.

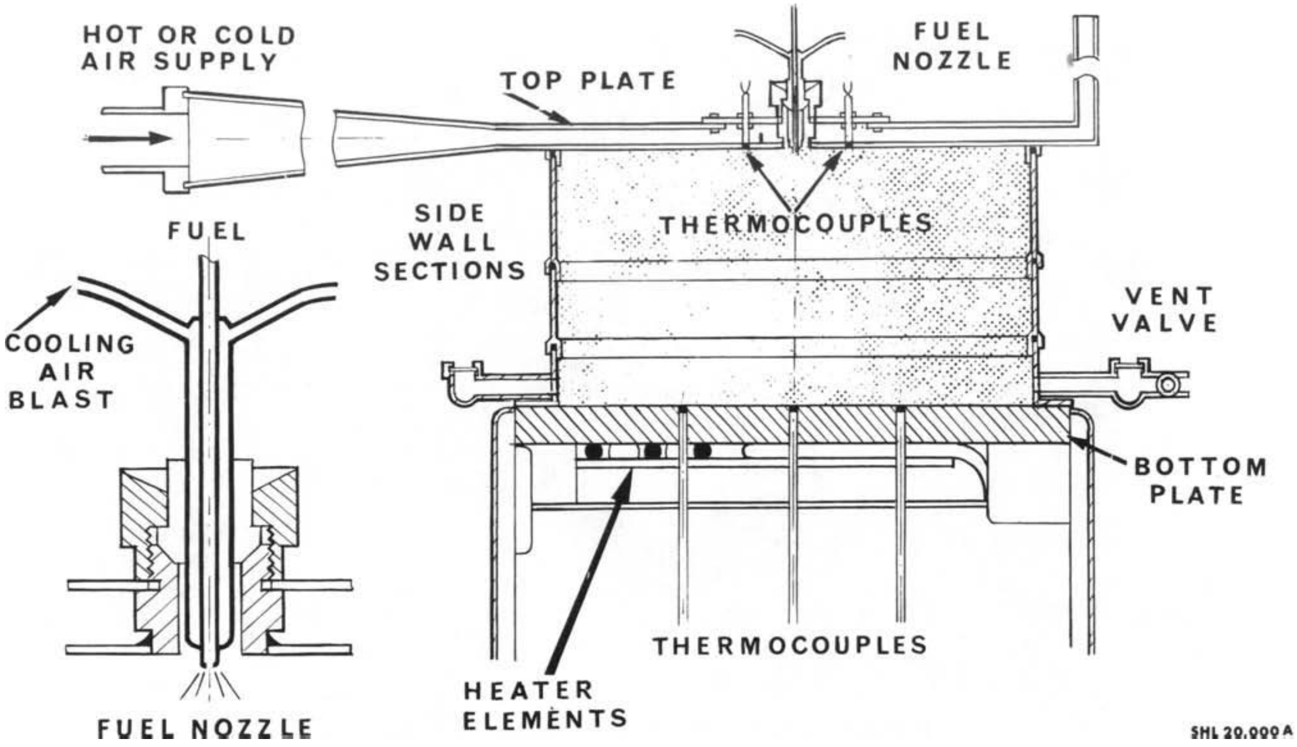

Figure 2. Schematic diagram of hot plate rig.

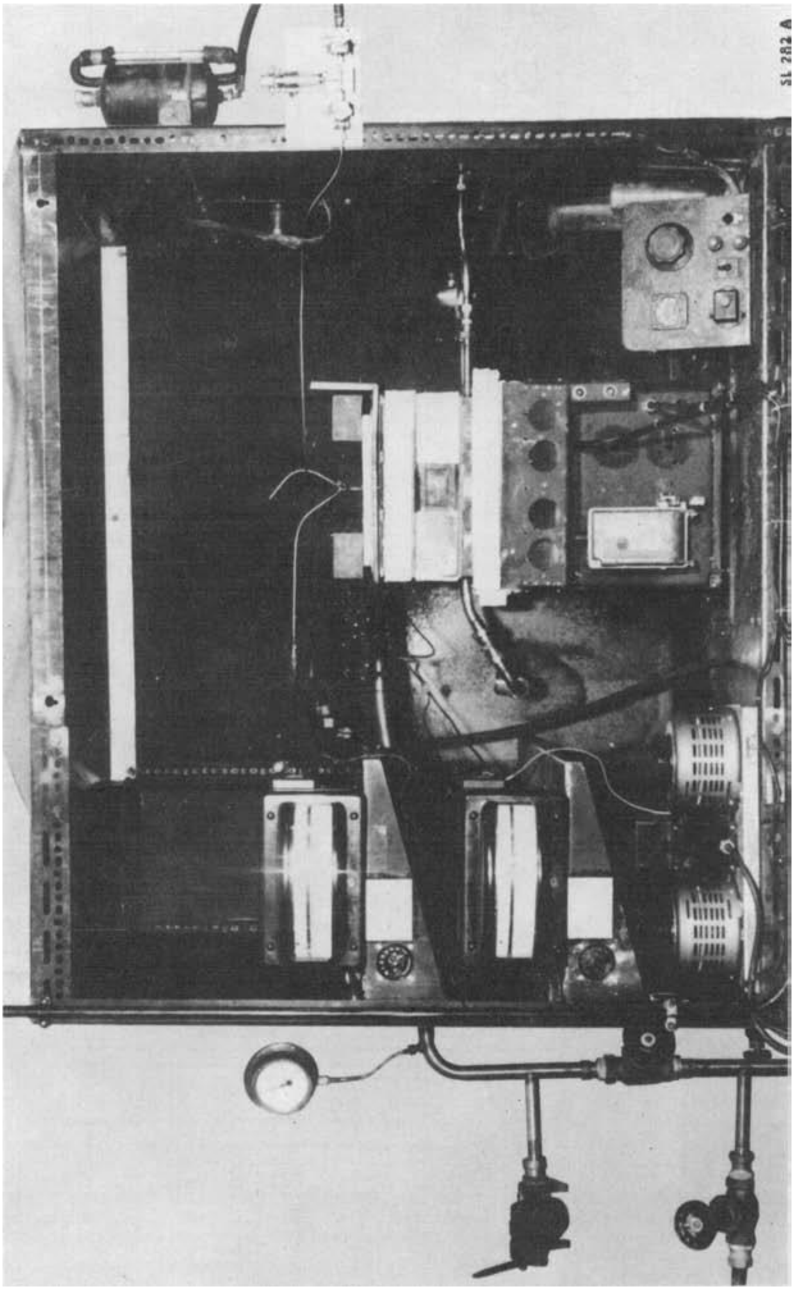

Figure 3. Static hot plate rig.

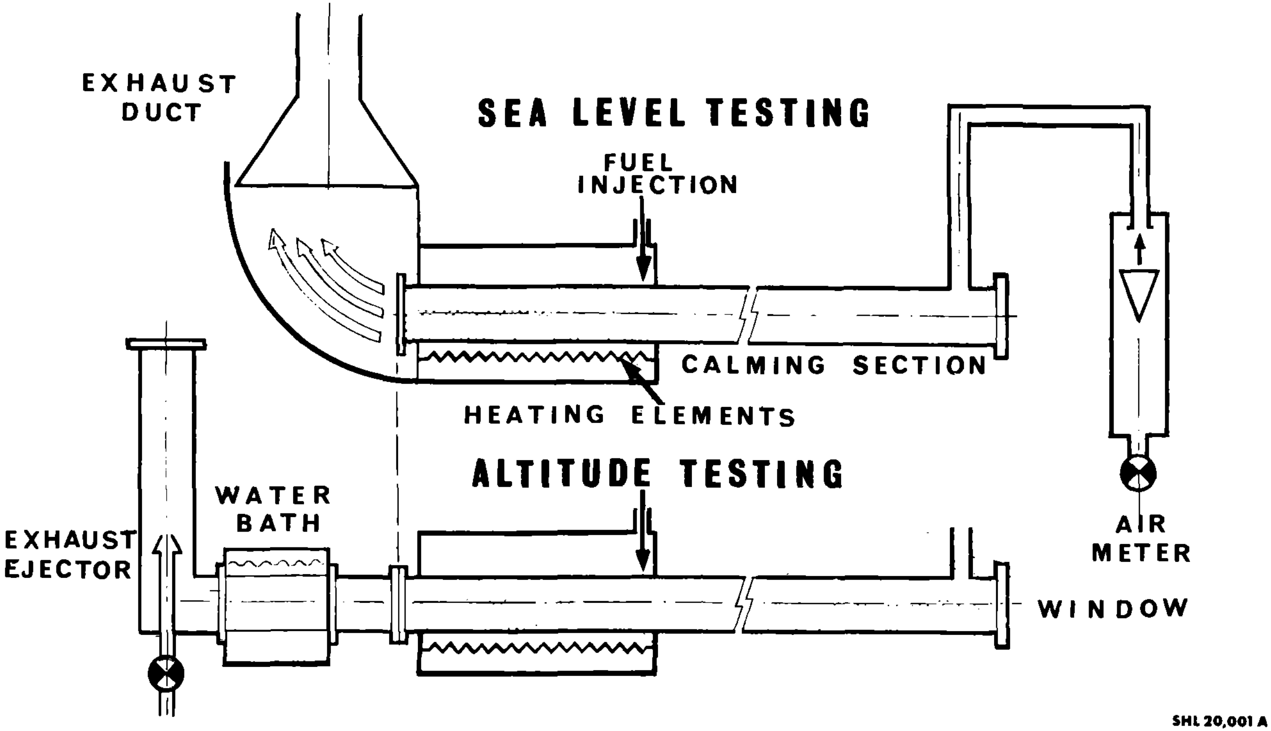

Figure 4. Schematic diagram of wind tunnel rig .

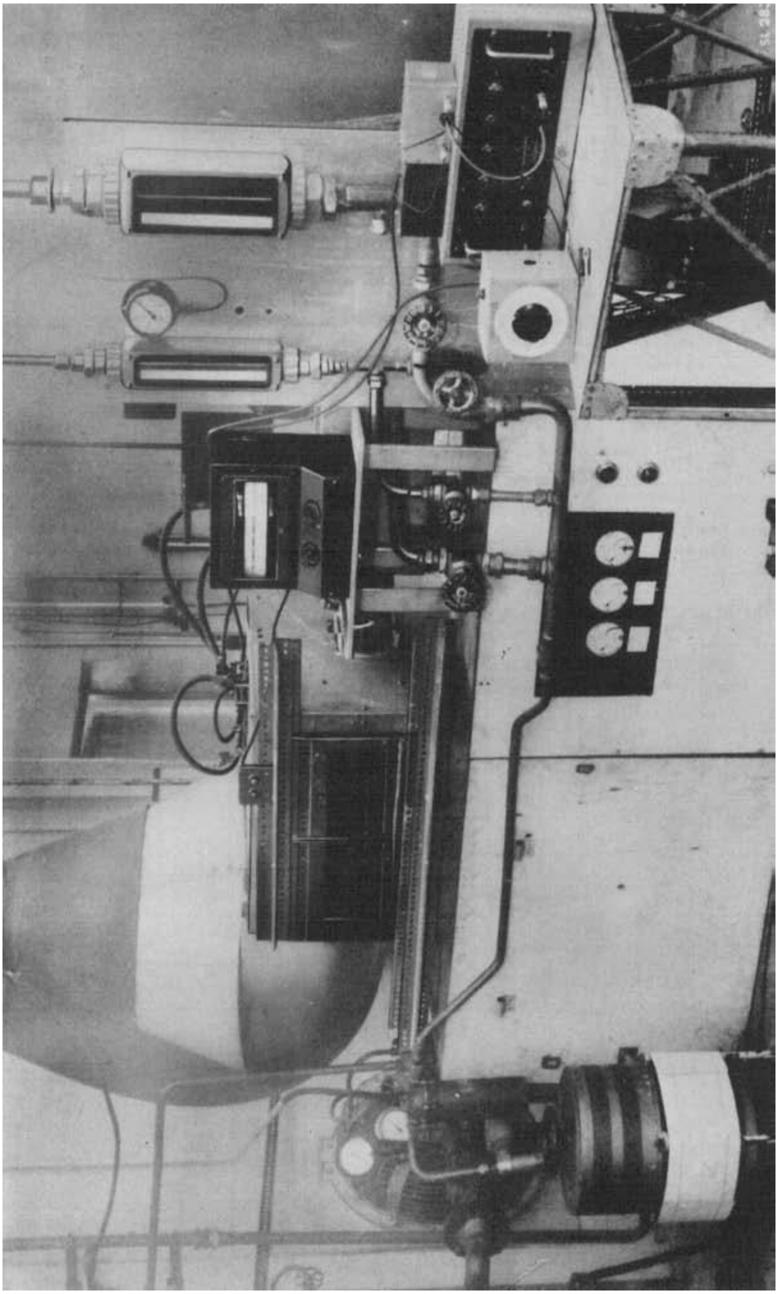

Figure 5. Wind tunnel rig.

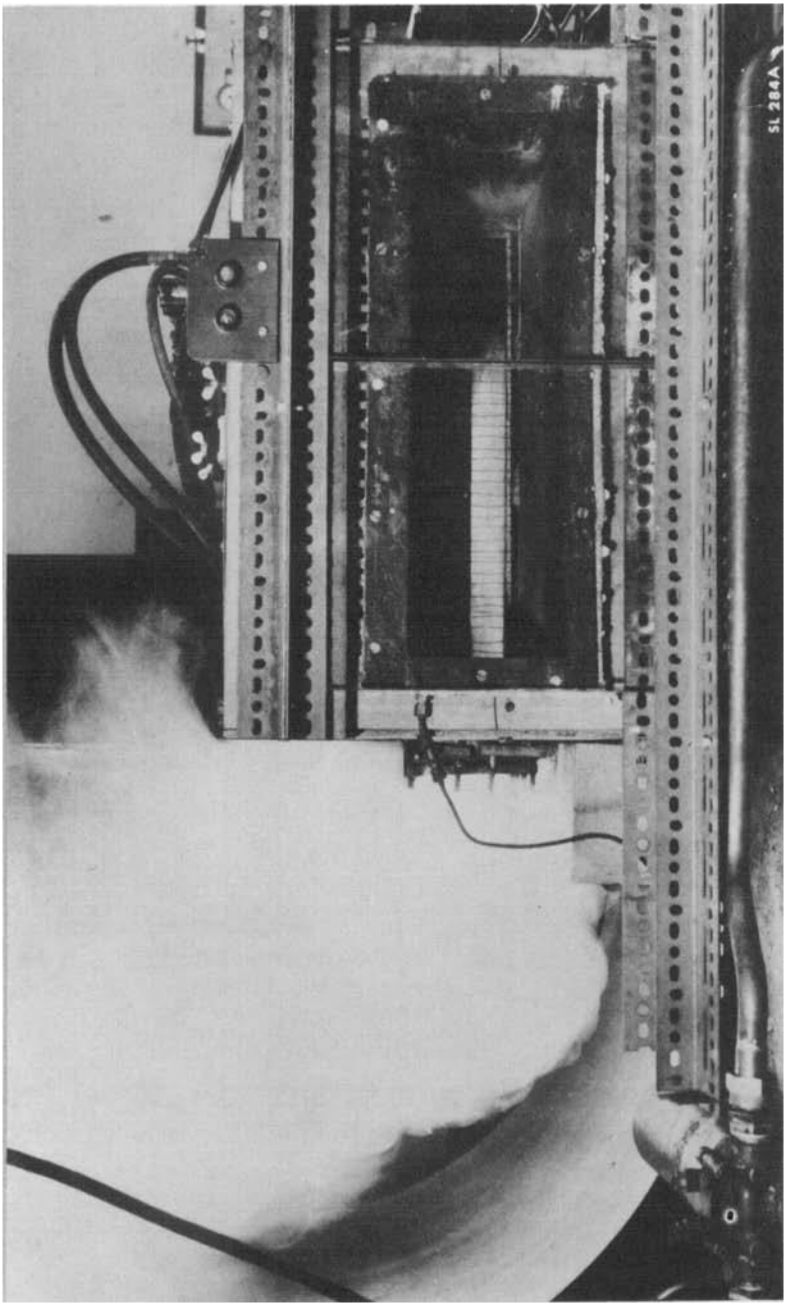

Figure 6. Wind tunnel rig - Typical ignition.

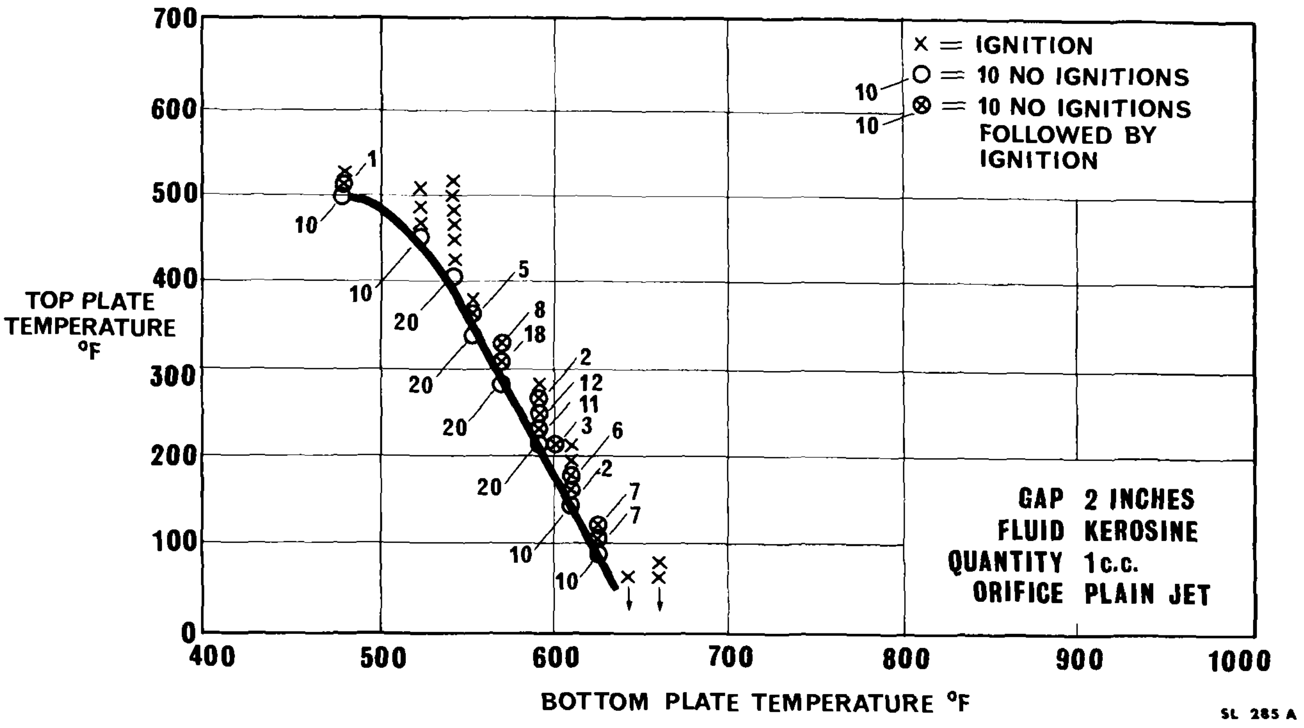

Figure 7. Static hot plate rig - Method of ignition boundary determination.

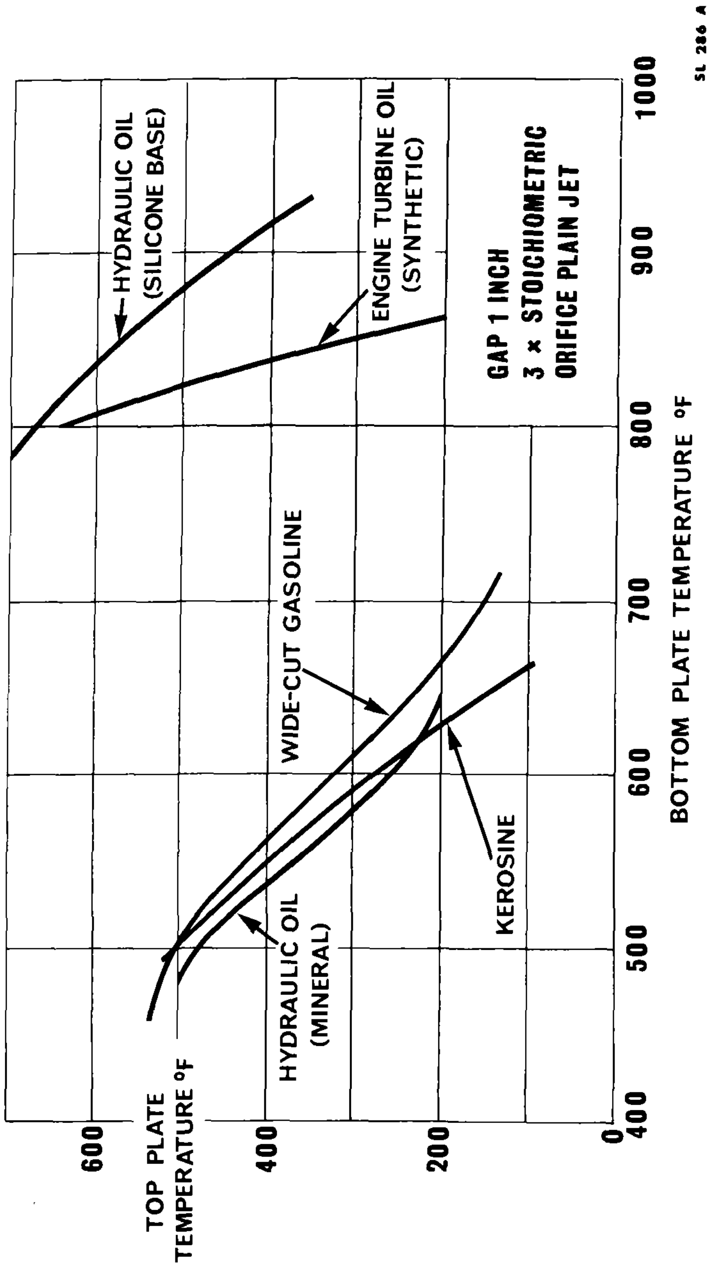

Figure 8. Static hot plate rig - Ignition boundaries for various fluids.

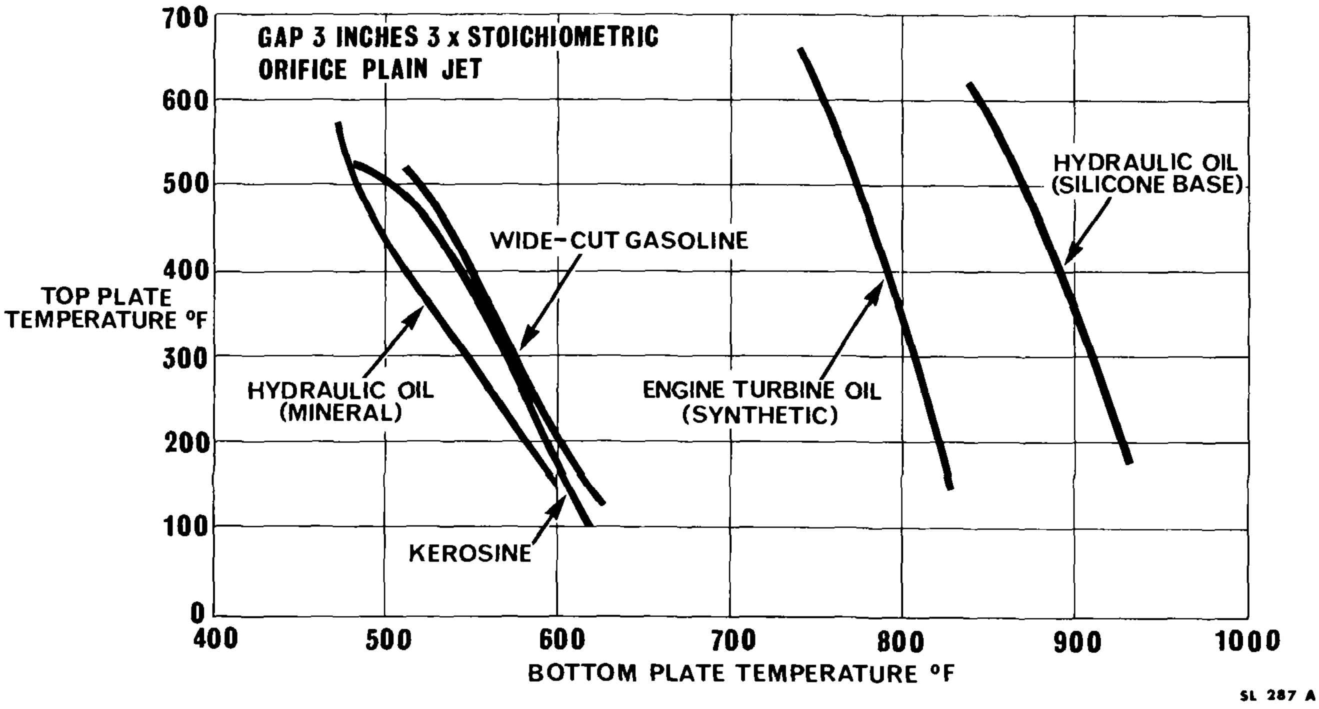

Figure 9. Static hot plate rig - Ignition boundaries for various fluids.

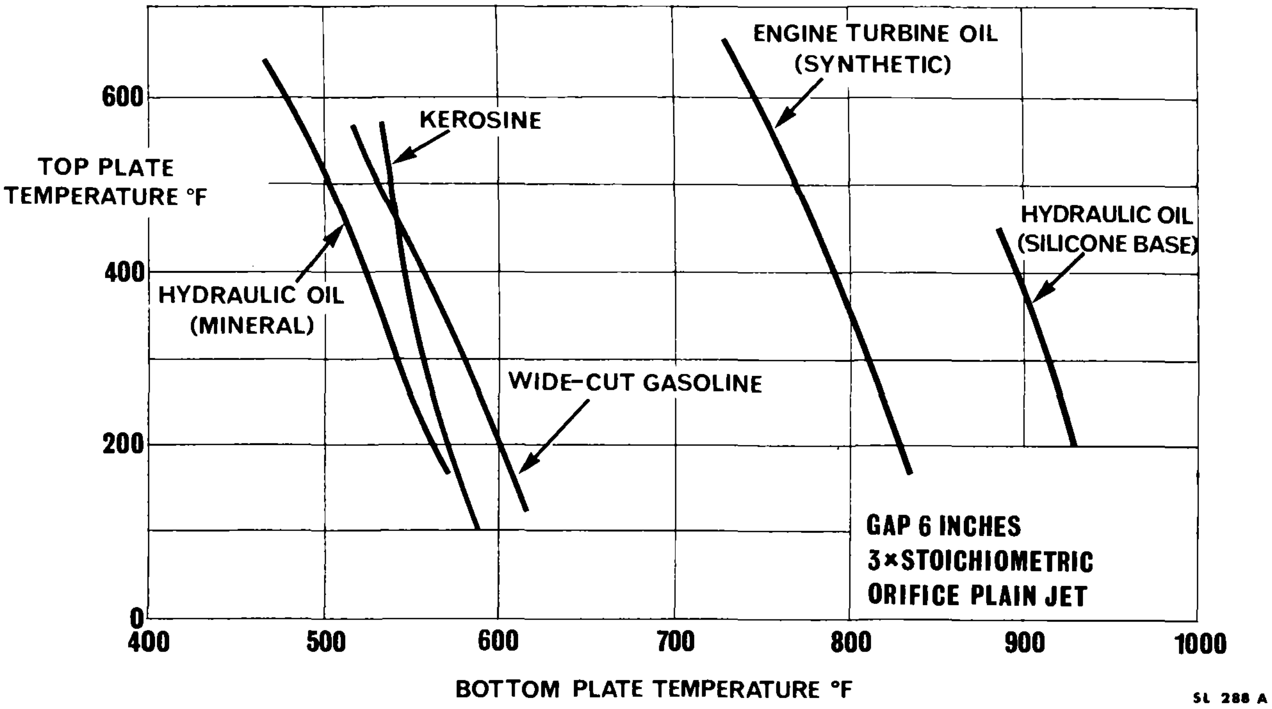

Figure 10. Static hot plate rig - Ignition boundaries for various fluids.

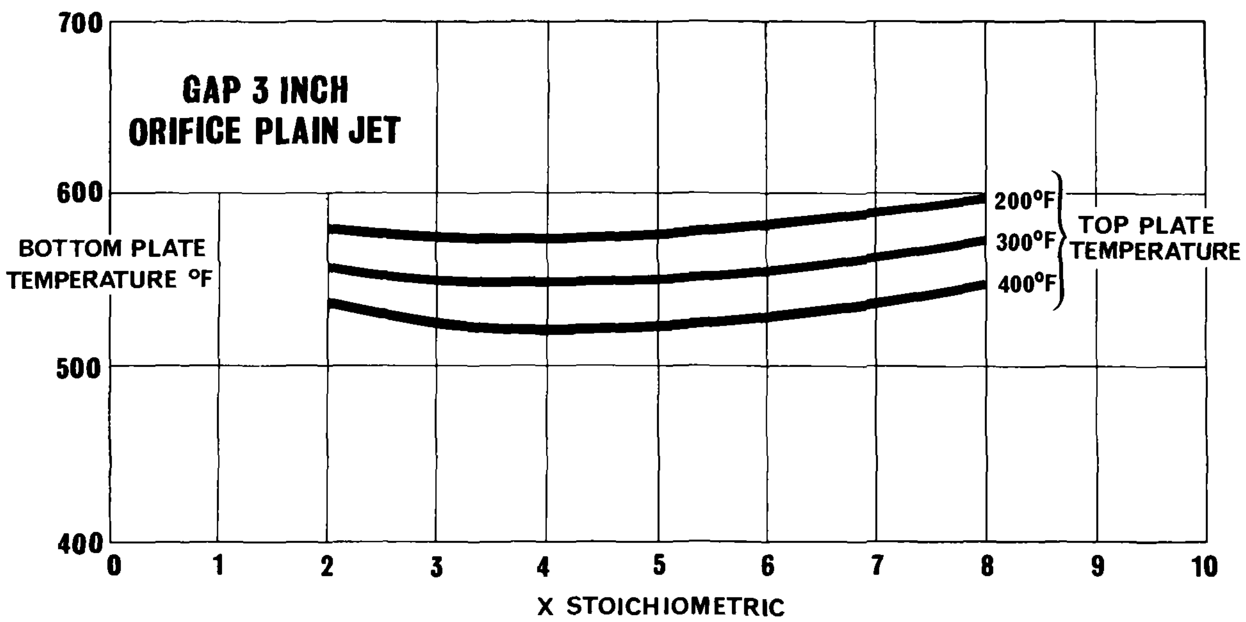

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Figure 11. Static hot plate rig - Effect of mixture strength.

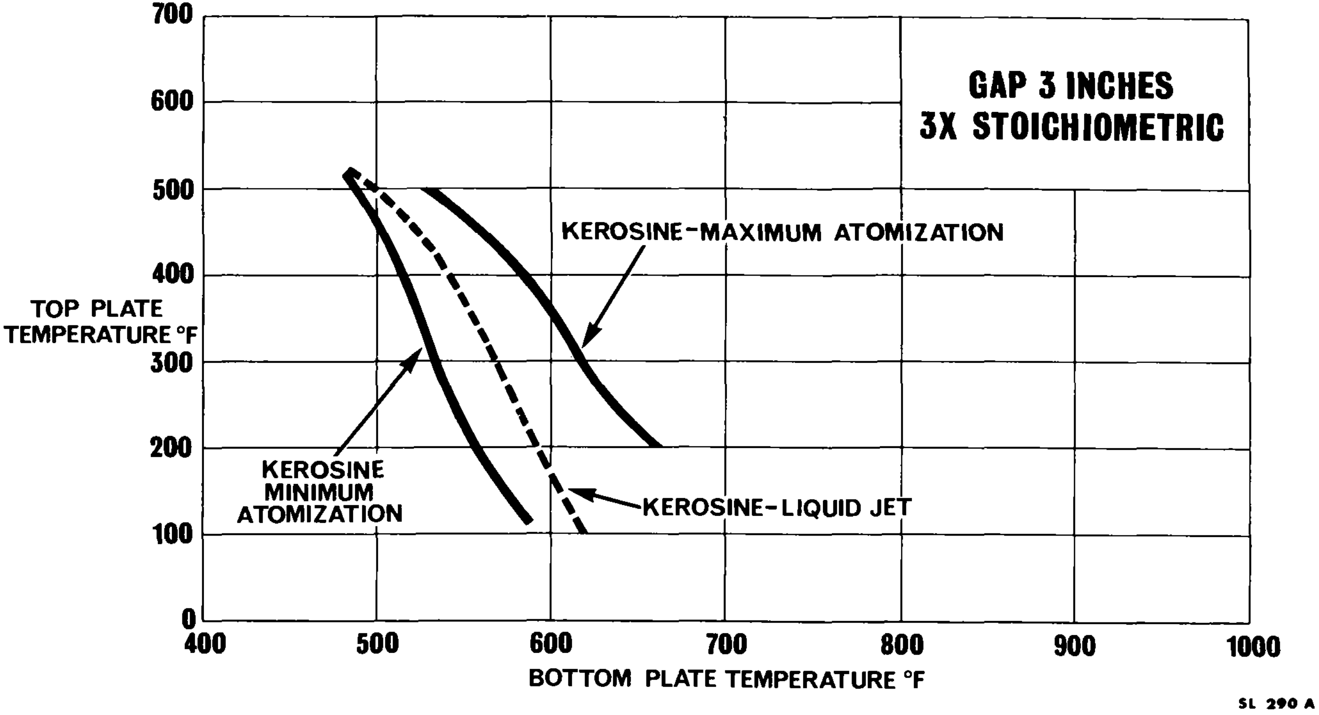

Figure 12. Effect of fluid atomization.

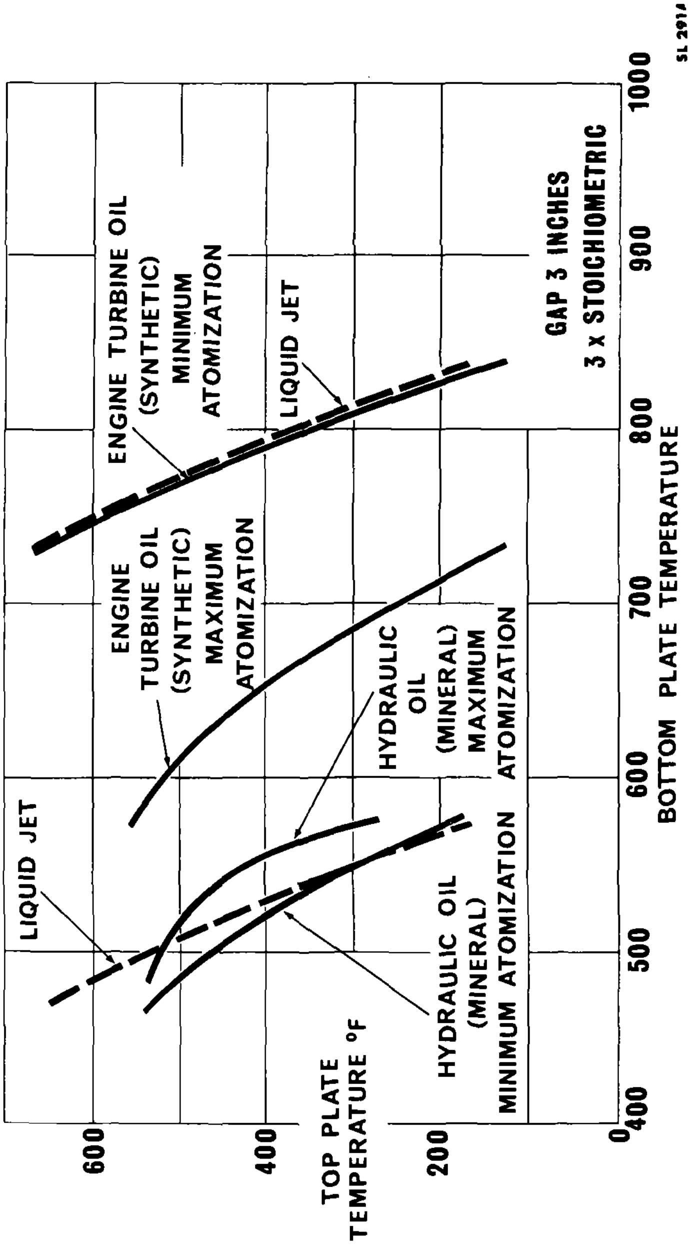

Figure 13. Effect of fluid atomization.

PLATE SURFACE	TOP PLATE TEMPERATURE °F							
	150	200	250	300	350	400	450	500
STAINLESS STEEL FOR COMPARISON	577	561	547	536	525	516	504	491
ALUMINIUM ALLOY PLATE AS CUT ALUMINIUM ALLOY PLATE ANODISED	581	570	561	556	547	536	520	496
ALUMINIUM ALLOY PLATE WITH ALUMINIUM PAINTED SURFACE	561	549	538	529	522	510	496	482
ALUMINIUM ALLOY PLATE WITH BLACK PAINTED SURFACE	572	560	545	534	525	514	504	491

KEROSINE GAP 3 INCHES MINIMUM ATOMIZATION 3X STOICHIOMETRIC

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Figure 14. Bottom plate ignition temperature deg F.

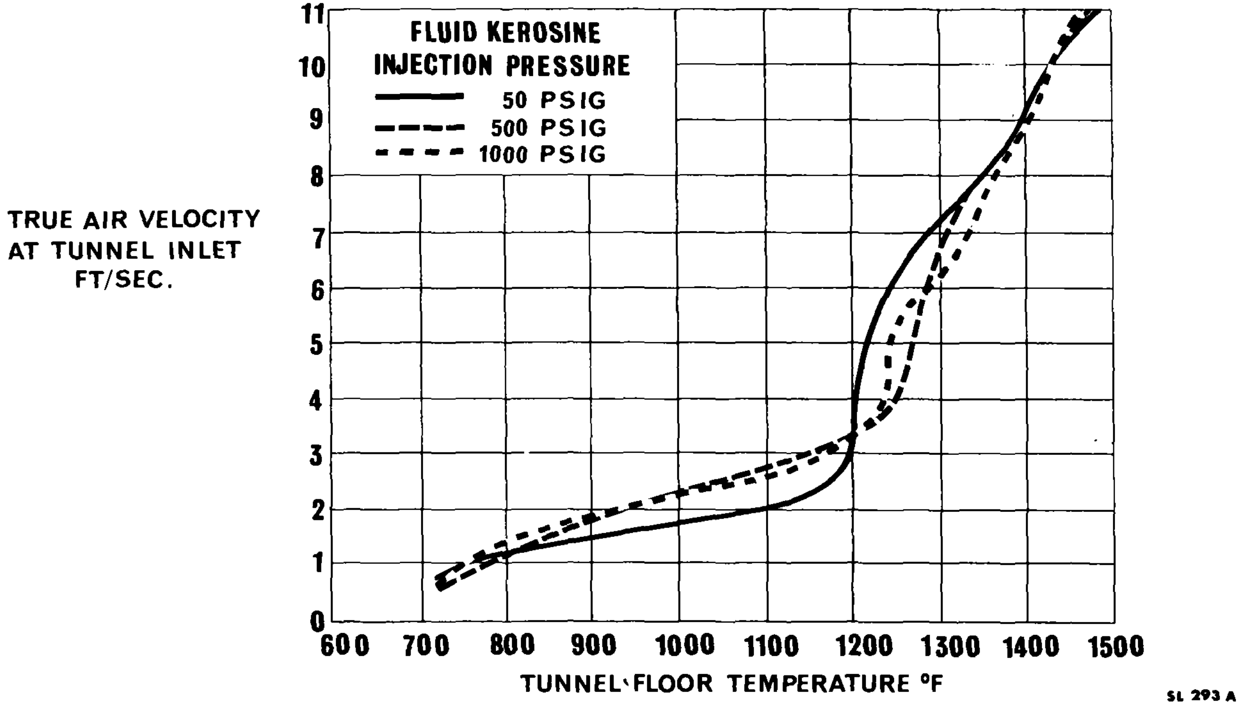

Figure 15. Wind tunnel rig - Ignition boundary at sea level.

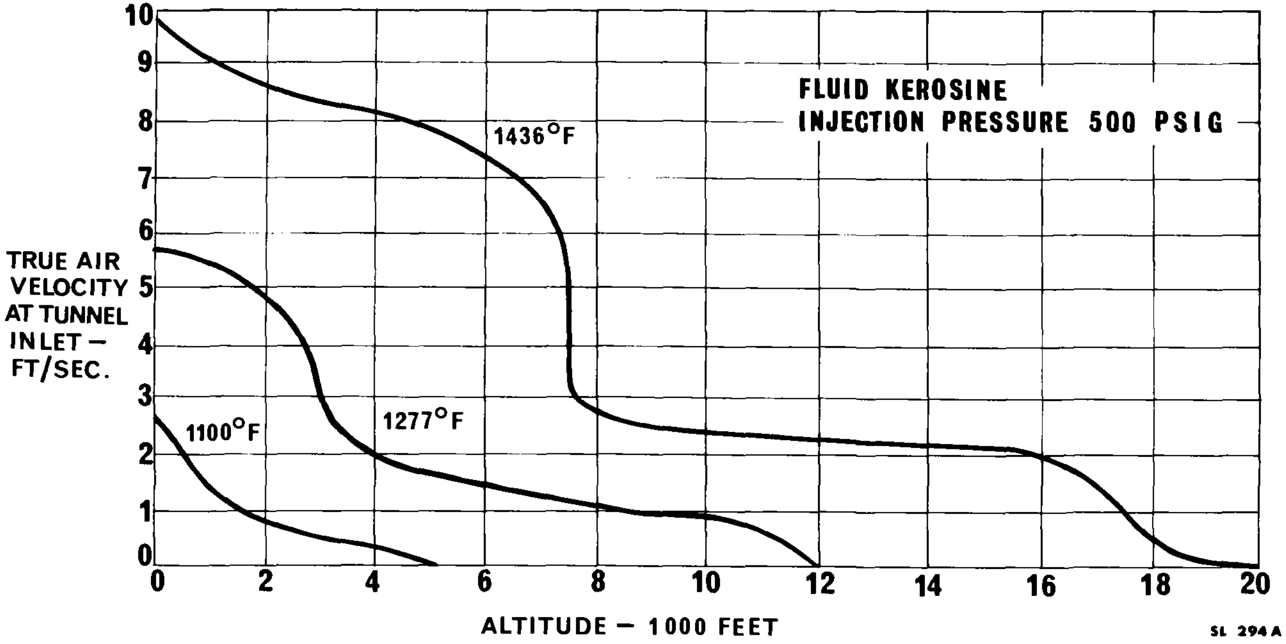

Figure 16. Wind tunnel rig - Ignition boundary at altitude.

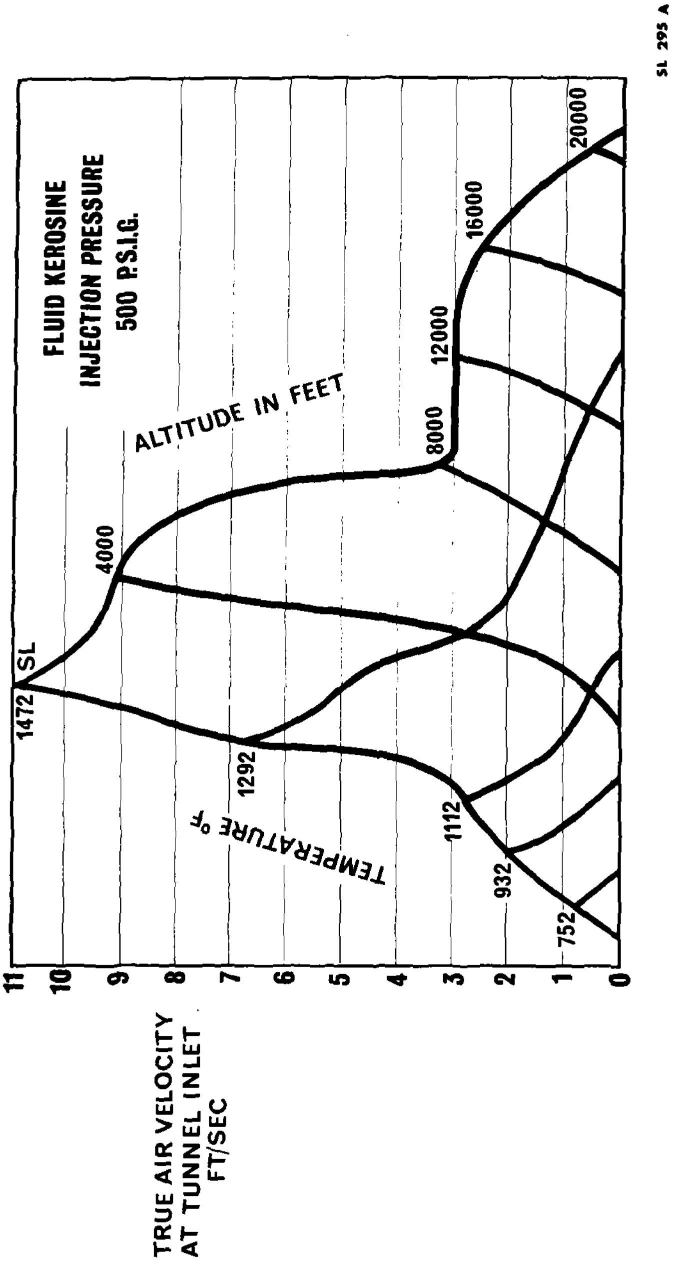

Figure 17. Wind tunnel rig - Ignition boundary at altitude.

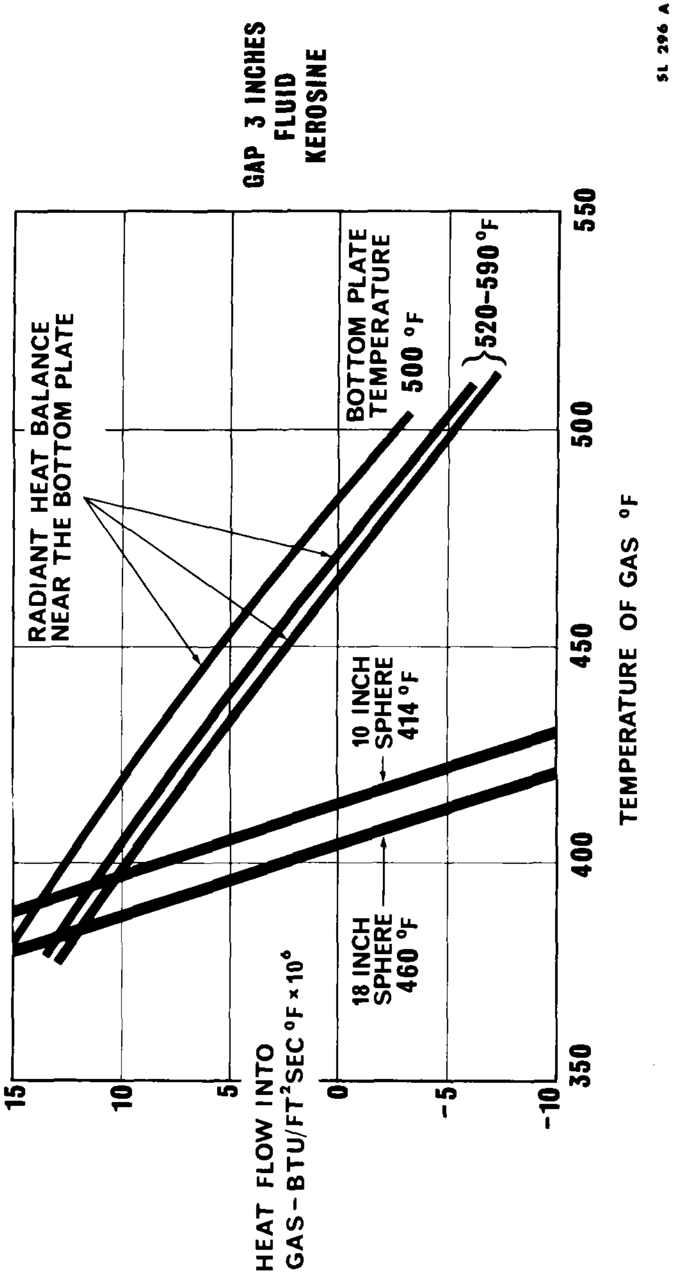

Figure 18. Static hot plate rig - Radiant heat balance.

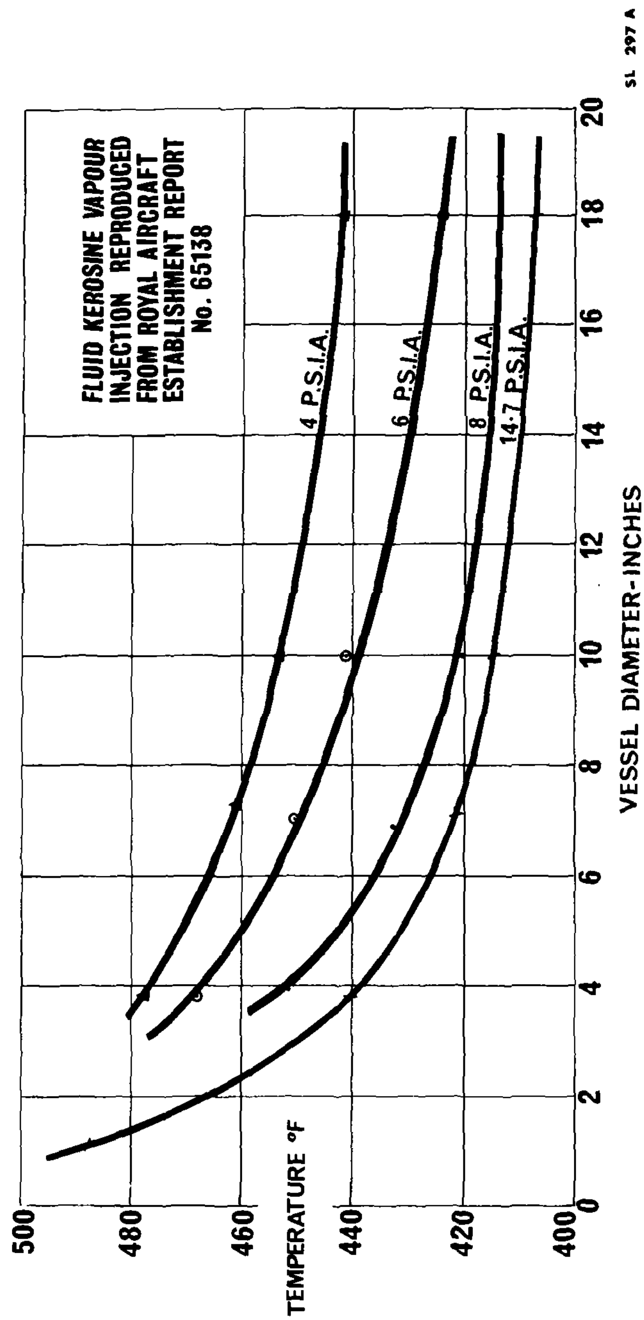

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Figure 19. Closed vessels - Minimum spontaneous ignition temperature.

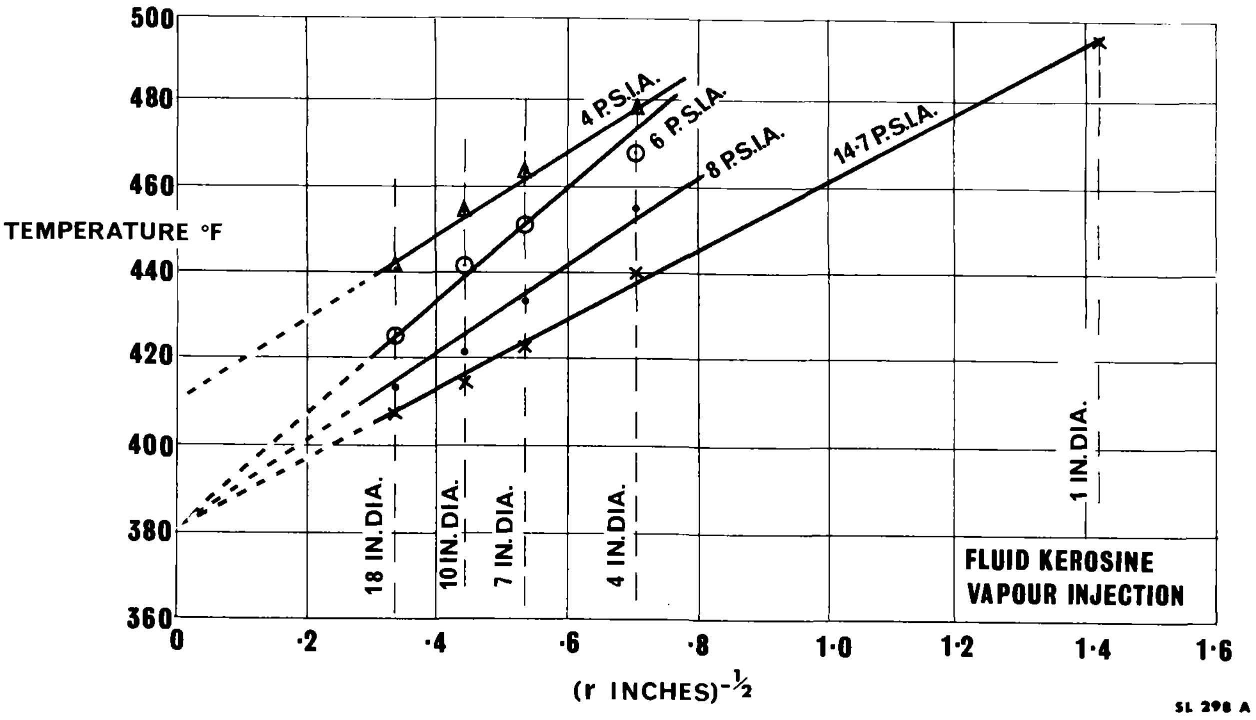

Figure 20. Closed vessels - Minimum spontaneous ignition temperature.

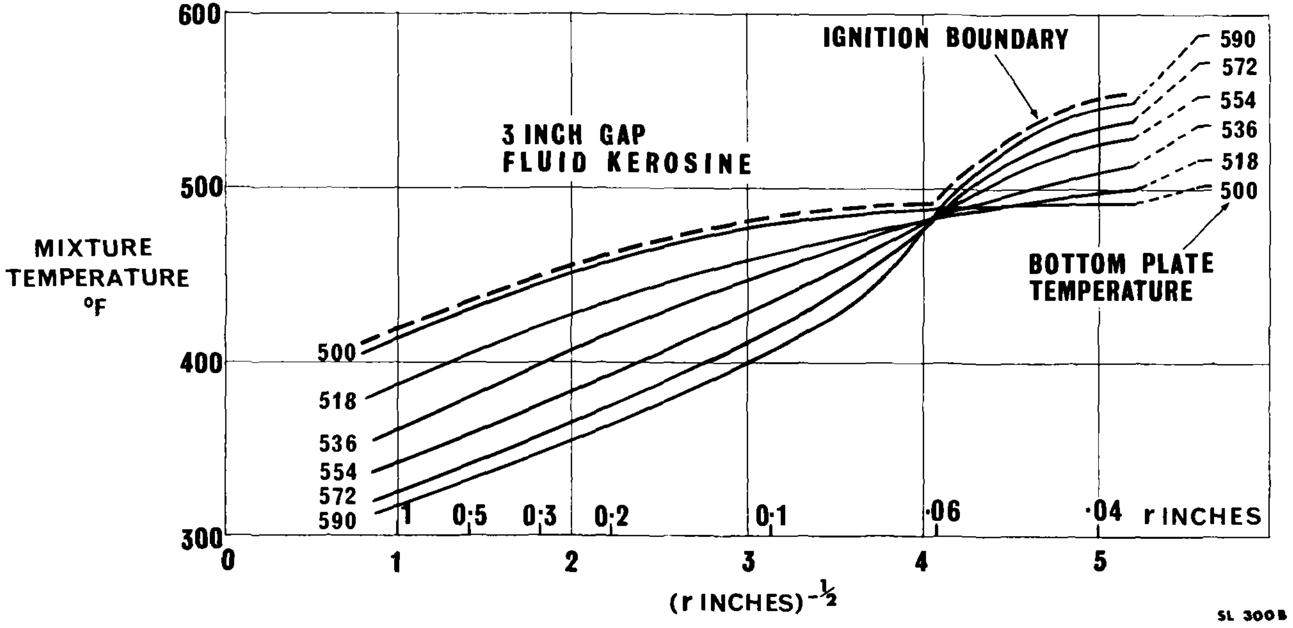

Figure 21. Static hot plate rig - Temperature profiles near the bottom plate.

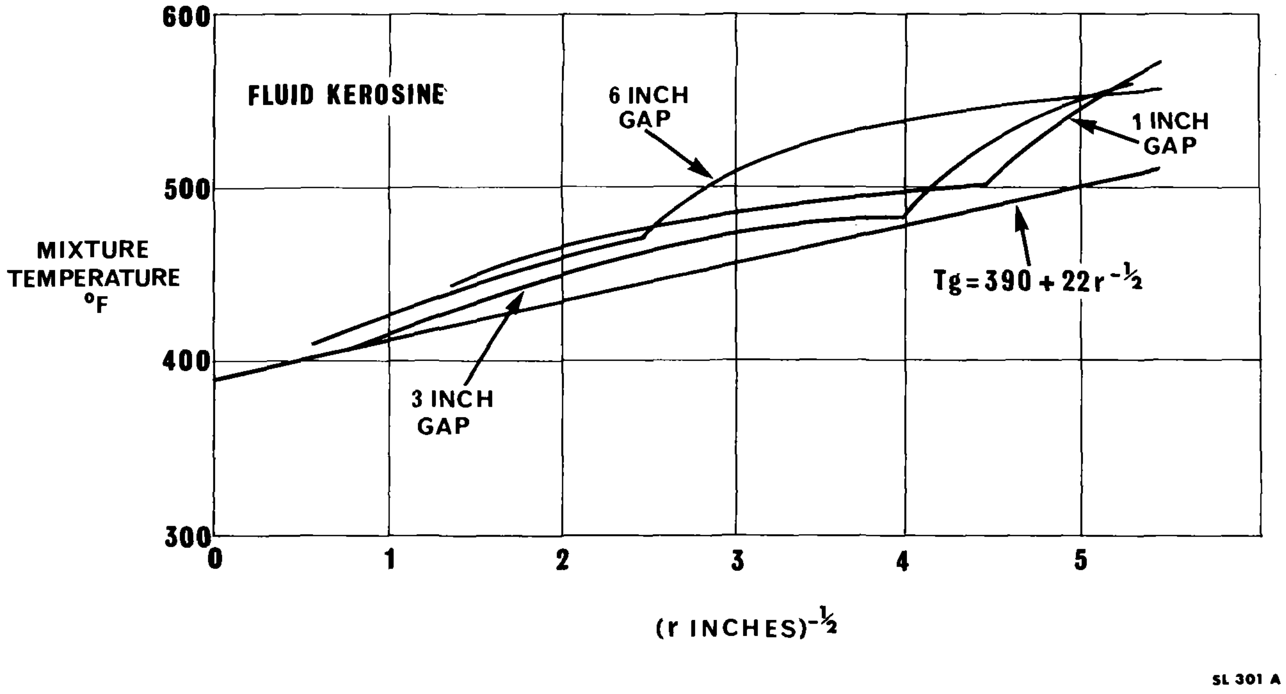

Figure 22. Static hot plate rig - Ignition temperatures and boundary.

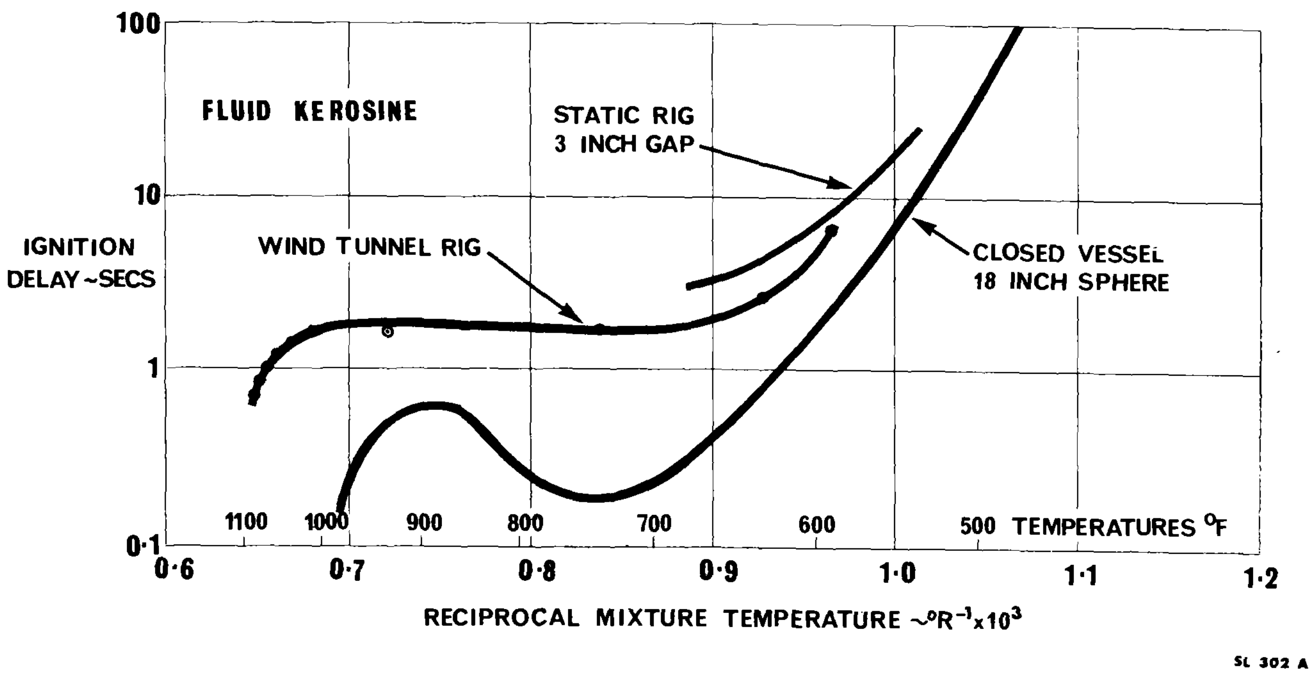

Figure 23. Ignition delay times and transit times .

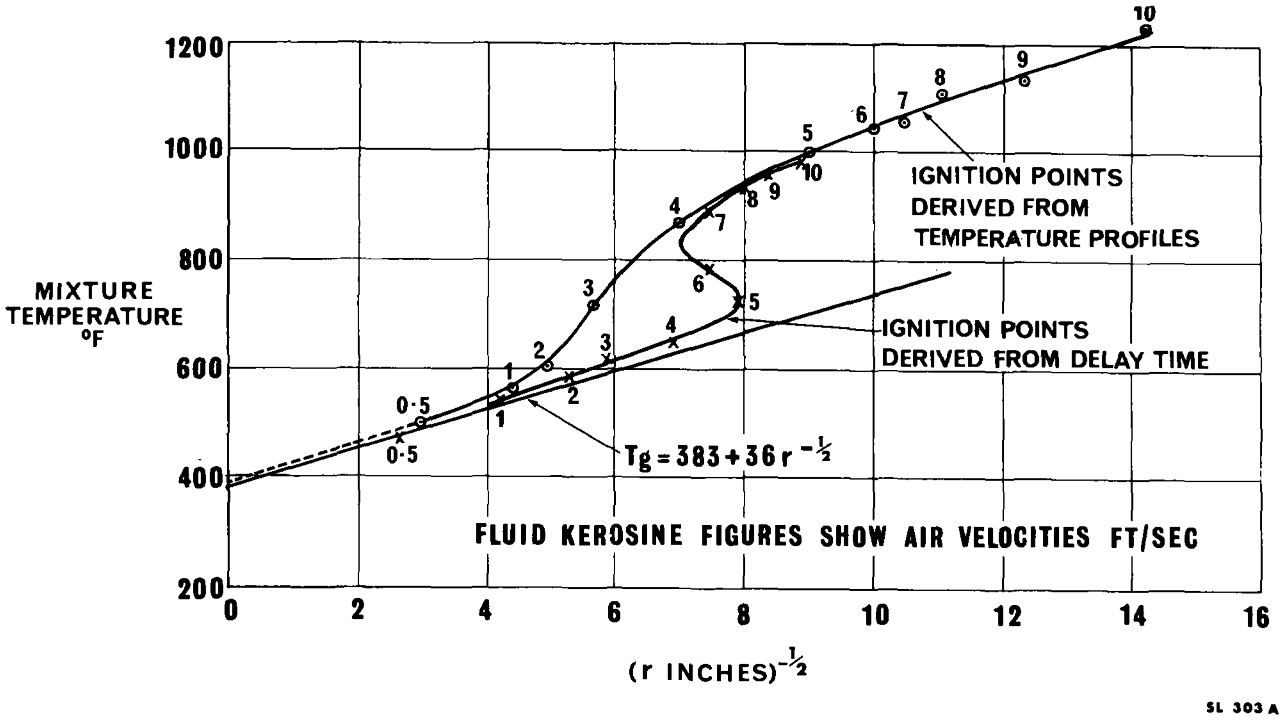

Figure 24. Wind tunnel rig - Ignition temperatures and boundary.

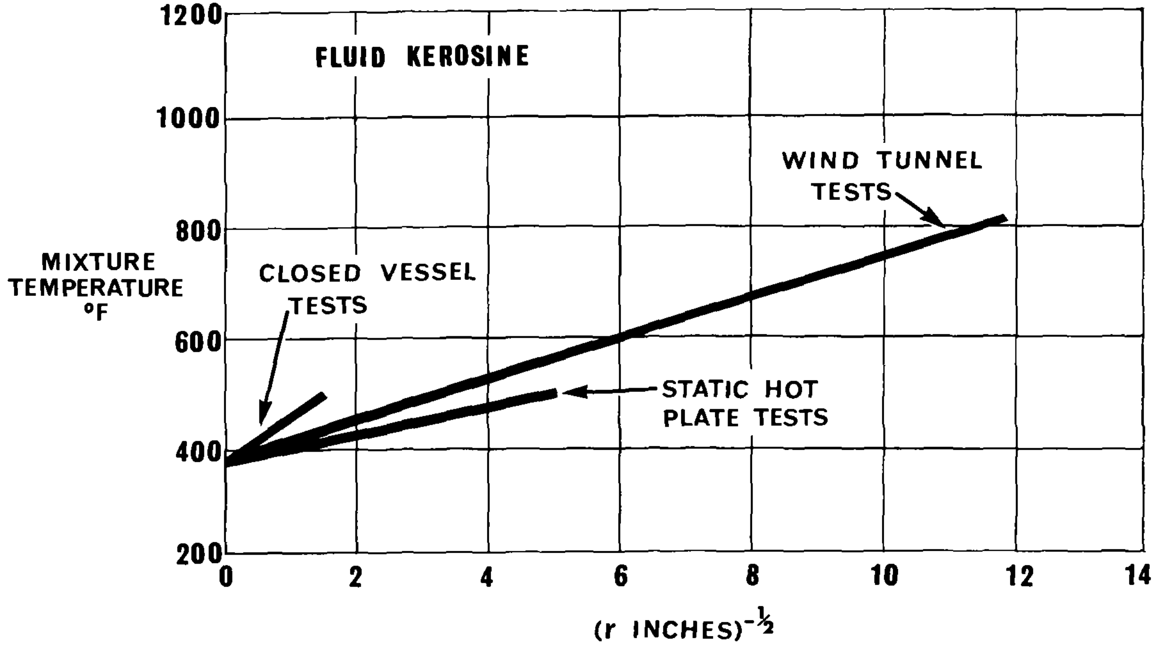

Figure 25. Boundaries of spontaneous ignition.

INDUSTRIAL AND ASTM FLUID FIRE-TEST PROGRAMS

By C. L. Early[1] and R. E. Hatton[2]

REFERENCE: C. L. Early and R. E. Hatton, "Industrial and ASTM Fluid Fire-Test Programs," Fire Resistance of Hydraulic Fluids, ASTM STP 406, Am. Soc. Testing Mats. 1966.

ABSTRACT: As the use of fire-resistant hydraulic fluids has increased over the past 15 to 20 years, so has the number and types of tests used for assessing fire resistance. Organizations which have studied this nebulous fluid property include ASTM, Factory Mutual, SAE, Underwriters' Laboratories, U.S. Bureau of Mines, U.S. Military groups, and several corporations. Tests developed by these groups fall into distinct classes; for example, pressure-spray, molten metal, hot manifold, wick type, etc., but many variations exist among specific procedures of a given test type.

A special group - Section VI of Technical Division N of ASTM Committee D-2 - was established 10 years ago to study fire resistance of fluids, but its progress has been slow. A major problem confronting industry today is the lack of test standardization and interpretation of test results to adequately measure fire resistance of fluids. Cooperative effort is recommended.

KEY WORDS: fire resistance, fire tests, flammability, hydraulic fluids

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[1] Project Manager, Development Department, Organic Division, Monsanto Company, St. Louis, Mo.

[2] Project Manager, Development Department, Organic Division, Monsanto Company, St. Louis, Mo.

Since World War II much time, work, and money have been devoted to the development of tests to measure the nebulous property of fire resistance of functional fluids. Organizations which have studied fire resistance include government and military groups, private testing organizations associated with insurance interests, technical societies, and individual companies. Communication and agreement among these testing groups generally has not been good, resulting in considerable duplication of effort. Such duplication is not necessarily undesirable since a greater number of test variables have consequently been evaluated.

This paper describes (1) the general types of industrial-fluid fire tests now in use or under development, (2) differences in techniques and conditions employed in performing specific types of tests, and (3) historically, the activities of ASTM regarding fire testing of fluids.

Review of Tests Employed by Various Organizations

The following organizations have listed test specifications or typical procedures for measuring fire resistance. In addition, some of the organizations are continuing test development work, either to improve existing tests or to provide entirely new methods.

American Society for Testing Materials
  (Committee D-2)
Factory Mutual Insurance Division
Society of Automotive Engineers (AMS 3150 C)
Underwriters' Laboratories, Inc.
U.S. Bureau of Mines
U.S. Bureau of Ships (MIL-H-19457A)
U.S. Military (MIL-F-7100)
Various corporations

With the exception of specifications and procedures developed by individual companies, which are sometimes treated as proprietary information, all other organizations have published their test methods. Some of the important characteristics of test methods reported by the above organizations (except corporations) are presented in Table 1.

It should be noted that frequently one type of test is employed by several organizations and that specific conditions for a given type of test may or may not vary from one organization's procedure to another. A suggested classification of test types is shown in Table 2.

Major differences in test procedures used by different testing groups for each classification (except Special) are shown in Table 3. When flash and fire points are specified, ASTM Methods D 92 and D 93 are used. The ASTM Autogenous Ignition Temperature (AIT) Method (D 2155) is commonly specified for industrial fluids, but other techniques are sometimes used by other groups, especially in the aerospace industry, to determine AIT. Experience has shown that minor changes in conditions can cause relatively large differences in AIT.

Conditions for performing the high-pressure spray ignition test vary rather broadly. The oxyacetylene torch (about 5000 F) is the most common ignition source used, but the Bureau of Mines and Underwriters' Laboratories employ several different ignition sources. Most groups test with a nitrogen pressure of 1000 psi on the fluid. However, Underwriters' Laboratories also test at lower pressures and the Bureau of Mines use 100 psi fluid pressure. Two groups perform this test at bulk fluid temperatures of ambient, two at 140 to 150 F, and Underwriters' Laboratories at temperatures ranging from ambient to over 200 F.

Orifice diameter and geometry vary, but a diameter of 0.0145 in. is most common. Experiences of the authors have shown that high-pressure spray test results can be substantially affected by changes in fluid temperature, droplet size, type of ignition source, and fluid properties such as viscosity, density, and surface tension.

In the high-pressure spray test, it is common to use as a criterion of fire resistance the propensity of a given fluid to propagate a flame downstream in the same direction as fluid flow. In practice, however, a major hazard is the tendency of flame to propagate "backward" from an ignition source to a line break. For example, a stream or spray of fluid from a broken hydraulic line on a piece of mobile equipment can be ignited by molten metal, hot steel, etc., propagating flame back to the mobile unit, and endangering the driver. An improvement over present high-pressure spray tests would be a means of measuring this "backward propagation" tendency. A stardardization of droplet size, now essentially ignored, would seem desirable.

Low-pressure spray ignition tests are not commonly used. This type of test is considered important, however, since high-pressure line breaks in service may become low-pressure breaks within seconds. Considerable progress has been made in developing a simple and convenient low-pressure spray test by one U.S. corporation. Low-pressure spray tests in which the fluid mist is produced by air aspiration may give results greatly different from those in which the mist is produced mechanically. This phenomenon may be due to differing air to mist ratios.

Hot metal test conditions vary greatly among different testing groups. Two groups employ an inclined cylindrical manifold heated to about 1300 F as the major test equipment. Factory Mutual employ a channel iron inclined 30 deg from horizontal and heated to 1300 F, whereas Underwriters' Laboratories normally use horizontal plates of three different metals heated to 1022 F for test purposes. Sometimes the quantity, rate, and method of fluid application and criterion of fire resistance are specified, sometimes not. As in the case of pressure spray tests, there is a real need for definition and standardization of hot metal type tests.

Surprisingly, molten metal tests have largely been overlooked by testing organizations except Underwriters' Laboratories and individual companies. Probably more fire-resistant hydraulic fluids are used in the die casting of aluminum and zinc than any other single application. Quite often, fire-resistant fluids will flash when in direct contact with molten metals in the 800 F (zinc) to 1200 F (aluminum) range. Test parameters which need defining and standardizing are quantity, temperature, and method of application of fluid, quantity and surface area of the molten metal, metal temperature, effect of external ignition sources, measurement of rate of burning (if any) on the molten surface, and assessment of tendencies to propagate flame to fluid-wet areas adjacent to the molten metal surface.

Flammability wick tests are used by most organizations. Generally, test conditions involve cycling a fluid-soaked pipe cleaner through a defined flame. The effect of evaporation of volatile (snuffer) components from fluids can be measured in this test by oven evaporation tests prior to fire testing. Careful control of test conditions; such as oven aging times and temperatures, cycling rate, location, control and type of flame, and amount of fluid on the pipe cleaner, is essential for reasonable test repeatability. The Underwriters' Laboratories asbestos strip test is entirely different from the cycling pipe cleaner types, but is related in that fluid-burning characteristics are assessed under thin-film, high-surface-area conditions.

Other tests which should be mentioned because of their practical importance are those employed and sometimes specified by individual companies. A number of typical industrial tests in this category are shown in Table 4.

As the test descriptions in Table 4 show, methods developed by individual corporations are sometimes rather specific and generally quite practical. The cardboard test is used by a company in the die-casting business because many large cardboard boxes for receiving and shipping goods are situated throughout its plant. A hydraulic-line break from a die-casting machine could soak cardboard boxes with fluid. The company desires that only those fluids be used in die-casting machines which will not allow "continued burning" of fluid-wet boxes in the event of fire or localized burning on the molten metal surface.

The mannequin-hot-plate test simulates a potential fire with a man standing near an ignition source. To some degree it is a combination high-pressure spray, hot metal, and flammability wick test. Much work could be done on this test to determine the effects of the many variables involved.

The in-plant molten metal test simulates a hydraulic-line break in which fluid is sprayed on a molten metal surface and adjacent cool surfaces. Flashing or burning of fluid on a molten metal surface is not considered a serious hazard, but propagation of flame away from the ignition source represents a very serious hazard.

Despite their specificity, important aspects of these practical user tests could well be incorporated into tests developed by ASTM and other independent groups. It is extremely important that fire test methods simulate or take cognizance of all conditions that may exist in actual fluid use, both within and outside the operating system.

ASTM D-2 Technical Division N Programs

Because of steadily increasing interest in hydraulic fluids and the introduction of numerous synthetic types, ASTM organized in 1955 Technical Committee N on Hydraulic Fluids under ASTM Committee D-2 on Petroleum Products and Lubricants. The basic responsibilities of Technical Committee N are to increase and disseminate knowledge and to standardize the specifications, test methods, and nomenclature in the area of hydraulic fluids. As a basis of the committee organization, it was agreed that the establishment of standard test procedures applicable to all types of hydraulic fluids and the definition of performance requirements in all end uses were the important areas to be considered. Consequently, a number of sections were established and Section VI was assigned the area of fire resistance of hydraulic fluids.

The organizational meeting of Section VI was held February 7, 1956. At this meeting a section scope was adopted as follows: "The study of fire resistant and flammability characteristics of hydraulic fluids and the development of test methods for their measurement." At this first meeting, it was agreed that a questionnaire would be circulated seeking information on all flammability test methods used by the various members of the committee. Comments were also requested concerning the significance of each test. At this meeting, it was also suggested that a panel be established to assemble data on the causes of actual hydraulic fluid fires. Such data would then be used to guide test method development.

Analysis of the returned questionnaires indicated that too large a number of tests were in use to allow any selection for round-robin testing. It was therefore agreed that the best approach to the problem would be to evaluate a series of standard reference fluids in each of the tests proposed by the various members. The results would then be collected and analyzed and some selection made of specific tests for further study. Six types of hydraulic fluids were included in this program: water-base (glycol), phosphate ester, petroleum oil, halogenated-base, phosphate ester-base, and oil-water emulsion. Standard samples of these reference fluids were sent to all those interested in cooperating with the program. A Study Group on Origin of Fires was also established.

By February, 1959, data had been submitted by 19 laboratories using the six reference fluids in 18 different procedures. These data were reviewed by an appointed Analysis Panel which recommended that two sub-sections be activated to carry on further work. One sub-section was to be concerned with spray-type tests. The objective of this group was to review the data accumulated on spray tests, to correlate such data with information from the origin of fires study and to devise one or more composite methods which would have the greatest possible significance in terms of field service. It was expected that round-robin testing would be required to arrive at workable procedures.

The other sub-section was to work on six relatively simple, non-spray type screening tests which in the opinion of the Analysis Panel showed considerable promise. These methods were selected to provide a range of ignition sources such as open flame, hot metal, hot glass, molten metal, radiation, electric arc, and bulk fluid, absorbed fluid, or sprayed fluid. The six methods suggested for further study were the hot manifold test, the molten metal pour test, the pipe cleaner-radiant test, the Tonawanda spoon test, the modified flame propagation test, and the pipe cleaner-burner test. Some of these tests had been run by only one laboratory, and therefore it was necessary that additional work be done to establish their relative usefulness. Thus, the six methods were compiled and submitted to the potential round-robin cooperators.

Subsection I on Origin of Fires collected and analyzed data on fires which involved hydraulic fluids. Data obtained on 308 different fires, (88 aircraft, 161 industrial, and 59 mining) were studied from the standpoint of ignition source. The following conclusions were offered by this study group:

1. "Ignition by hot surfaces, the most important ignition source, caused 46% of all fires. In aircraft applications, there is a significant trend toward increased relative importance of hot surface ignition."
2. "Chemical spark ignition accounted for 25% of all fires. This ignition source is of minor importance in all but mining applications, where it is of over-riding importance, accounting for 95% of all mine fires."
3. "Open flame and frictional ignition source are of minor importance, together accounting for only 13% of the fires."
4. "Molten metal ignition is of significant importance in industrial applications, causing 32% of industrial fires."

During the next few years very little success was obtained in securing any real progress on round-robin testing in the development of standard procedures. This was attributed to the press of other activities and considerable difficulties in developing consensus of opinion on test conditions.

In an effort to increase interest in the Section VI activities, an informal symposium was held in January, 1963. Six presentations, accompanied by slides and movies, demonstrated fluid flammability testing. This symposium pointed out the extreme difficulty of testing fluid fire resistance, the wide range of parameters involved, the range of performance of various types of fluids in different tests, and the requirement for more than one procedure to adequately describe fire resistance.

Early in 1964 a survey was sent to all members of Section VI and other groups who had expressed some interest in fire-resistance tests. The purpose of this questionnaire was to determine the extent of interest in or need for the development of bench-test methods for examining the fire resistance of hydraulic fluids. By the end of 1964, 32 out of 102 questionnaires had been returned. These answers were used to reorient the Section activities. Four task groups were organized under Subsection II Bench Test Method Development, each to work on a specific method aimed toward developing a standard procedure. These four task groups are 1) Hot Manifold Test, 2) Molten Metal Test, 3) Pipe Cleaner-Burner Test, 4) Modified Flame Propagation Test. Subsection III on Development of Spray Test Methods was continued and is actively working on one test procedure aimed toward round-robin testing. It has been recognized that any one spray-test method will probably not be acceptable to all groups.

This review of the activities of Section VI on Fire Resistance of Hydraulic Fluids indicates very little progress in developing standard test methods over a 10-year period. Much of the delay can be attributed to the difficulties in finding test methods suitable for wide ranges of chemical materials which burn in different ways and which show variable performance in different tests. Section VI has served a useful function in calling attention to the problems involved in fire testing, in spreading information and knowledge about fire resistance, and in defining fire hazards in industrial, mining, and aircraft operations. Future Section VI activities should perhaps be aimed more at test standardization than test development in view of the multiplicity of existing simulative tests described above.

No discussions of ASTM activities on fire resistance of fluids would be complete without mentioning previous work on flash and fire point test methods. These methods are well accepted and are useful in assessing flammability under certain conditions. Care should be exercised in interpreting the results in relation to actual fluid-use conditions.

Research Division XIII of ASTM Committee D-2 has recently studied autogenous ignition temperature tests and developed a new procedure, now adopted as Tentative Method D 2155. This method shows good reproducibility on hydrocarbon products. Research Division XIII has asked Section VI to determine the applicability of this test method to other types of chemicals.

Conclusions

Field experience has demonstrated that fire-resistant fluids make a real contribution to increased safety of operations. Although the term "fire resistance" is difficult to define, considerable progress has been made on the development of meaningful tests by fluid consumers and producers, government agencies, and independent testing organizations. It has been recognized that use conditions determine the degree and type of fire resistance required in any specific application and that several bench tests may be required to assess adequately the fire resistance of any given fluid. A number of test procedures have been developed which are applicable to specific fire hazards. Effort is needed to define further fire hazards as influenced by use conditions, to devise more simulative test techniques, and particularly to standardize specific test methods. The hazard of fluid fires is real. The reduction of this hazard by the use of less flammable fluids should be of primary interest to the entire industry.

Table 1--Current Fire Tests .

Test Identification	Approximate Test Conditions	Criteria of Performance
I. <u>ASTM Committee D-2 Fire Tests</u>		
A. Established Tests		
1. Flash Point, Cleveland Open Cup (COC), Method D 92	Test fluid is heated in open container at a controlled rate and a small flame is passed over the surface periodically.	Temperature at which flashing is first observed.
2. Flash Point, Pensky-Martin Closed Cup, Method D 93	Similar to COC method, but closed container is used.	Same as with COC method.
3. Fire Point, Method D 92	COC Method D 92 test is continued beyond the flash point until burning is continuous or until fluid boils.	Temperature at which burning becomes continuous.

(Continued on next page)

Test Identification	Approximate Test Conditions	Criteria of Performance
I. <u>ASTM Committee D-2 Fire Tests</u>		
A. Established Tests (Continued)		
4. Autogenous Ignition Test, Method D 2155	Various quantities of test fluid are charged to Erlenmeyer flask while progressively increasing or decreasing temperature in 5 F increments.	Minimum temperature at which spontaneous ignition occurs.
B. Tests Under Study (Tech Committee N)		
1. High-Pressure Spray Flammability Tests	To be determined	To be determined
2. Hot Manifold Test	To be determined	To be determined
3. Molten Metal Pour Test	To be determined	To be determined
4. Pipe Cleaner - Burner Test	To be determined	To be determined

(Continued on next page)

Test Identification	Approximate Test Conditions	Criteria of Performance
I. ASTM Committee D-2 Fire Tests		
B. Tests Under Study (Tech Committee N) (Continued)		
5. Modified Flame Propagation Test	To be determined	To be determined
II. Factory Mutual Insurance Division Fire Tests		
A. High-Pressure Spray Ignition Test	1000 psi, 140 F, Oxy-acetylene torch ignition.	Flashing at 6 in. from nozzle must be self-extinguishing in 5 sec.
B. Hot Channel-Iron Test	1300 F iron surface at 30 deg incline sprayed from 6 in. distance, 1000 psi, 140 F.	No flashing or ignition.

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Test Identification	Approximate Test Conditions	Criteria of Performance
III. SAE Aeronautical Military Specifications, AMS 3150 C	(In all tests performance must be equivalent to or better than HS-1 Reference Fluid.)	
A. Flammability Spray Test (Low Pressure)	Test sample sprayed at 40 psi, room temperature, into oil-soaked burning cotton.	Observe for flame increase, no change, or decrease in intensity.
B. Flammability Wick Test (Pipe Cleaner Test)	Cycle fluid-soaked pipe cleaner at 30 to 40 cycles/min through 4-in. Bunsen flame.	Count number of cycles without self-sustaining flame.
C. Flammability, High-Temperature Ignition (High Pressure) Test	Spray fluid at 1000 psi, room temperature. Apply oxy-acetylene torch at increasing distances from nozzle.	Observe distance at which flashing starts and for propagation of flame.
D. Flammability, Manifold Test	Pour 10 ml of test sample in 40 sec on exterior of 1300 F steel tube.	Observe for flashing or burning on the tube and for fluid burning in "catch pan" below.

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Test Identification	Approximate Test Conditions	Criteria of Performance
III. SAE Aeronautical Military Specifications, AMS 3150 C (Continued)		
E. Autogenous Ignition Temperature Test	ASTM Method D 2155 test used.	Ignition temperature >750 F required.
IV. Underwriters' Laboratories, Inc. (Typical Tests)		
A. Volatility		
1. Vapor Pressure	Various laboratory methods.	Low vapor pressure desired.
2. Flash Point, Closed Cup	ASTM Method D 93.	High flash temperature desired.
3. Fire Point, COC	ASTM Method D 92.	High fire temperature desired.
B. Autogenous Ignition Temperature	ASTM Method D 2155; Heated Erlenmeyer flask with various amounts of sample.	Minimum temperature for ignition without spark.

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Test Identification	Approximate Test Conditions	Criteria of Performance
IV. Underwriters' Laboratories, Inc. (Typical Tests) (Continued)		
C. Explosiveness of Vapors	Heat steel cylinder with central electrodes to progressively higher temperature. Charge sample to hot cylinder and spark periodically.	Minimum temperature of ignition, flame propagation and pressure effects.
D. Explosive Range Tests (Upper and Lower Limits)	Electric spark over sample at 752 F in glass explosion chamber.	Maximum and minimum %v of sample vapor in air which is explosive.
E. Spontaneous Heating Test	Sample maintained at 212 F on cotton gauze in jacketed steel chamber.	Spontaneous temperature rise.
F. Heat of Combustion	Calorimetric method.	Amount of heat generated per unit weight of sample.

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Test Identification	Approximate Test Conditions	Criteria of Performance
IV. <u>Underwriters' Laboratories, Inc. (Typical Tests)</u> (Continued)		
G. Combustion Intensity		
1. When liquid is vaporized in definite proportions with air	Special	Rate of burning
2. When liquid is ignited in open vessel	Special	Rate of burning
H. Decomposition Temperature Tests		
1. AIT Erlenmeyer Test	AIT Equipment; temperature progressively raised and sample charged.	Minimum temperature to effect litmus color change of vapor.

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Test Identification	Approximate Test Conditions	Criteria of Performance
IV. Underwriters' Laboratories, Inc. (Typical Tests)		
H. Decomposition Temperature Tests (Continued)		
2. Hot Metal Surface	Use "Explosiveness of Vapors" equipment, heated to 1112 F; sample charge at uniform rate and gas samples of decomposition products taken.	Analyze gas samples for hydrogen chloride, free chlorine, carbon monoxide, carbon dioxide, oxygen and other gases.
3. Electric Arc	15-lb sample charged to steel vessel at room temperature. Electrodes at 230 v dc were both partially and totally immersed in fluid and operated at 170 amp for 5 min.	Analyze for same gases as above.

(continued on next page)

Test Identification	Approximate Test Conditions	Criteria of Performance
IV. <u>Underwriters' Laboratories, Inc. (Typical Tests)</u> (Continued)		
I. Pressure-Spray Ignition Test	Sample sprayed as mist at 200 to 1000 psi; ignited with oxyacetylene torch and electric spark.	Flashing and flame propagation.
J. Soaked Asbestos Flammability Test	Soaked asbestos strip ignited with Bunsen burner flame.	Height of flame produced.
V. <u>U. S. Bureau of Mines Fire Tests</u>		
A. Autogenous Ignition Test	Similar to ASTM Method D 2155.	Ignition temperature > 600 F required.
B. Temperature/Pressure Spray Ignition Test	Atomized sample sprayed at 100 psi, 150 F and ignited with (1) burning, kerosene-soaked rags, (2) 12,000-v electric arc, and (3) a propane torch.	Flame propagation of < 6 sec at a distance of 18 in. from orifice.

(Continued on next page)

Test Identification	Approximate Test Conditions	Criteria of Performance
V. U. S. Bureau of Mines <u>Fire Tests (Continued)</u>		
C. Effect of Evaporation on Flammability (Pipe Cleaner Test)		
1. Fresh sample evaluation	Pipe cleaner soaked in fresh sample, drained and cycled at prescribed rate through Bunsen burner flame.	Minimum of 24 passes without a self-sustaining flame.
2. Two-hour heated sample evaluation	Same as above except test fluid aged in Petri dish two hours at 150 F.	Minimum of 18 passes without a self-sustaining flame.
3. Four-hour heated sample evaluation	Same as above except test fluid aged in Petri dish four hours at 150 F.	Minimum of 12 passes without a self-sustaining flame.

(Continued on next page)

Test Identification	Approximate Test Conditions	Criteria of Performance
VI. U. S. Military Specification MIL-H-19457A (Ships)	A standard CFR cetane rating engine is operated at progressively decreasing compression ratios while injecting 30 ml per minute of test fluid into the fuel air mixture. Test conditions of engine speed, temperature, spark advance, etc., are specified in "ASTM Manual of Engine Test Methods for Rating Fuels."	Combustion of test fluid is evidenced by an increase in pressure on the compression stroke. The minimum compression ratio at which combustion occurs is recorded; ratios of 40:1 and below are failing.
VII. U. S. Military Specification MIL-F-7100		
A. High-Temperature/ Pressure Ignition Spray	Test fluid at 60 and 100 F, 1000 psi, sprayed and ignited with oxyacetylene torch at increasing distances from orifice.	When ignited at 18 in. from orifice, there can be no flame propagation downstream.

(Continued on next page)

Test Identification	Approximate Test Conditions	Criteria of Performance
VII. U. S. Military Specification MIL-F-7100 (Continued)		
B. Hot Manifold Test	Drip 10 ml of test fluid in 40 to 60 sec on 1300 F manifold.	The fluid shall not burn on the hot manifold or upon leaving the manifold.
C. Incendiary-Gun Fire Test	Test fluid is pressurized at 1000 psi in a vertically mounted aluminum tube. A 0.30-calibre incendiary bullet is fired through the tube.	The fluid shall not burn.
D. Effect of Evaporation (Pipe Cleaner Test)	Test fluid in a Petri dish is stored for 24 hours at 120 F. A pipe cleaner saturated (a) with fresh test fluid, and (b) with evaporated fluid is cycled 25 times per minute through a laboratory burner.	Number of cycles completed with evaporated fluid must at least be 80% of the number completed with fresh fluid.

Table 2--Classification of Current Fire Tests.

Flash and Fire Points
Autogenous Ignition Temperature
High-Pressure Spray Ignition
Low-Pressure Spray Ignition
Hot Metal
Molten Metal
Flammability Wick
Special[a]

[a] Generally, tests in this classification are those employed by only one organization; for example, incendiary gun fire test, explosive range tests, etc.

Table 3--Comparison of Typical Procedures.

	ASTM	Factory Mutual	SAE AMS-3150 C	Underwriters' Laboratories	Bureau of Mines	Military MIL-F-7100
Flash and Fire Points	D 92 and D 93	Not Required	Not Required	ASTM Method Used	Not Required	Not Required
Autogenous Ignition Temperature	D 2155 Various quantities (drops) of test fluid added to 200 ml Erlenmeyer flask while progressively increasing or decreasing flask temperature by 5 F increments. Minimum temperature for spontaneous ignition is determined.	Not Required	ASTM Method Used	ASTM Method Used	ASTM Method Used	Not Required

(Continued on next page)

	ASTM	Factory Mutual	SAE AMS-3150 C	Underwriters' Laboratories	Bureau of Mines	Military MIL-F-7100
High-Pressure Spray Ignition Test	Test Under Study	Atomized spray;1000 psi; 140 F fluid; 1.5 gal/hr flow; 80 deg angle nozzle;oxyacetylene torch ignition applied at various distances from orifice.	Spray of fluid; 1000 psi;0.0145 in.dia.orifice; oxyacetylene torch ignition applied at 1.5 to 12 in. from orifice. No fluid temperature specified (presumed room temperature)	Streams and sprays of fluid; 200 to 1000 psi; 0.0145 in. dia. orifice for streams; fluid temperature from ambient to 200 F; three ignition sources; gas-air blast burner, oxyacetylene torch, electric arc applied at various distances from orifice.	Spray of fluid; 100 psi; 0.025 in.dia. orifice; 150 F fluid temperature; three ignition sources, burning kerosene-soaked cotton waste,electric arc, and propane torch applied for one minute at various distances from orifice.	Fluid spray; 1000 psi; 0.0145 in. dia.orifice; 60 to 100F fluid temp.; oxyacetylene torch ignition at 1 to 18 in. from orifice.

(Continued on next page)

	ASTM	Factory Mutual	SAE AMS-3150 C	Underwriters' Laboratories	Bureau of Mines	Military MIL-F-7100
Low-Pressure Spray Ignition Test	Test Will be Studied	Not Required	Dense, cone-shaped spray from 40 psi paint spray gun(0.070 in. dia.orifice) is directed on burning oil-soaked cotton waste.	Not Required	Not Required	Not Required
Hot Metal Test	Test Under Study	Channel iron mounted at 30 deg from horizontal is heated with gas burner to 1300 F. Fluid at 1.5 gal/hr directed at bottom of incline.	Simulated manifold, 3 in. dia. heated electrically to 1300 F. 10 ml fluid at ambient temp. poured on manifold in about 40 sec. Heaters thermostatically controlled.	Horizontal metal plates (steel, copper, aluminum) heated with gas burners to 1022 F. Various amounts of fluid applied. Burners shut off before fluid applied.	Not Required	SAE AMS-3150 C Method Used

(Continued on next page)

	ASTM	Factory Mutual	SAE AMS-3150 C	Underwriters' Laboratories	Bureau of Mines	Military MIL-F-7100
Hot Metal Test (Continued)		Burners shut off before starting spray.				
Molten Metal Test	Test Under Study	Not Required	Not Required	Various amounts of fluid are applied to molten zinc at 1022 F.	Not Required	Not Required
Flammability Wick Test	Test Under Study	Not Required	Soak pipe cleaner in fluid at room temperature and drain.Adjust Bunsen burner for non-luminous 4-in.flame. Cycle fluid-soaked pipe cleaner through hottest part of flame at 30 to	Asbestos strip soaked with test fluid is ignited by a (defined) Bunsen burner flame. Test fluids are compared with petroleum oils with	Soak pipe cleaners in (a) fresh fluid, (b) fluid aged 2 hr at 150 F, and (c) fluid aged 4 hr at 150 F and drain. Cycle at	Soak pipe cleaners in (a) fresh fluid, and (b) fluid aged 24 hr at 120 F and drain. Cycle fluid-soaked pipe cleaners through laboratory burner

(Continued on next page)

	ASTM	Factory Mutual	SAE AMS-3150 C	Underwriters' Laboratories	Bureau of Mines	Military MIL-F-7100
Flammability Wick Test (Continued)			40 cycles/minute.	respect to burning in flame area, max. height of flame, burning tendency after removal of flame, and burning rate.	25 cycles/minute through non-luminous, 4-in. Bunsen burner flame.	flame. Flame and cycle rate not defined.

Table 4--Special Corporate Fire Tests.

Cardboard Test

One end of a corrugated cardboard strip is soaked with test fluid, drained, and the dry end ignited. Test criterion is whether there is an increase, decrease, or no change in flame intensity when the flame front reaches the wet section. The flame is extinguished by some commercial fire-resistant fluids.

Mannequin Hot-Plate Test

A clothed mannequin is placed near a vertical hot steel plate. A stream or spray of test fluid is impinged on the plate. The minimum distance the mannequin can be placed from the hot plate without catching fire in a short time interval can be taken as the criterion of fire resistance. Variables which should be controlled are many; for example, temperature, pressure, droplet size and duration of fluid stream, hot plate temperature, orientation of mannequin and hot plate, and type of cloth on mannequin.

Special Pressure Spray Test

Similar to the mannequin hot-plate test, but the mannequin is omitted.

In-Plant Molten Metal Test

This is an in-plant test whereby materials and equipment adjacent to a commercial vat of molten metal are wet with test fluid. A continuouous film of fluid is formed from these wet areas leading into the vat of metal. Criteria of the test are the burning characteristic of the test fluid on the molten metal surface and flame propagation tendencies adjacent to (cool) fluid-wet areas.

Table 4--Special Corporate Fire Tests.
(Continued)

Meker Burner Test

Similar to the Bureau of Mines' pipe cleaner test, except a larger and hotter (Meker) burner is used.

STP406-EB/Nov. 1966

MINIATURIZED TESTS FOR FIRE RESISTANCE OF HYDRAULIC FLUIDS

By D. E. Johnson[1] and N. W. Furby[2]

ABSTRACT: Some of the conventional tests for assessing the fire resistance of hydraulic fluids use relatively large amounts of fluid in relationship to quantities available from exploratory research programs. To alleviate this problem, four miniaturized tests were devised for evaluating fire resistance of aerospace fluids. These were intended to simulate the following normal-size tests: (1) flash point (Cleveland open cup), (2) hot manifold, (3) low pressure spray, and (4) high pressure spray. The microflash apparatus uses a spark plug and a heated aluminum cylinder. Three drops of fluid are applied at each temperature of the test. Results were somewhat higher than for full-scale equipment. Low pressure spray test employs an airless spray gun (rotating disk) with reservoir modified to hold a small volume of fluid. In the high pressure spray apparatus, gas pressure forces fluid from a small orifice in stainless steel tubing, and only 20 ml of fluid is used in each determination. The small-scale hot manifold test uses a 1/2-in. diameter stainless steel heater. These miniaturized tests require a total of 40-ml of fluid, as compared to about 2500 required for the corresponding full-scale tests. The small units are easy to operate, and repeatability is good.

KEY WORDS: fire resistance, flammability, hydraulic fluids, flash point, hot manifold test, spray flammability test, fire tests

[1] Mechanical Engineering Dept., University of Idaho, Moscow, Idaho.
[2] Chevron Research Co., P.O. Box 1276, Richmond, California 94802.

Only very small quantities of newly synthesized compounds are available in early stages of exploratory research on hydraulic fluids. We have devised miniaturized versions of four existing flammability tests to facilitate early judgment of the fire resistance of these new materials. This paper describes these tests but does not discuss the value of such tests as related to the end use of fluids.

The four miniaturized tests are (1) flash point (Cleveland open cup), (2) hot manifold, (3) low pressure spray flammability, and (4) high pressure spray flammability. Some of these can be related to the test methods of Specification AMS-3150C [1].[3] As little as 40 ml of fluid is required to obtain a flammability rating by the four micromethods. (The full-scale tests would require about 2500 ml.) Each test method is detailed as follows:

Microflash Test

The microflash unit was developed to provide flash point data on small experimental samples for which there was insufficient quantity to run Cleveland Open Cup (COC) flash point [2]. The microflash unit consists of a heated aluminum block, which has a small cup and a small spark plug. Fig. 1 is a cross-section drawing of the apparatus. Fig. 2 is a photograph of the apparatus and shows the variable transformer control, the spark plug, the control button, and the insulated, heated aluminum block.

Aluminum foil disks are placed in the pan for each test in order to avoid effects of residual decomposition products. A six-volt, dry cell battery furnishes the electric energy; and this is transformed to high voltage by an automotive spark coil. The spark plug electrode is located 3/16 in. above the pan surface. Tests can be run in two ways: (1) For initial screening purposes, the block temperature is held at 450, 500, and 550 F; and the lowest temperature at which flash occurs is noted. (2) A more definitive procedure consists of increasing the block temperature in 50 F increments until flash occurs, then lowering the temperature in 5 F increments until flash ceases to occur. In this routine, the lowest temperature at which flash occurs is reported. Fig. 3 shows results as compared to the full-scale COC flash points. The

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[3] The numbers in brackets refer to the list of references at the end of this paper.

sample is introduced in all cases by a 5-ml syringe and 26-gage needle. Three drops are placed in the pan before each determination, and spark is applied 30 sec later. As suggested above, some experimental fluids require a new aluminum pan before each sample introduction because the decomposition residue produced alters the flash point. Fig. 3 shows that the microflash unit produces flash temperatures approximately 20 F higher than COC. This is believed due to a slightly different vapor-air ratio at the ignition point.

Small-Scale, Hot Manifold Test

Hot manifold ignition sources have become less important in the jet age. However, hot surface ignition at brakes is a similar hazard. The standard hot manifold test equipment [1] is rather large and uses considerable hood space. A smaller version and one requiring less sample was desired. The small-scale apparatus is shown in Fig. 4. The 250-w heater, variable transformer, and 5-ml syringe are visible. Details of equipment used appear in Fig. 5. The thermocouple is Chromel-Alumel bonded to the inner side of the stainless steel heater. The standard hot manifold test is run at 1300 F. However, because the tube diameter of the standard size is larger, producing longer fluid residence time, it was necessary to raise the temperature of the small-scale device to 1450 F. This temperature was selected because a reference fluid burned at this temperature similarly to the result on the full-scale manifold at 1300 F. Fluids are given a numerical rating determined by the extent of burning and whether or not the fluid continues to burn as the drops fall below the hot tube. This test requires 5 ml of fluid.

Low Pressure Spray Flammability Test

The low pressure spray flammability test was intended to provide small-scale results that would correlate with the spray flammability test of ASM-3150C [1]. The low-pressure spray test was based on suggestions of L. S. Sargent, Jr., and D. H. Nail and in connection with our ASTM Technical Committee N work except we devised a modified reservoir to lower the capacity. Fig. 6 shows the finished apparatus, which is an Electr-O-Airless paint gun [3] (rotating disk) mounted on a stand. The dimensions locating port opening with relationship to the Bunsen burner are shown in Fig. 7. The gate opening on the gun is fixed at 1/4 in. by a mechanical stop. The cone form (Fig. 8) was constructed to make aluminum foil cups to hold a small sample size for screening tests. The cups are formed by pressing foil by hand about the form. The formed cups are placed in the original reservoir.

Test conditions employ the "fast" setting on the paint gun, a natural gas flame which is lazy and mostly yellow, and preheated fluid samples (140 to 150 F). The test appears to be extremely critical as to fluid viscosity. Low viscosity fluids are sprayed more readily by the paint gun, and this affects the flammability result. Tests are currently being conducted on this apparatus to relate flame size to viscosity. Flames produced are photographed on black and white film and are rated small, medium, and large. Approximately 40 ml of fluid is required to run this test, of which about 10 ml is consumed; the remaining 30 ml is recoverable for other tests. Usually, this test is run first on small experimental samples and followed by the other tests.

High Pressure Spray Flammability Test

A small-scale, high-pressure spray flammability test was developed to permit testing of 20 ml of fluid as compared with about 2500 ml required for the normal apparatus. (High-pressure spray tests are intended to simulate spray from leaking pressurized hydraulic lines.) Fig. 9 shows the apparatus installed out-of-doors. Outdoor or large hood operation is recommended because of the copious fumes produced by some fluids. Fig. 10 is a schematic showing the pilot-operated solenoid valve, nitrogen gas bottle, reservoir column, and 0.006-in. orifice. Fluid is introduced through the valve at the top, and the tubing is filled (30 ml). Valves are closed, and an acetylene torch is ignited and held 4 ft from the orifice. Push-button operation of the solenoid valve places 2000-psi nitrogen over the fluid column. Fluid sprays from the orifice, and the operator moves the torch toward the orifice. Distance from the orifice where ignition occurs and flame size are reported. This test appears to correlate to some extent with both the full-scale, high-pressure spray flammability test, and also the new low-pressure spray flammability test.

References

1. Aeronautical Material Specification AMS-3150C, "Fluid, Hydraulic-Fire Resistant," (an SAE publication).
2. Method of Test for Flash and Fire Points by Cleveland Open Cup, D 92 - 57, 1965 Book of ASTM Standards, Parts 11, 17.
3. Electro Engineering Products Company, Chicago, Illinois.

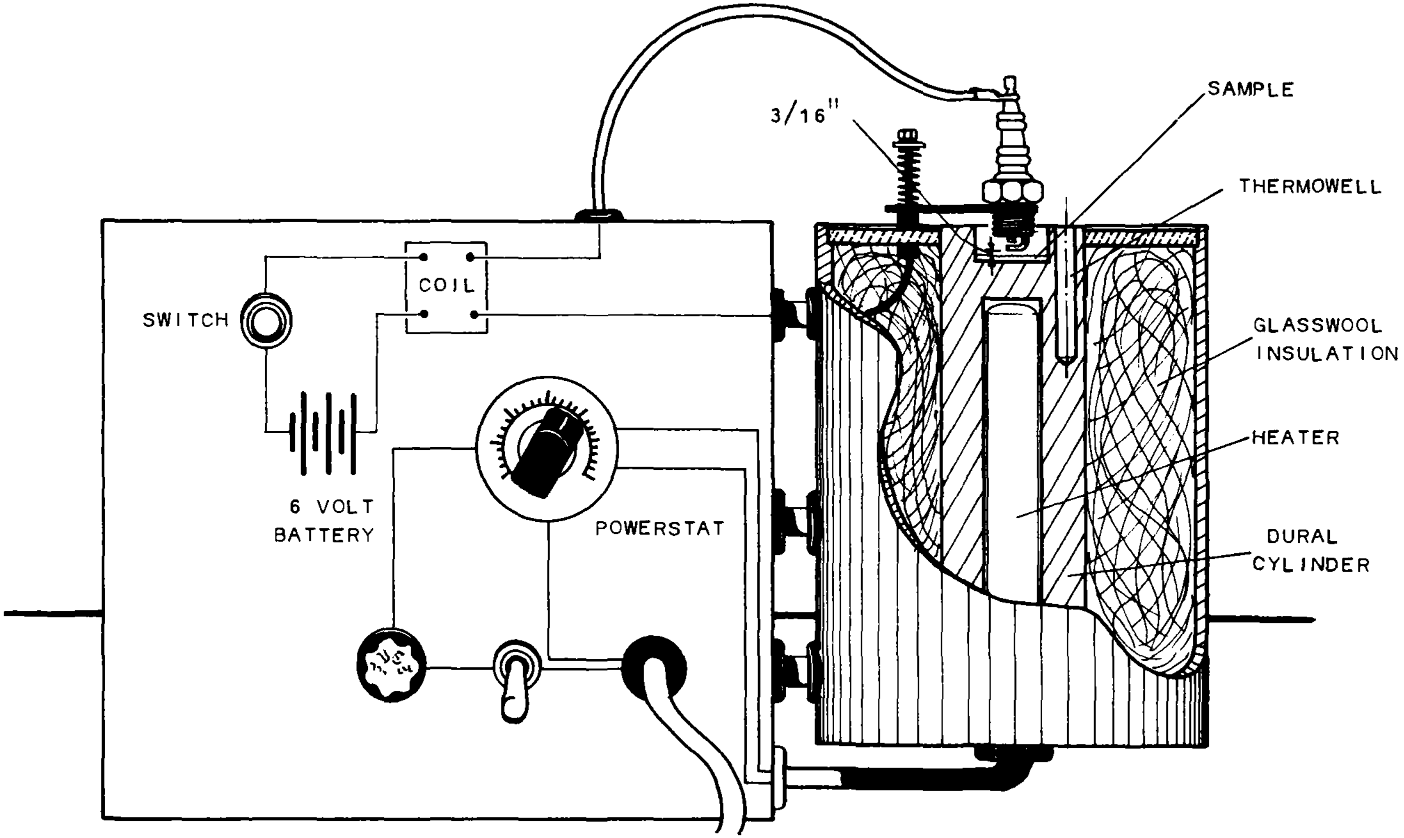
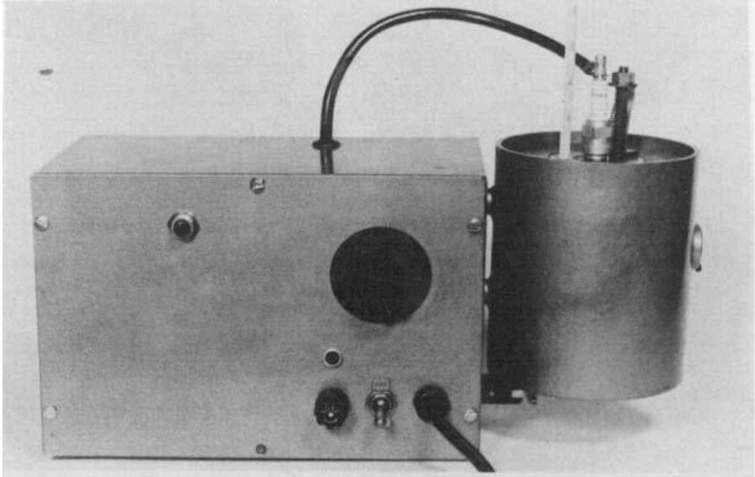


Fig. 1--Microflash Apparatus.



Assembled

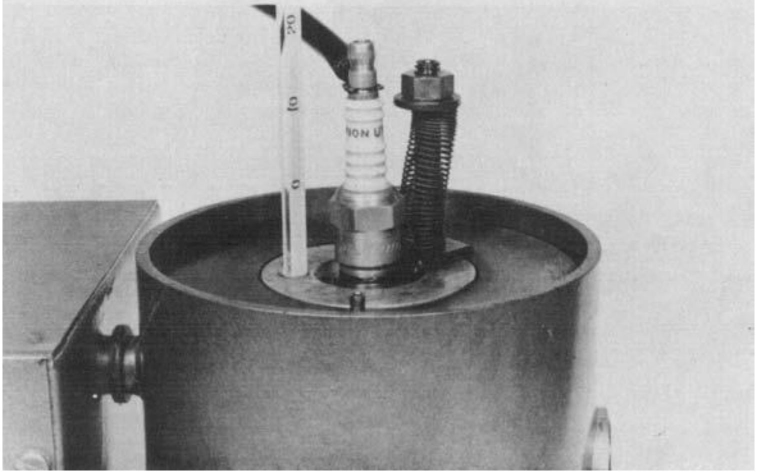


Fig. 2--Microflash Apparatus (Assembled) and Test Cup.

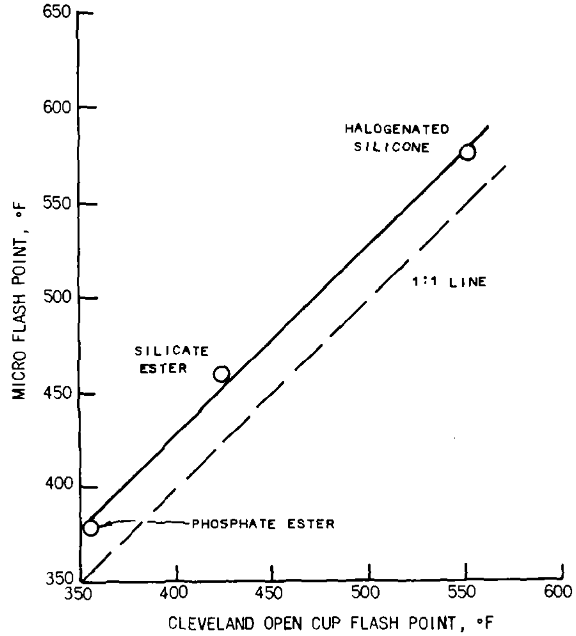


Fig. 3--Flash Points of Hydraulic Fluids Micro Versus Cleveland Open Cup.

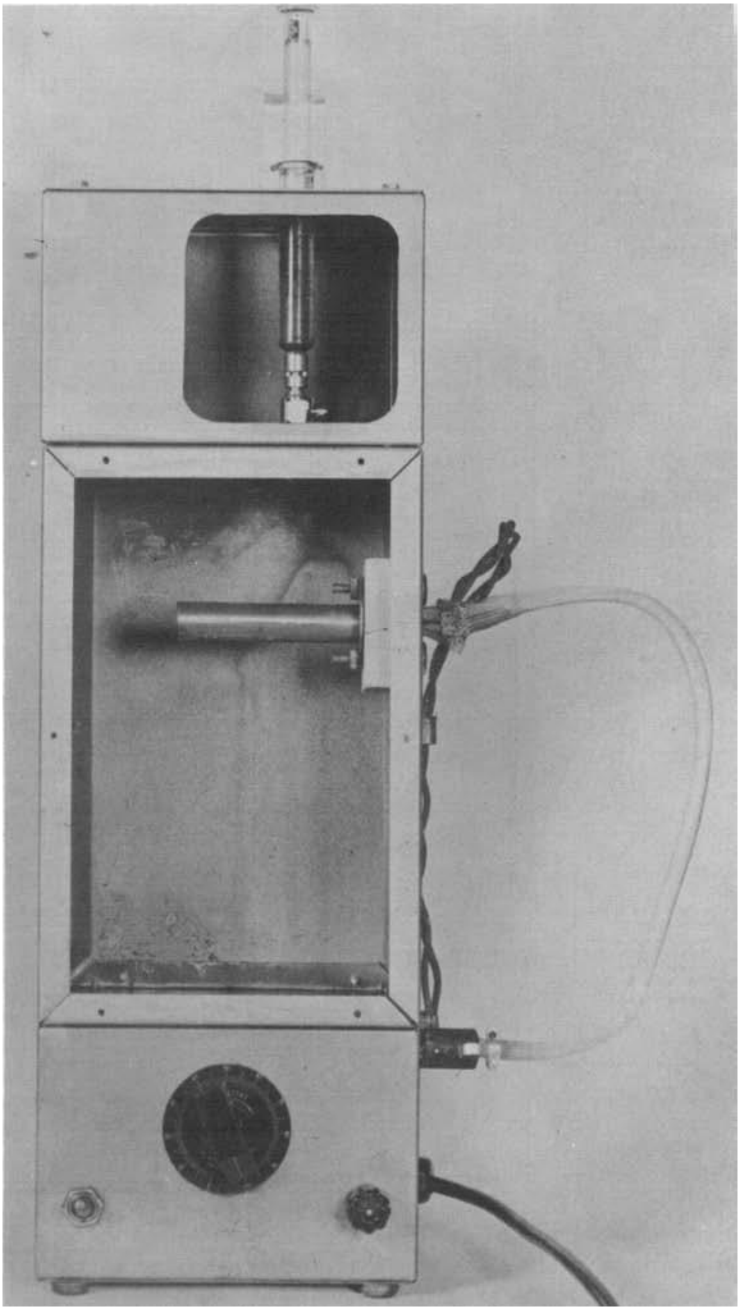


Fig. 4--Photo of Small Scale "Hot Manifold" Apparatus.

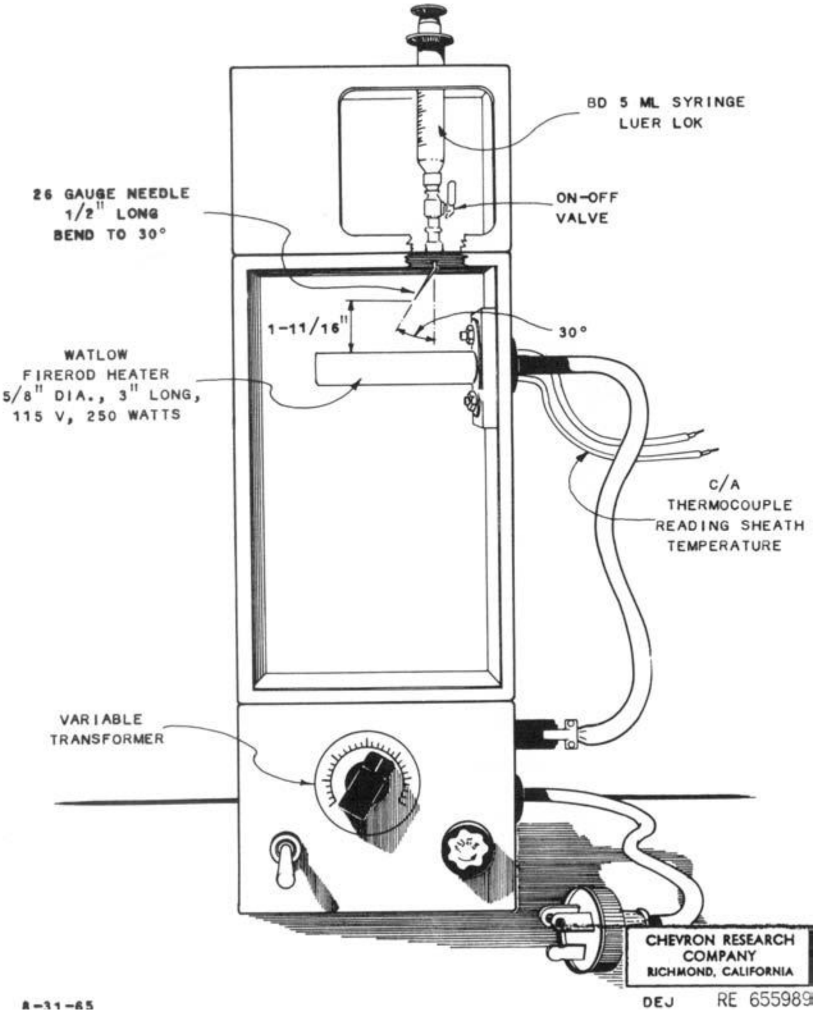


Fig. 5--Diagram of Small Scale "Hot Manifold" Apparatus.

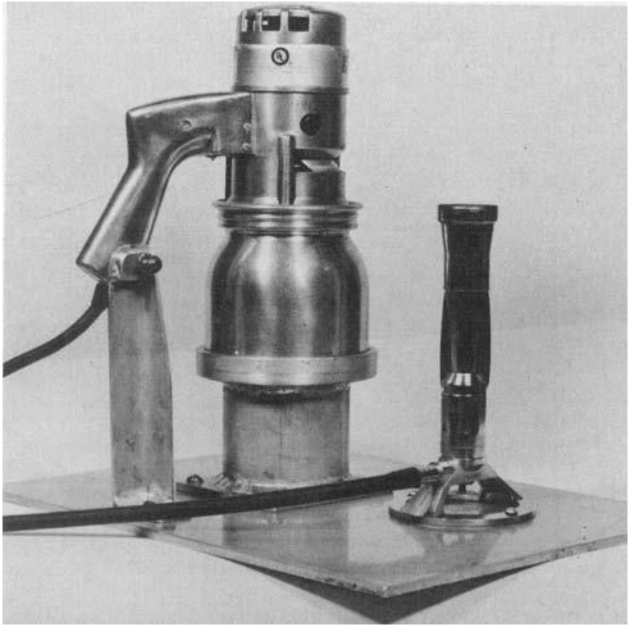


Fig. 6--Photo of Low Pressure Spray Flammability Apparatus.

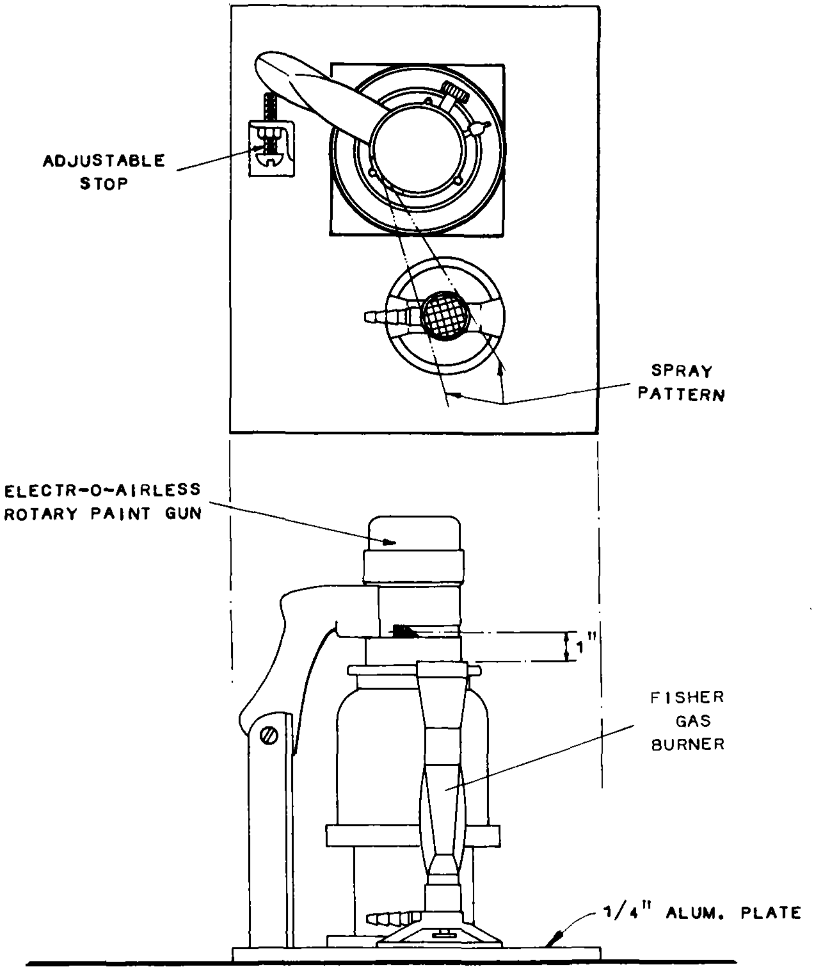


Fig. 7--Diagram of Low Pressure Spray Flammability Apparatus.

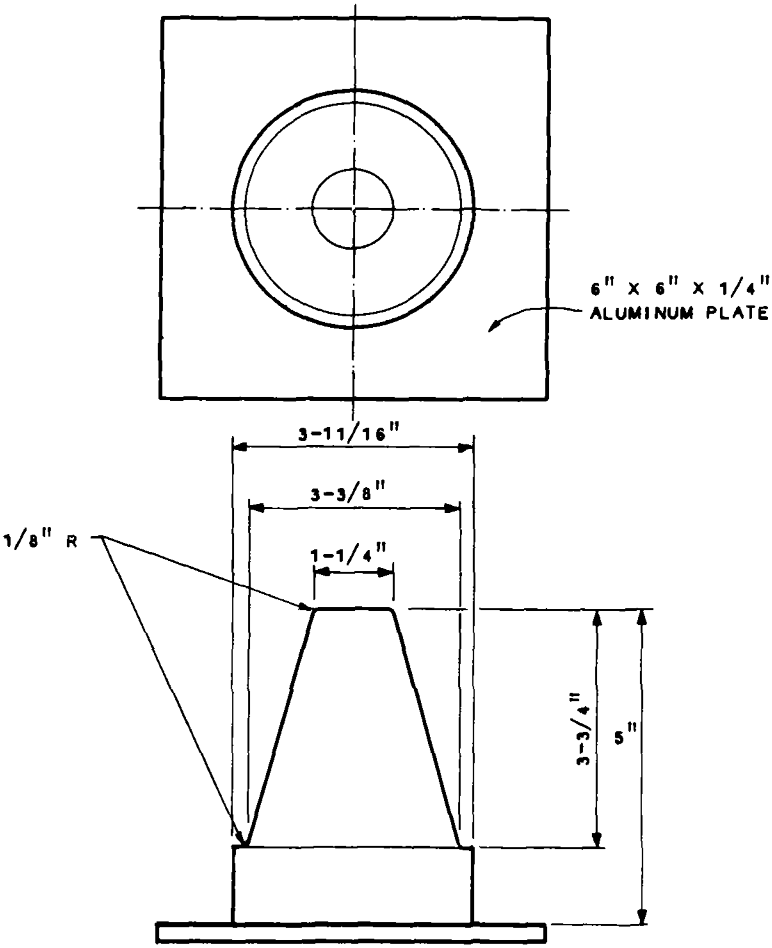


Fig. 8--Aluminum Cone Form.

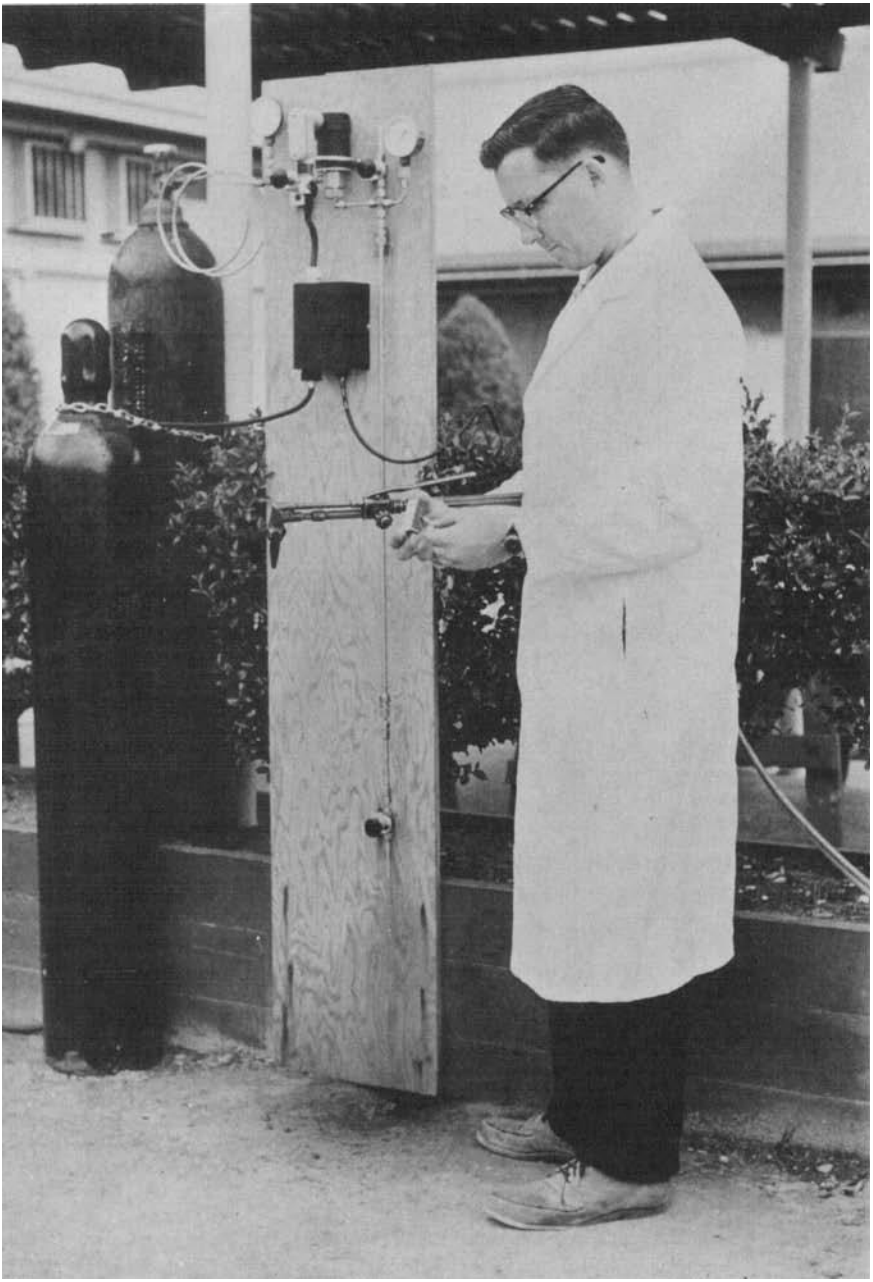


Fig. 9--High Pressure Spray Flammability Apparatus.

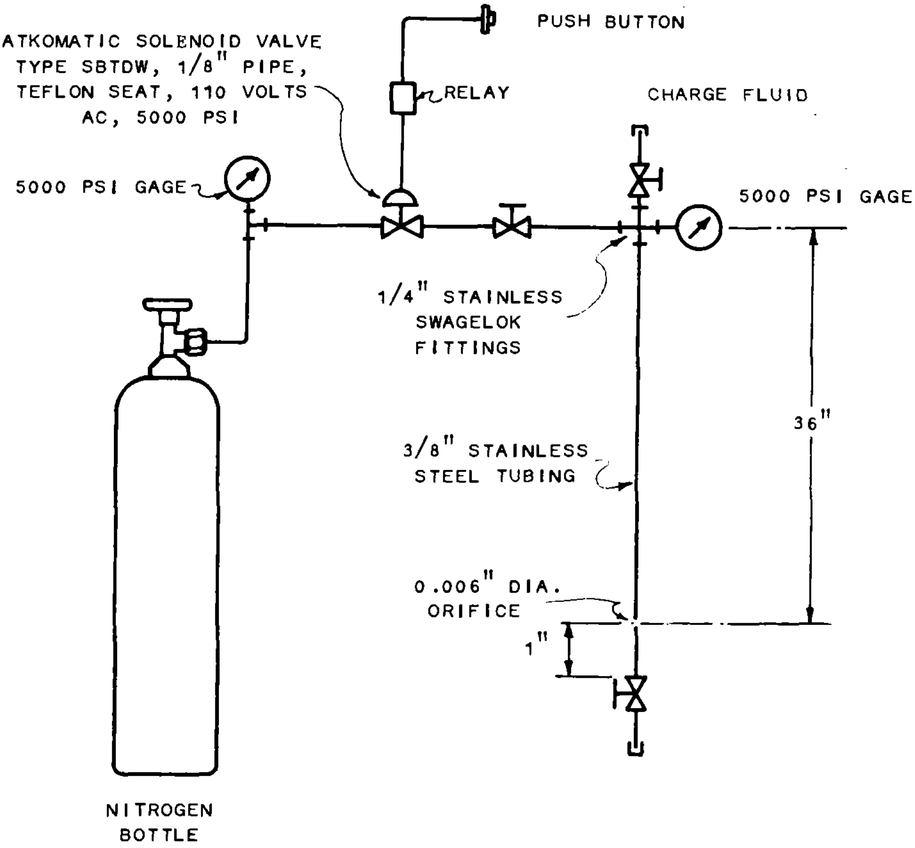


Fig. 10--Small Scale High Pressure Spray Flammability Apparatus.

SPONTANEOUS IGNITION TESTING FOR HIGH SPEED AIRCRAFT

By W. F. Hamilton[1] and R. A. Holloway[2]

ABSTRACT: The hydraulic systems of supersonic aircraft which are designed to cruise at altitudes of approximately 70,000 ft must operate under conditions which impose severe physical and chemical stresses on the fluid. Appraisals of the thermal stability, the oxidative stability, the lubricity, the effect of close-tolerance moving parts, and the flammability of the fluid should be made before its pumpability is determined. Performance tests have been selected which permit a practical estimation of the five prerequisite factors, but the most complex and difficult evaluation is that of flammability.

Under precisely defined conditions, spontaneous ignition of a flammable fluid occurs in the presence of an oxidant only when the temperature is at or above that required to generate heat by oxidation at a rate which is greater than the rate of the dissipation of this heat to the surroundings. This temperature may or may not be the "spontaneous ignition temperature" as preflame of combustion usually occurs by reaction mechanisms which differ from those encountered in the flames themselves. However, ignition will not occur below this temperature, so in order to evaluate the hazard of a hydraulic fluid fire in high speed aircraft, measurements must be made in surroundings which closely approximate the actual environment and conditions in the airplane. Essentially, for aircraft now being developed, this entailed the fabrication of a small hot compartment of titanium alloy, using milled skins with typical stiffeners and fastenings, means for introducing precisely variable quantities of hot, atomized test fluids, controlling and measuring tempertures and pressures ("altitudes"), and also measuring pressure transients and ignition delays. The resulting apparatus, measurements and techniques are described and discussed.

KEY WORDS: fire resistance, flammability, fire tests, spontaneous ignition, hydraulic fluids, supersonic transport

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[1] Structures and Materials Laboratory, Chemical Group, Rye Canyon, Lockheed-California Company, Burbank, California.

[2] Structures and Materials Laboratory, Physics Group, Rye Canyon, Lockheed-California Company, Burbank, California.

Aircraft designed to cruise at altitudes of approximately 70,000 ft at speeds approximating Mach 2.7, impose severe high-temperature stresses and severe loads on the hydraulic fluid used for controls. This fluid, circulating continually, must be capable of "start-up" at -45 F. Also, it could acquire a bulk operating temperature of about 425 F during flight and may be subjected to considerably higher local "hot spot" temperatures during use at locations where heat is generated by mechanical or fluid friction. An operating pressure of as high as 4000 psi may be used to supply the necessary energy to the servo mechanisms, so the pumps which maintain this pressure will operate under conditions which require a relatively good lubricant. The environment of portions of the hydraulic system may reach temperatures as high as 560 F near the bleed air duct at cruising altitudes where ambient pressures are low; or in an uncooled section of the pressurized structure, temperatures approaching 420 F may accompany atmospheric pressures between about 600 and 760 torr.

The figures quoted above, with others, enable prediction of a minimum desirable spontaneous ignition temperature (SIT) versus altitude, as shown in Fig. 1. As the SIT is subject to many variables, 100 F has arbitrarily been added to the maximum expected flight temperatures to establish the minimum allowable SIT at various altitudes. Ignition of flammable liquids occurs only when the liquid is in the presence of an oxidant under conditions such that the rate of heat generation by oxidation is greater than the rate of dissipation to the environment. The fact that an exotherm occurs, accompanied by a rise in temperature, does not necessarily mean that the SIT has been reached as preflame combustion usually occurs by a different reaction mechanism than that involving a flame. Thus, the conditions under which the SIT is measured have a decided effect on the value obtained, and the value obtained by introducing a hydraulic fluid into an Erlenmeyer flask heated in an electric furnace(1) may be entirely different from the one obtained by spraying the same fluid into a heated compartment which is a facsimile of a typical portion of the aircraft structure. For example, at ambient pressure a typical deep dewaxed mineral oil has a SIT of 740 F in the ASTM Method D-286 apparatus, whereas in our facsimile apparatus the SIT value is 485 F. This is 35 F below the minimum allowable value (Fig. 1) and could indicate a fire hazard in case of leakage or rupture of a hydraulic line.

Present conventional testing methods are not entirely adequate for prediction of the kind of service a candidate fluid will deliver under actual operating conditions. Accordingly, in selecting hydraulic fluid for the proposed new high-speed/high altitude transport, the usual inspection information (namely, flash, fire, pour, thermal and oxidation stability, etc.) has been supplemented with four simple performance tests:

1. Stiction: A close-fitting Nitralloy 135 (see Fig. 2) spool and sleeve valve assembly is submerged in approximately 150 ml of test fluid in a 500 ml round-bottom flask. Inerting nitrogen is slowly bubbled through the fluid which is cycled by heating about 2 hr to a maximum of 450 F, then cooling 2 hr. The number of cycles obtained before piston seizure provides a practical appraisal of thermal stability and the effect of the fluid on typical hydraulic system parts. To some degree, this performance test anticipates corrosion and thermal stability behavior.

2. Lubricity: A Shell 4-ball tester, modified to provide for heating the oil and inerting the fluid with nitrogen, is used. The scar formed on SAE No. 52100 steel balls at 1020 rpm, with a 20-kg load, at 400 F, after running 1/2 hr affords an indication of the lubricity of the fluid. Thermal stability in the presence of finely divided metal particles is also indicated.

3. Seal Test for Fluid Evaluation: A steel block with a cavity of approximately 50 ml volume, fitted with a reciprocating rod passing through metal seals at two opposite sides of the cavity, is provided with electric heaters and means for introducing the test fluid into the cavity under pressure (Fig. 3). The cavity is heated to 425 F, the rod stroke is 3 in., and the rate is 60 cycles/min. Both seals and shaft may be faced with tungsten carbide. The test indicates performance to be expected with metal particles present in the pressurized fluid under thin film oxidative conditions at elevated temperatures.

4. Flammability: The spontaneous ignition temperature of the hydraulic fluid, depending as it does on rates of heat generation and dissipation, should be measured in an environment which closely approximates a representative portion of the aircraft itself. As one of the more likely causes of a hydraulic fluid fire would be accidental failure of a line or fitting, and because the working fluid pressure will be high, the fluid would probably be ejected as a mist or spray. The structure of the compartment enclosing the line may be titanium alloy, with temperatures and pressures depending upon the speed, altitude, operating conditions, location in the airplane, and the like. The test should indicate the hazard which would be involved in case of sudden release of hot fluid spray into a hot compartment.

SIT APPARATUS

A titanium alloy (8Al-1Mo-1V) box, 8 in. by 8 in. by 4 in. was constructed using milled skins for the top and bottom, and suitably flanged sheet for the sides (Fig. 4). The bottom and top were bolted on and a spring-loaded circular plate was fitted over an opening on one side for pressure relief. Five specially made electric heating plates covered practically all of the external surface except the pressure relief disk. Temperatures of each side of the box were measured and controlled by peening and welding thermocouples into adjacent pairs of small holes drilled in the milled skins and box sides. Two additional thermocouples were mounted in the top of the box to measure gas temperatures. One was 1 in. above the center of the enclosure, and the other in the left front corner at the same level.

Test fluid was introduced from a mechanical atomizing nozzle fitted through the front of the box. Use of an electric interval timer and a solenoid valve enabled selection of the amount of fluid to be sprayed into the apparatus, and also enabled multiple shots of a controlled fluid volume. The fluid was heated electrically to 425 F and was under pressure approximating 1500 psi immediately upstream of the valve.

The entire box assembly was insulated with mineral wool (Fig. 5) and placed inside a large chamber (Fig. 6) which could be easily closed and sealed (Fig. 7) for evacuation to a pressure corresponding to any desired altitude. All ambient pressure measurements were made in the laboratory at the Lockheed Rye Canyon Research Laboratory at an altitude of 1310 ft. Pressure changes inside the titanium box were indicated and recorded by means of a pressure transducer and recorder. The system response was flat to 200 cps. Fluid pressure was regulated by means of a hand-operated piston pump and indicated by a pressure gage.

After a test, the atmosphere in the box could be completely changed, and the combustion products or oil vapor scavenged by flushing with compressed air. The final blast of dry air for this purpose was supplied from a bottle of compressed gas.

PROCEDURE

The fluid to be tested was charged into the clean vertical reservoir, hand pump, lines, filter, accumulator, and nozzle. The hand pump was clamped horizontally to the right-hand bench shown in Fig. 8. The oil pressure was then raised to 1500 psig. The temperatures of the five heating plates on the walls of the titanium box were then adjusted and controlled by means of four indicating and one of the two recording instruments on the left-hand bench (Fig. 9). After equilibrium was attained, the ignition chamber gas temperature and pressure recorders were turned on, the fluid temperature, pressure and injection interval checked and set, and the solenoid valve energizing switch closed to inject hot atomized fluid into the titanium enclosure. The chamber pressure recorder was closely observed, and normally a second injection of fluid was made as the pressure approached a maximum due to the first charge. By trial and error, a minimum ignition temperature[3] was determined by changing the chamber wall temperature in increments of 5 to 10 F, and varying the charge size to produce maximum pressure and air temperature rises.

SIT's at various altitudes were measured by the same general procedure, except that the outer box was closed and partial vacua were established to conform to the ARDC Model Atmosphere (1959) ("U. S. Standard Atmospheres," Supt. of Documents, U.S. Government Printing Office, Washington 25, D.C.)

RESULTS

Typical data recorded during runs with a specially refined hydrocarbon oil have been compiled to show what happens above, at, and below the SIT at several atmospheric pressures (Figs. 10, 11, and 12). Typical temperature and pressure change recordings of the air in the box, for runs at or above the SIT and below the SIT are illustrated in Figs. 13 and 14. These recordings indicate the magnitude and timing of the changes. Due to the mass of the thermocouples, the true time lags are actually shorter and peak temperatures are higher than indicated. Both are additionally limited by the relief plate, which serves the purpose of preventing excessive pressure build-up in the box.

Spontaneous ignition temperature measurements of several candidate fluids for use in the hydraulic system of high-speed aircraft are summarized in Fig. 15. For purposes of comparison, kerosine ("pearl oil") was measured at atmospheric pressure, and its SIT is appended, as is that for MIL-H-5606 fluid.

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[3] Usually spontaneous ignition is associated with a very rapid rise of temperature, and is normally accompanied by loud sounds and considerable smoke. With some fluids, at temperatures just below the SIT, some relatively small exotherms and pressure rises occur which might be considered hazardous. In case spontaneous ignition was inconsistent, the lowest temperature at which repeatable ignition occurred was considered to be the SIT. The criterion used for SIT determination was a relatively sudden rise of either temperature or pressure, or both.

DISCUSSION

With the apparatus used, it was important to ascertain the minimum box temperature at which ignition would occur. Accordingly, the duration of the spray was changed to vary the amount of fluid injected, and repeated trials were made. Spray durations approximating 90 to 110 msec were found to be very effective for injections of hydrocarbon fluids, and these were equivalent to volumes approximating 0.3 to 0.4 ml at 1500 psig with fluids having viscosities approximating 1.5 to 2.0 cs at 425 F.

The lowest SIT was ordinarily obtained by making two injections of about 100 msec each. The first injection usually caused a pressure rise in the box; and at the peak of this rise, the second spray injection was made. The ignition delay normally encountered approximated 20 sec. Greater time delays seldom occurred. However, in the case of the fluorosilicone fluid, about 1/2-sec injections resulted in ignitions with delays of 60 to 70 sec.

As the interior of the box became coated with soot, "varnish," silica, or other products of combustion, the SIT decreased until it reached an apparently constant value. Figure 4 shows the appearance of a typical coating from a hydrocarbon fluid, and Fig. 16 shows a similar coating from a silicate ester fluid.

The studies of Zabetakis and his co-workers(1,2,3) encompassed the effects of atmospheric pressure, of surface, and of atomization on the SIT of aircraft hydraulic fluids. Their results are not directly comparable with those reported in this work, and they differ primarily in the time lag before ignition. In general, the greater the lag, the lower will be the SIT.

The fluid pressure of 1500 psig was selected as an optimum between satisfactory atomization and dependable valve operation. Above about 1000 psig, injection pressure has little effect on ignition temperature.(1,2,3)

SUMMARY

1. A new apparatus is described which is a reasonable facsimile of a typical portion of the projected structure of a high-speed aircraft.

2. A procedure is outlined for determination of the SIT of a flammable fluid in the apparatus, and the effect of altitude on the measured value.

3. Experimental results are given for several candidate hydraulic fluids for the projected high-speed aircraft.

ACKNOWLEDGMENTS

The authors are partciularly indebted to R. E. Middleton for his interest and assistance in many phases of the work described; to Lane Hauck and Alvin Smith for help in experimental work; to D. G. Thompson and F. A. De Wan for many fruitful mechanical suggestions, and to D. Elliot for electrical aid and guidance.

REFERENCES

1. Zabetakis, M. G., Furno, A. L., and Miller, J. J. — "Research on the Flammability Characteristics of Aircraft Hydraulic Fluids," WADC TR57-151, Wright Aeronautical Development (May, 1957).

2. Zabetakis, M. G., Imhof, A. C., and Lang, F. W. — "Research on the Flammability Characteristics of Aircraft Hydraulic Fluids," WADC TR57-151, Supplement 1 (May, 1958).

3. Zabetakis, M. G., Scott, G. S., Imhof, A. C., and Lambiris, S. — "Research on the Flammability Characteristics of Aircraft Hydraulic Fluids," WADC TR 57-151, Part II (March, 1959).

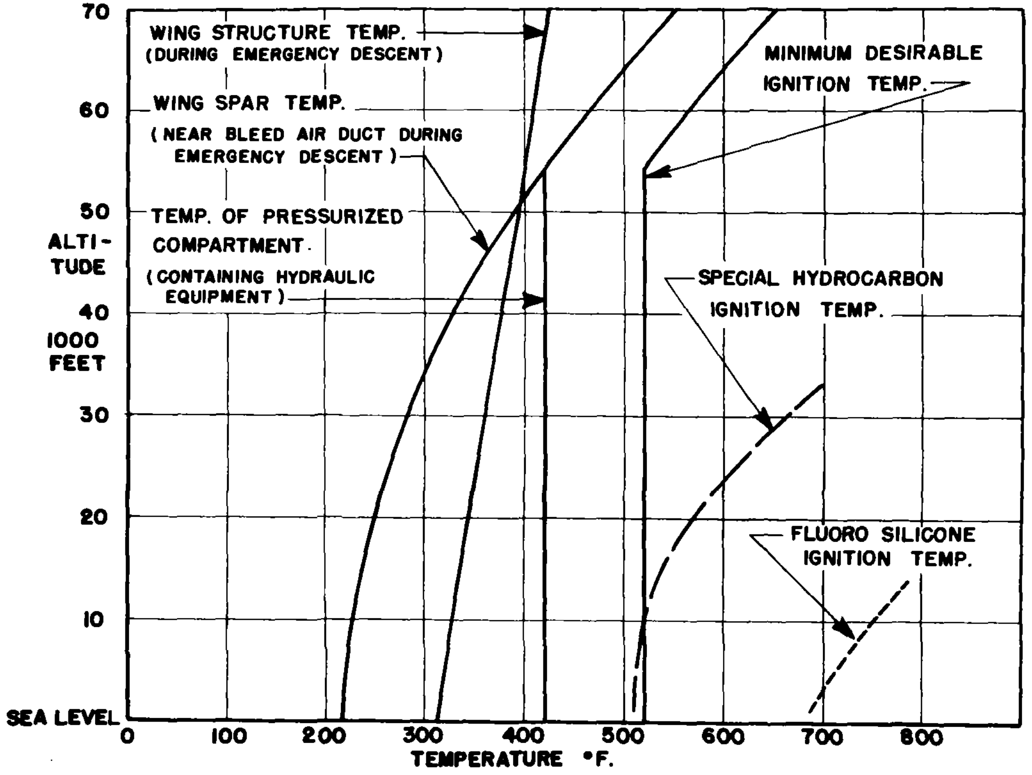
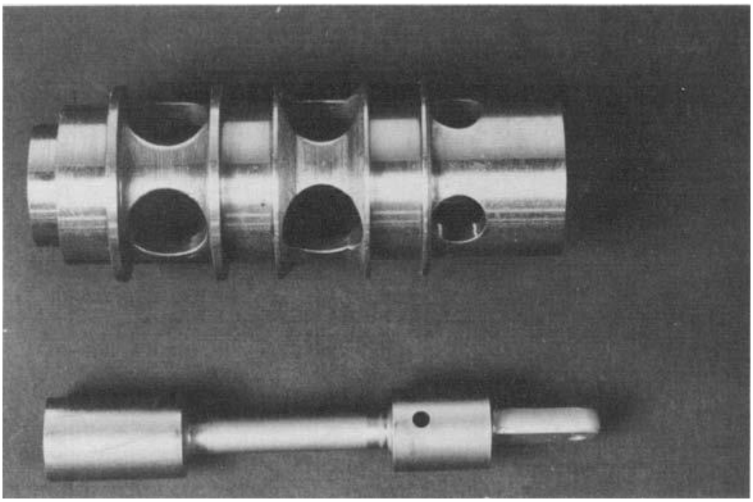
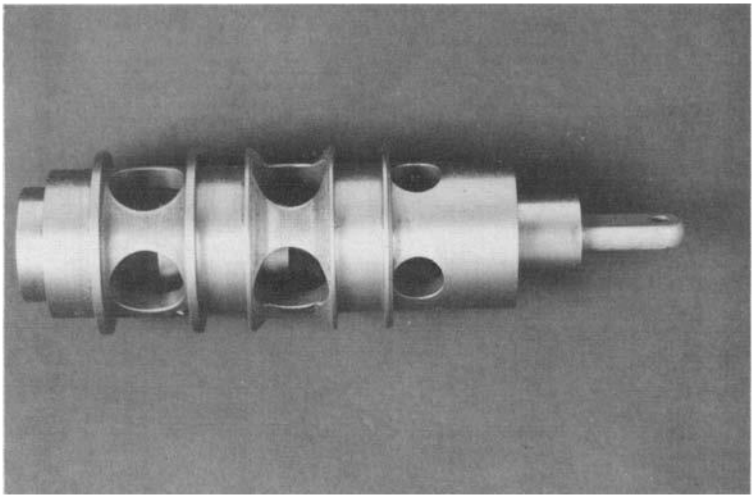


FIGURE 1 - HIGH SPEED AIRCRAFT TEMPERATURE VERSUS ALTITUDE.



(a) Sleeve and Spool



(b) Sleeve and Spool Valve Assembly

FIGURE 2 - STICTION TEST ELEMENT .

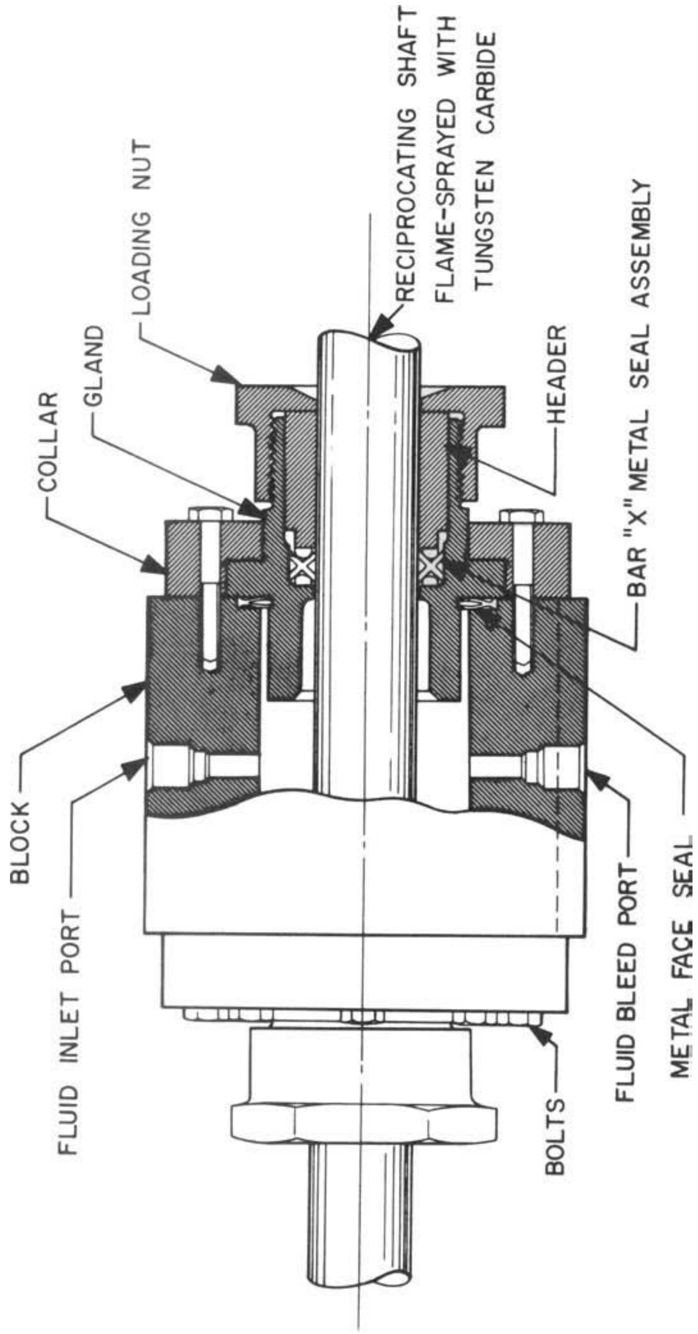


FIGURE 3 - FLUID EVALUATION SEAL TEST GLAND.

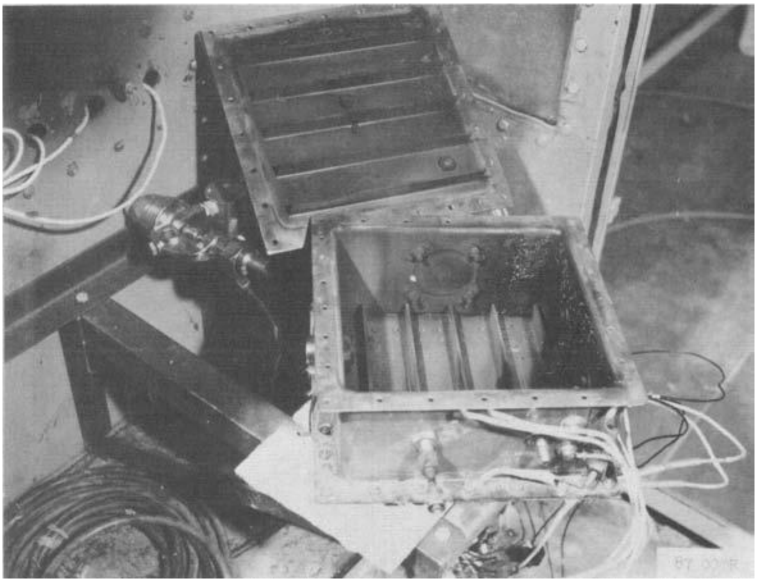


FIGURE 4 - INTERIOR OF SIT APPARATUS.

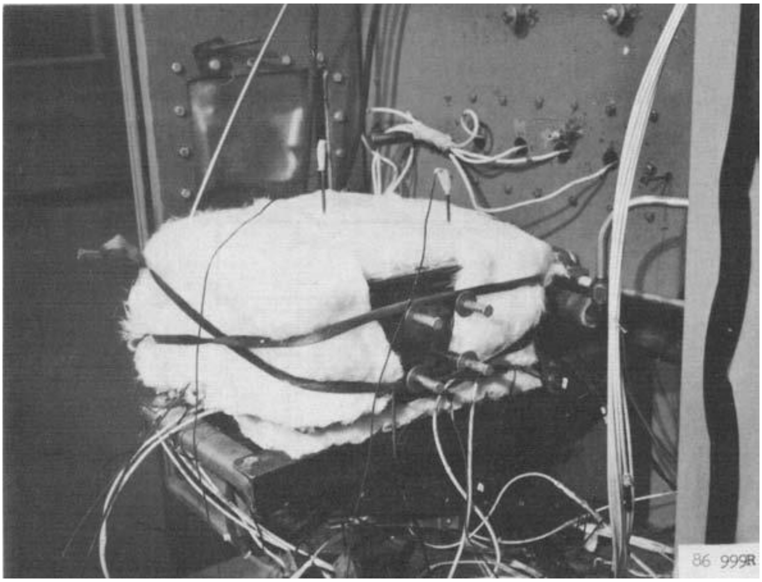


FIGURE 5 - EXTERIOR OF SIT APPARATUS.

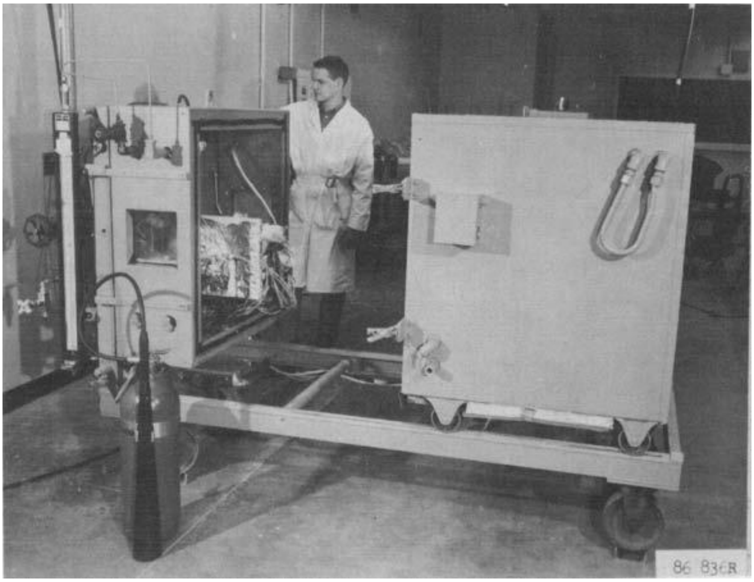


FIGURE 6 - APPARATUS IN OPEN ALTITUDE CHAMBER .

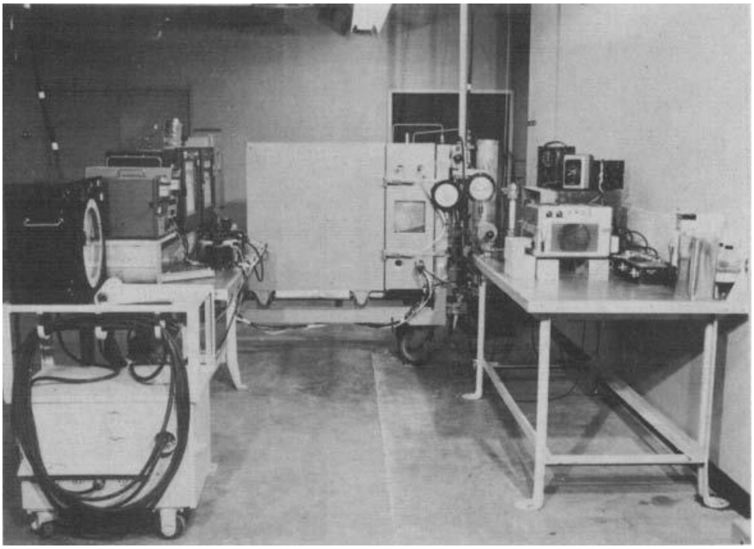


FIGURE 7 - CLOSED ALTITUDE CHAMBER WITH INSTRUMENTATION.

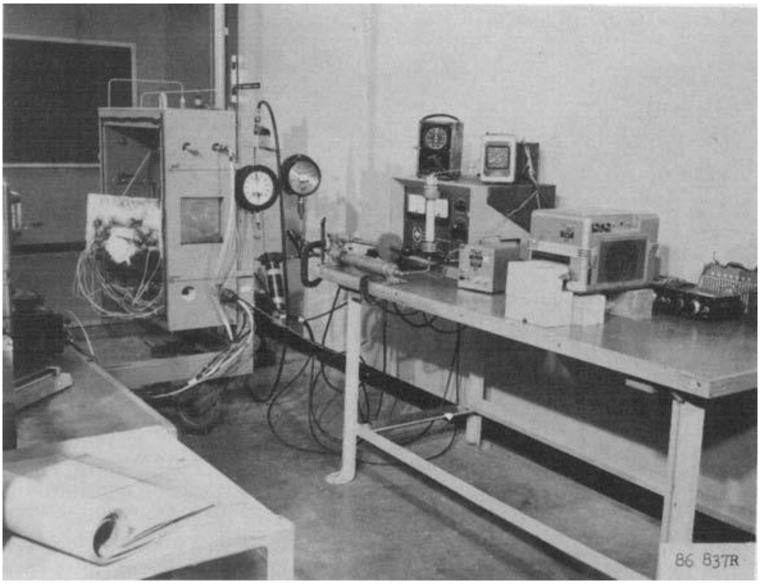


FIGURE 8 - METERING AND PRESSURE INSTRUMENTS.

FIGURE 9 - TEMPERATURE INSTRUMENTS

FIGURE 10 - AMBIENT PRESSURE SPONTANEOUS IGNITION TESTING DATA

FOR A SPECIAL HYDROCARBON FLUID.

VARIABLE	RUN NUMBER						
	1	2	3	4	5	6	7
Spray, msec[a]	300	290	388	348	382	417	388
Box Temp., deg F	540	520	510	510	500	500	500
Air Temp., deg F (center)	515	496	490	486	478	477	476
Explosion Temp., deg F	875	897	918	870	-	-	-
Cool Flame Temp., deg F	-	-	-	-	595	580	567
Temp. Increase, deg F[b]	360	401	428	384	117	103	91
Press. Increase, psi	3.5	3.4	3.9	5.1	0.7	0.9	0.6
Sound[c]	poof	hiss	whistle		-	-	-

[a] Total time required for two spray injections.

[b] Change of the gas temperature in the box.

[c] A definite sound was produced by each explosion, as indicated.

Runs were made at ambient pressure, 28.5 in. Hg (altitude 1310 ft).

FIGURE 11 - 6000-FT ALTITUDE SPONTANEOUS IGNITION TESTING DATA

FOR A SPECIAL HYDROCARBON FLUID .

VARIABLE	RUN NUMBER				
	1	2	3	4	5
Spray, msec[a]	325	328	316	320	318
Box Temp., deg F	520	520	510	510	500
Air Temp., deg F (center)	477	484	484	481	481
Explosion Temp., deg F[b]	823	880	-	-	-
Cool Flame Temp., deg F	-	-	602	597	575
Temp. Increase, deg F[c]	346	396	118	116	94
Press. Increase, psi	3.3	2.9	0.9	0.8	0.6

a Total time required for two spray injections.

b The sound produced by an explosion could not be heard outside of the vacuum chamber.

c Indicates change of the gas temperature in the box.

Runs were made at 24 in. Hg (altitude 6000 ft).

FIGURE 12 - 20,000-FT ALTITUDE SPONTANEOUS IGNITION TESTING

DATA FOR A SPECIAL HYDROCARBON FLUID.

VARIABLE	RUN NUMBER							
	1	2	3	4	5	6	7	8
Spray, msec[a]	255	240	250	240	239	255	253	266
Box Temp., deg F	600	590	580	570	560	550	540	530
Air Temp., deg F (center)	575	568	556	545	535	527	519	506
Explosion Temp., deg F[b]	699	682	666	687	-	-	-	-
Cool Flame Temp., deg F	-	-	-	-	676	681	657	620
Temp. Increase[c]	124	114	110	142	141	154	138	114
Press. Increase, psi	1.8	1.3	1.3	1.4	0.8	0.7	0.6	0.6

[a] Total time required for two spray injections.
[b] The sound produced by an explosion could not be heard outside of the vacuum chamber.
[c] Indicates the change of the gas temperature in the box.

Runs were made at 13.8 in. Hg, (altitude 20,000 ft).

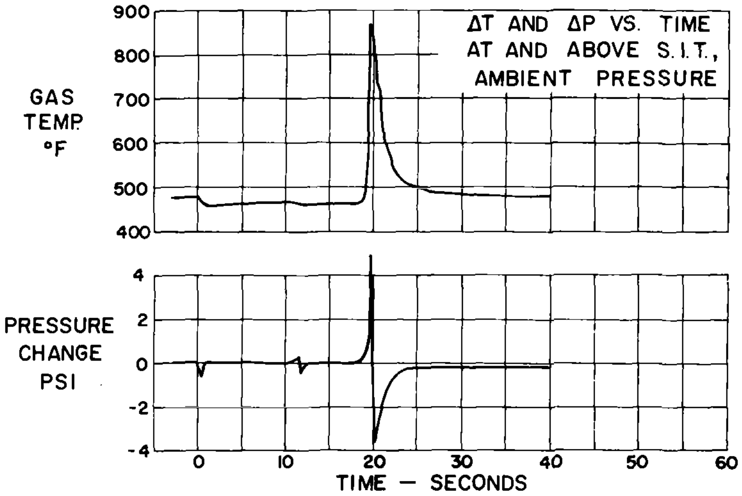


FIGURE 13 - TYPICAL TEMPERATURE AND PRESSURE CHANGE RECORDINGS FOR RUNS AT OR ABOVE THE SIT.

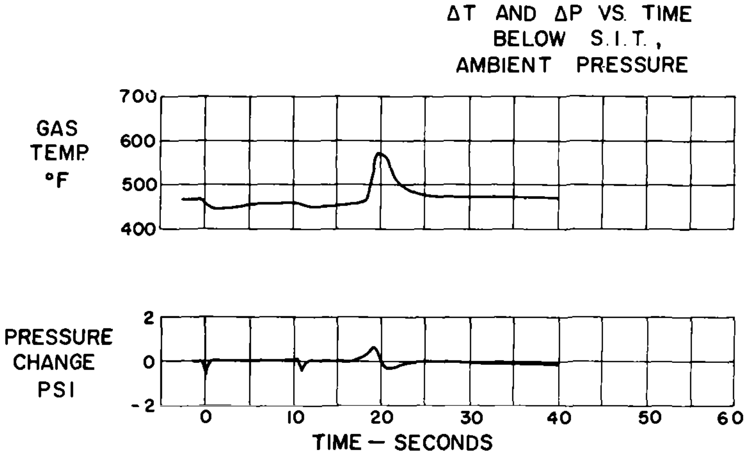


FIGURE 14 - TYPICAL TEMPERATURE AND PRESSURE CHANGE RECORDINGS FOR RUNS BELOW THE SIT

FIGURE 15 - SPONTANEOUS IGNITION TEMPERATURE OF TYPICAL HYDRAULIC FLUIDS

FLUID	SIT, DEG F AT ALTITUDE, FT				
	1,310	6,000	20,000	40,000	70,000
Deep Dewaxed:					
Mineral Oil	485	-	-	-	-
N_2 Inerted	490	-	-	-	-
CO_2 Saturated	490	-	-	-	-
Silicate Ester	520	520	540	$> 700^a$	$> 700^a$
Special Hydrocarbon	510	515	570	700 at Approx. 35,000 ft	$> 700^a$
Fluorosilicone	685	720	750 at 10,000 ft	> 750	> 750
MIL-H-5606	490	-	-	-	-
Kerosine	455	-	-	-	-

[a] Highest temperature tested.

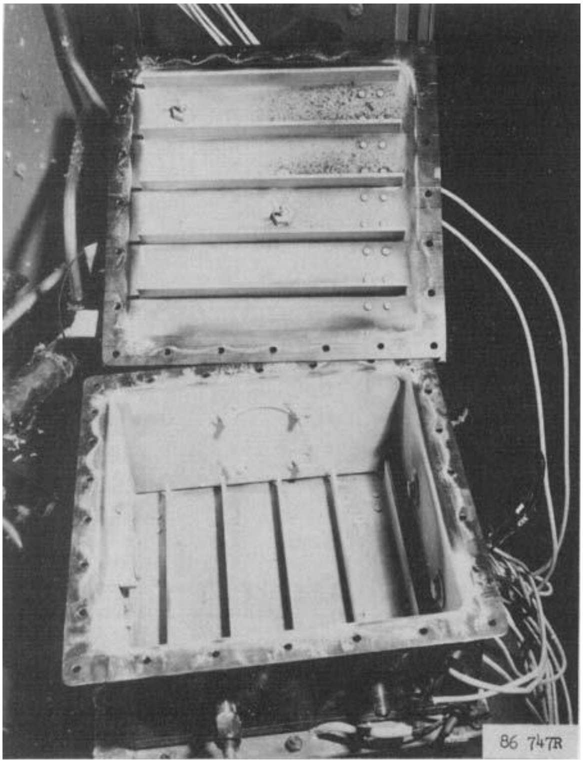


FIGURE 16 - SILICA RESIDUE IN APPARATUS.

SURVEY OF TECHNIQUES FOR THE EVALUATION OF THE FIRE RESISTANCE PROPERTIES OF AVIATION HYDRAULIC FLUIDS

By R. Alan C. Ker [1]

REFERENCE: R. Alan C. Ker, "Survey of Techniques for the Evaluation of the Fire Resistance Properties of Aviation Hydraulic Fluids," Fire Resistance of Hydraulic Fluids, ASTM STP 406, Am. Soc. Testing Mats., 1966.

ABSTRACT: Five types of aviation hydraulic fluid were examined - mineral, snuffer, phosphate ester, siloxane, and chlorosilicone.

Statistics show that more aircraft accidents are associated with wheel brakes than with any other components. A hot brake test simulating conditions that could develop from a fractured hydraulic pipe during braking revealed little difference in the ignitability of phosphate esters, mineral type and a snuffer fluid D.

Only one fluid, a snuffer fluid C, withstood ignition with brake heat pack temperature of 700C.

Several well known simpler laboratory techniques were used. Of these a combined high pressure spray/manifold test not only gave a reasonable degree of correlation with the hot brake test results, but also differentiated between the degree of flame propagation evidenced by these fluids.

The shape of the nozzle in the spray tests influenced results whilst fluid temperature and pressure were found to have generally little significant effect.

KEY WORDS: fire resistance, fire tests, flammability, hydraulic fluids

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Modern aircraft employ considerable lengths of hydraulic line for the actuation of moving parts. The hydraulic fluid in most subsonic aircraft is pressurized at 3000 psi and certain supersonic transport (SST) projects will employ 4000 psi. Fracture of these lines or leakage from faulty couplings will inevitably give rise to the fine mists or sprays which are a serious flammability hazard.

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[1] Chemist-in-Charge, Hydraulic Fluids Laboratory, Castrol Limited, Bracknell, Berkshire, England.

Hydraulic lines pass through most sections of the aircraft, and any leak in the vicinity of hot engine manifolds, overheated brakes and wheels, electric sparks, or sparks generated by friction, could be disastrous.

The fire resistance behavior of a hydraulic fluid will be influenced by many factors: the basic physical properties of the fluid, the amount of air or draft present to assist combustion, the nature of the source of ignition, and the amount of the flow of fluid whether in the form of spray or stream, the pressure under which the fluid flows, and other factors. Therefore, the behavior of a fluid involved in an inboard fire such as in a wheel bay or inside an engine nacelle is likely to be very different from that when sprayed over an overheated wheel brake during landing or rejected take off as a result of a fractured hose.

It appears that wheel brake fires are the major cause of aircraft accidents, from a study made by R.L. Paullin(1)in 1959 of 93 accidents. In this survey it was found that 78 were caused by spray or stream of fluids and mainly on hot surfaces such as wheel brakes. More recent investigations of airline statistics bear out this earlier finding.

It is for this reason that particular emphasis is given in this paper to the study of wheel brake fires in considering the applicability of known techniques for the evaluation of the fire resistance performance of hydraulic fluids by comparing the results of such tests with those obtained under simulated conditions. Inboard fires in confined compartments would warrant equally specific evaluations.

SELECTED LABORATORY TECHNIQUES

In order to investigate the relationship between methods of evaluating the fire resistance performance of aircraft hydraulic fluids and a method simulating wheel brake fires, certain known laboratory tests have been selected as follows:-

1. Open Flash Point(2)(Pensky-Martens IP Method 35/42)
2. Closed Flash Point(3)(Pensky-Martens IP Method 34/58)
3. Fire Point(2)(Pensky-Martens IP Method 35/42)
4. Spontaneous Ignition Temperature (SIT)

A Moore's ignition tester (4) was used for these tests. Dry air is blown at a controlled rate through a nickel crucible 3.5 cm deep, 3.5 cm lip, and 2.7 cm base diameter placed in a steel block heated by a gas flame. The temperature is measured by a thermometer inserted in a hole drilled into the block. The block is heated to a temperature above the expected

SIT, and one drop of fluid is allowed to fall into the crucible. Ignition occurs if this temperature is above the SIT of this test fluid. The temperature is allowed to fall, and the lowest temperature at which ignition occurs is recorded as the SIT.

5. Spray Ignition Test

The AMS 3150 C (5) procedure requires fluid at room temperature pressurized under nitrogen at 1000 psi sprayed through a straight-edged orifice 0.0145 in. in diameter. Attempts are then made to ignite the spray at increasing distances to the limit of the spray. Other modifications were carried out in order to examine:-

(i) The effect of raising the temperature of the fluid by the use of heating coils round the fluid pressure vessel.
(ii) The effect of producing a finer spray by the use of an 80 deg. oil burning nozzle.
(iii) The influence of reducing the fluid pressure to 200 psi.

6. Hot Manifold Test

AMS 3150 C (5) was adopted which involved an internally heated stainless steel manifold maintained at 700C. Ten milliliters of the fluid are poured onto the tube from a buret within 40 to 60 sec. The manifold was thoroughly cleaned each time by scraping with an iron wire brush. Additional tests were conducted at different manifold temperatures.

7. Hot Manifold/High Pressure Spray Test(6)

The high pressure spray of test (5) was allowed to impinge on the hot manifold of test (6). One pass consisted of spraying at a distance of 4 ft from the manifold, slowly approaching to 6 in. and then withdrawing to 4 ft before shut-off.

The following aspects were examined:-

(i) Manifold temperatures up to 780C(maximum temperature obtainable) with certain fluids.
(ii) The influence of straight-edged and 80 deg. oil burning nozzles.
(iii) Fluid temperature variations.
(iv) Fluid pressure variations.

8. Shrouded Manifold/Spray Test

As a result of experience gained with the Vanguard hot brake test (described later) it was considered that greater correlation between the results on this test and the manifold/spray test could be achieved by restricting the air supply in the vicinity of the manifold.

The manifold of the AMS 3150 C test was therefore enveloped by a cylindrical steel shroud allowing a ½ in. clearance between the shroud and the manifold. The spray from the pressure vessel at 1000 psi and using the straight-edged nozzle was directed through a 1 in. aperture running partly the length of the shroud. The spray was allowed to fall tangentially upon the heated manifold.

In order to clean the manifold after each test, the shroud was made in two sections attached by clips as indicated in Fig.1. A hole was made at one end at the periphery of the shroud to ensure that any fluid trapped might drain away rapidly.

FLUIDS EXAMINED

The classes of aviation hydraulic fluids examined were as follows:-

a. Phosphate esters (A and B)
b. Snuffer fluids (C and D)
c. A chlorosilicone fluid
d. A siloxane fluid
e. Mineral oil to MIL-H-5606

The snuffer fluids consisted of mixtures of halogenated materials (providing the snuffing action) and synthetic esters.

RESULTS

1. Open and Closed Flash, Fire Point, and Spontaneous Ignition Temperatures.

Fluid	Temperature deg C			
	Closed Flash	Open Flash	Fire Point	SIT
Phosphate Ester A	182	193	224	> 500
Phosphate Ester B	a	179	207	> 500
Snuffer Fluid C	163	200	357	429
Snuffer Fluid D	132	188	232	411
Chloro-silicone	204	302	335	459
Siloxane	100	218	260	428
Mineral Oil MIL-H-5606	93	104	110	328

a Unobtainable; pilot flame extinguished by vapor.

2. Hot Manifold Test (AMS 3150 C Type)

Fluid	Manifold Temp Deg C	Results
Phosphate Ester A	700	No burning
	725	Burnt on manifold not in tray
Phosphate Ester B	700	No burning
	725	Burnt on manifold not in tray
Snuffer Fluid C	750	No burning
	775	Some Flashing. No burning in tray
Snuffer Fluid D	600	No burning
	625	Burning on manifold not in tray
Chloro-silicone	525	No burning
	550	Burnt on manifold not in tray Burning droplets fell into tray
Siloxane	500	No burning
	525	Burnt on manifold not in tray
MIL-H-5606	600	No burning
	625	Burnt on manifold and in tray

3. Spray Ignition

Fluid	Fluid Temp Deg C	Fluid Pressure, psi	Fluid applied at distance from orifice	Results	
				Straight-edged nozzle	80 deg Oil Burning nozzle
Phosphate Ester A	R.T.	1000	4 ft to 3 in.	'Flame thrower' effect s/e[a] on removal of flame	Burning 1 to 2 sec Burning at 3 in. for 5 sec s/e
	R.T.	200	4 ft	Flashing	---
			3 ft to 3 in.	No burning	
	70	1000	4 ft	---	Burnt for 2 sec s/e
			3 ft		Sustained burning
Phosphate Ester B	R.T.	1000	4 ft to 3 in.	('Flame thrower' effect (s/e on removal of (flame	---
	70	1000	4 ft to 3 in.		Flashing 4 to 2 ft Sustained burning for 12 sec s/e below 2 ft
Snuffer Fluid C	R.T.	1000	4 ft to 1 ft	'Flame thrower' effect s/e on removal of flame	No burning at any distance
			<1 ft	No burning	
Snuffer Fluid D	R.T.	1000	4 ft to 2 ft	Occasional flashing	
			<1 ft	'Flame thrower' effect s/e on removal of flame	
	70	1000	4 ft to 2 ft		Flashing at 4 to 2 ft Burnt for 7 sec s/e at 1ft

[a] self-extinguished

3.Cont'd Fluid	Fluid Temp Deg C	Fluid Pressure, psi	Fluid applied at distance from orifice	Results: Straight-edged nozzle	Results: 80 deg Oil Burning nozzle
Snuffer Fluid D Cont.			1 ft to 3 in.		Continuous burning at 3 in.
	100	1000	4 ft to 3 in.	'Flame thrower'effect s/e on removal of flame	
	200	1000	4 ft to 3 in.	'Flame thrower'effect s/e on removal of flame	
Chloro silicone	R.T.	1000	4 ft to 3 in.	Intense'flame thrower' effect,s/e on removal of flame	4 to 3 ft Flashed 2 ft Burnt for 10 sec 1 ft Continuous burning
	70	1000	4 ft to 3 ft 2 ft		Flashing 4 to 3 ft Burnt continuously at 2 ft.
	R.T.	200	4 ft	Flashing	
			3 ft to 6 in.	Hardly flashing	
	160	1000	4 ft to 3 in.	Intense'flame thrower' effect s/e on removal of flame	
	160	200	4 ft to 3 ft	Flashing	
			2 ft to 6 in.	Burnt for 2 sec after flame removal s/e	
Siloxane	R.T.	1000	4 ft to 3 in.	('Flame thrower'effect	Continuous burning
	R.T.	200	4 ft to 3 in.	(s/e on removal of flame	
	160	1000	4 ft to 6 in.	'Flame thrower'effect s/e on removal of flame	

3.Cont'd Fluid	Fluid Temp Deg C	Fluid Pressure, psi	Fluid applied at distance from orifice	Results Straight-edged nozzle	 80 deg Oil Burning nozzle
Siloxane			6 in.	Burnt for 2 sec after flame removal s/e	
	160	200	4 ft to 6 in.	'Flame thrower'effect s/e on removal of flame	
			6 in.	Burnt for 2 sec after flame removal s/e	
MIL-H-5606	R.T.	1000	4 ft to 3 in.	'Flame thrower'effect s/e	Immediate sustained burning at 3 ft
	70	1000	4 ft to 3 in.	('Flame thrower'effect (burnt for 1/2 sec	
	120	1000	4 ft to 3 in.	(after flame removal s/e	

4. Hot Manifold Spray Test

	Fluid			Results	
Fluid	Temp Deg C	Pressure, psi	Manifold Temp Deg C	Straight-edged nozzle	80 Deg Oil Burning Nozzle
Phosphate Ester A	R.T.	1000	725	Burnt immediately	
	R.T.	1000	700	No burning	Immediate burning on manifold
	R.T.	1000	650		No burning
	70	1000	650		No burning
	R.T.	1000	725	Burnt immediately	
Phosphate Ester B	R.T.	1000	700	No burning	
	70	1000	700	No burning	
Snuffer Fluid C	R.T.	1000	760	No burning.Flashed when spray turned off.	
			750	No burning at all	Slight flashing when spray turned off
			780	Burnt on manifold with difficulty immediately s/e	
	R.T.	1000	725		No burning
	R.T.	200	750	Burnt at 2 ft on way in.No burning in tray	
	R.T.	200	725	No burning	
Snuffer Fluid D	R.T.	1000	600	(No burning till spray (turned off,then last	As with straight-edge nozzle

4. Cont'd

Fluid	Fluid		Manifold Temp Deg C	Results	
	Temp Deg C	Pressure, psi		Straight-edged nozzle	80 Deg Oil Burning Nozzle
Snuffer Fluid D Cont.				(few drops burned on (manifold not in tray	
			550	No burning	No burning
	R.T.	200	600	No burning until after 5 sec at 6 in.on way in. No burning in tray	
Chloro silicone	R.T.	1000	550	Burnt immediately	
	R.T.	1000	500	Burnt immediately	Immediate burning
			475	No burning	
Siloxane	R.T.	1000	550	Burnt explosively	Immediate burning, setting alight spray
	R.T.	1000	500	Burnt	Immediate burning but spray did not catch fire
			475	No burning	
	R.T.	200	500	Burnt at 4 ft on way in.	
	R.T.	200	475	No burning	
MIL-H-5606	R.T.	1000	600	(No burning till spray (turned off then burnt (on manifold and in (tray	No burning till spray turned off then burnt on manifold and in tray
	R.T.	1000	700		
			550	No burning	

4. Cont'd

Fluid	Fluid		Manifold Temp Deg C	Results	
	Temp Deg C	Pressure, psi		Straight-edged nozzle	80 Deg Oil Burning Nozzle
MIL-H-5606 Cont.	R.T.	200	600	Result as 1000psi	No burning till spray turned off then burnt on manifold and in tray

5. SHROUDED MANIFOLD SPRAY TEST

Fluid	Manifold Temp Deg C	Result
Phosphate Ester A and B	700	Burnt immediately
	675	Burnt after 5 sec delay
	650	No burning
Snuffer Fluid C	700	No burning
	725	Burnt with difficulty
Snuffer Fluid D	600	Burnt immediately
	500	Burnt after 3 sec delay
	525	No burning
MIL-H-5606	700	Burnt immediately
	600	Did not burn till pressure turned off
	550	No burning
Chloro silicone	550	Burnt explosively
	500	Burnt immediately
	475	Burnt after 2 sec delay
	450	No burning
Siloxane	-	Not tested

VANGUARD HOT BRAKE ASSEMBLY TESTS

Aircraft Braking Experience

The amount of heat generated in a typical multi-disk aircraft brake varies considerably during a braking operation. Ambient temperature, load, runway length, landing speed, the amount of reverse thrust used, and other factors would make their contribution to the amount of heat energy build-up in the brake. A modern large jet liner, fully laden, landing at Mexico City at the height of summer could produce temperatures in the vicinity of 700 to 800C heat pack under heavy brake conditions and as little as 100 to 200C unladen at a place like Montreal in the depth of winter using the full runway lengths. Rejected take-offs can put these figures up to over 1100C (7). All these temperatures are built up in a matter of approximately half a minute during braking after landing.

Figures 2 and 3 indicate the exposed position of high pressure hoses leading to the wheel brakes of Hawker-Siddeley Trident and Vickers Vanguard aircraft respectively. A burst tire on disintegration will often flail the high pressure hose and pipework, which may result in a stream or spray of hydraulic fluid over the wheel brakes. An attempt to simulate these conditions was therefore made.

Apparatus

A Vickers Vanguard wheel and brake were selected as being reasonably typical of units of the larger type in use in modern aircraft. (Fig.4 describes the whole assembly.) The rotors were replaced by 3 kw electric heaters shaped appropriately and shrouded by steel cases (Fig.5). Thermocouples were embedded in the stator pads. The brake was bolted to a flange supported horizontally by a long pipe which simulated the wheel axle.

In order to ensure the easy removal of the heavy magnesium alloy wheel thus to avoid excessive temperature build-up and the risk of causing a serious fire, the wheel was mounted on a frame borne by castors which moved on positioning rails. A series of switches linked to a pyrometer enabled the temperature of each of the pads to be taken at any moment. A thermocouple was also embedded in the wheel to exercise careful control of the temperature. Any wheel rim temperatures above 400C were considered dangerous for the magnesium alloy.

A slot in the wheel casting was made to allow the heating element terminals and the stator pad thermocouples to be accommodated during the test (Figs. 2 and 5).

A 9 in. fan was situated directly at right-angles to the brake and wheel rim to simulate the wind velocity encountered (Fig.6). It was estimated that about 60 mph wind velocity was achieved with this set-up.

Water was passed through the outer heavy magnesium brake casting which accommodated the eight pistons and cylinders using the channels through which the hydraulic fluid would normally flow. This was a precaution in the event of overheating.

A frame supported a movable table which carried the high pressure vessel used in the spray ignition and manifold/spray tests described earlier.

Hot Brake Spray Test

The heaters were turned on and the heat pack temperature adjusted to achieve a steady reading throughout the pack approximately 10C above the required test temperature. This involved certain manipulation of the heating elements. The heaters were then switched off to enable the temperature to drop to the required figure and eliminate hot spots. An optical pyrometer was also used as a further check on the heat pack temperature.

The straight-edged orifice attached to the pressure vessel was used throughout the tests. The fluid under test was at ambient temperature and was pressurized to 1000 psi under nitrogen.

The wheel was drawn into position, the fan switch ed on, and the spray allowed to fall on a position directly between the periphery of the brake and the inside of the wheel (see Fig.6). The spray distance was decreased rapidly from 2 ft to about 4 in., allowed to dwell for 2 sec, and then withdrawn. The wheel was pulled clear and the results recorded.

After each test, deposits were removed from the heat pack by means of a wire brush.

Hot Brake Stream Test

A funnel was fitted with rubber tubing, at the end of which was a 1/4 in. diameter copper tube, positioned by a clamp to direct fluid between the wheel and the brake periphery behind the first heater disk. Fifty ml of fluid was used for each test.

The object of this was to investigate the effect of straight injection of the fluid in the event of a hose or pipe being sheared off cleanly to cause a stream as opposed to a spray. It might also simulate a seal failure inside the brake.

The procedure was as before; the wheel was positioned, the fan turned on, and the fluid allowed to flow into the brake.

Results: Hot Brake Tests

Fluid	Brake Temp Deg C	High Pressure Spray Test	Stream Test
Phosphate Ester A & B	750	Immediate continuous burning	Immediate burning
	700	Immediate continuous burning	
	600	No burning	Immediate burning
	625	No burning	
	650	Immediate burning	
	575		Immediate burning
	550		No burning
Snuffer Fluid C	700	No burning	Immediate burning s/e
	650		No burning
	725	Burnt immediately s/e. Burnt again as spray withdrawn. No burning when wheel pulled out.	
	775	Burnt immediately s/e. Burnt again as spray withdrawn. No burning when wheel pulled out.	
Snuffer Fluid D	650	Immediate burning	
	625	Burnt, delay time 1 sec.	Immediate burning
	575	Burnt, delay time 2 to 3 sec.	
	550	No burning till fan switched on half-way through then continuous burning.	
	525	No burning	Immediate burning
	500		No burning
Chloro silicone	475	Burning after delay time 5 sec. Flaming droplets fell out on floor	Immediate burning at bottom of brake
	450	No burning	Burning
	425		No burning
Siloxane	500	Burnt after delay time of 5 sec.	Immediate burning
	475	No burning	Immediate burning
	450		No burning

Results: Continued   Hot Brake Tests

Fluid	Brake Temp Deg C	High Pressure Spray Test	Stream Test
MIL-H-5606	625	Burnt violently after delay time of 2 to 3 sec.	Immediate burning
	600	Immediate burning violently	Immediate burning
	575	No burning	Immediate burning
	550		Immediate burning
	525		No burning

Note: Burning continued after wheel had been pulled away from the brake unless otherwise stated.

DISCUSSION OF RESULTS

1. Hot Manifold Tests

These showed the clear superiority of snuffer fluid C and phosphate esters. The fact that snuffer fluid D and the chlorosilicone did not burn in the pan below the manifold showed some advantage from the point of view of flame propagation over the siloxane and mineral oil.

2. Spray Ignition Tests

a. Straight-edged orifice

This test showed up comparatively little difference between all the fluids in that 'flame thrower' effects were evidenced under most conditions with each fluid. At a certain critical distance, snuffer fluid C did not burn at all.

Phosphate esters and snuffer fluid D could be said to be slightly better than the other three types in that no burning was achieved after the removal of the flame at high or low pressure. The chlorosilicone, siloxane and mineral oil burnt only briefly. Results in the main were not affected by fluid pressure. In the case of a fluid flashing when sprayed at room temperature, the influence of higher temperatures usually was to produce a 'flame thrower' effect.

b. 80 deg Oil Burning Nozzle

This was far more stringent than the straight-edged nozzle test, and the results were more susceptible to fluid temperatures and the distance of the orifice from the flame. Snuffer C, however, showed a clear superiority over all other fluids in not burning at any orifice distance.

The fact that the siloxane and MIL-H-5606 burnt continuously at any orifice distance indicated an advantage of phosphate esters, snuffer fluid D and the chlorosilicone which burnt only at critical distances.

3. Hot Manifold/Spray Test

Neither the pressure under which the spray was operating nor the use of the 80 deg oil burning nozzle had any significant effect upon the results.

Snuffer fluid C again showed its superior properties in that even at 780C conflagration took place only with difficulty.

Phosphate esters gave good results in that no burning occurred up to 700C manifold temperature. Fluid temperature had no significant influence.

MIL-H-5606 showed up remarkably well on this test while the pressure was maintained in that no burning occurred up to 700C. However, it evidenced its flame propagating property in that immediately the spray was turned off, conflagration on the manifold and in the pan occurred. With snuffer fluid D burning was not propagated in the tray. The chlorosilicone and siloxane burnt readily, the latter explosively.

4. Shrouded Manifold Spray Test

In the main, ignition of the spray occurred at temperatures a little below those achieved with the ordinary manifold spray test.

There was insufficient of the siloxane fluid for a test to be conducted.

5. Vanguard Hot Brake Assembly Tests

1. Spray Test: Snuffer fluid C showed up outstandingly on this test. Though 700C was the maximum temperature at which no combustion occurred, even at 780C ignition took place only momentarily and was self extinguished.

Though phosphate esters showed themselves to be next best in order of fire resistance, the most singular feature of the test was that MIL-H-5606 behaved almost as well as phosphate esters. The maximum heat pack temperature at which MIL-H-5606 did not ignite was 575C while for phosphate esters the temperature was 625C. Snuffer fluid D at 525C, the siloxane at 475C, and the chlorosilicone at 450C gave the lowest maximum non-combustion temperatures.

2. Stream Test: Results indicated that this test was even more severe than the hot brake spray test. Maximum non-combustion temperatures were usually 25 to 50C below those achieved on the spray test.

Though snuffer fluid C caught fire at 700C in this test, the fact that the flame was self extingiushed fairly rapidly evidenced that no serious effects would result under such conditions.

Fluid Merit Rating

The fluids were assessed according to their fire resistance properties as judged from results of each type of test.

In the manifold tests the fluids were rated on the basis of:-

(a) their capacity to resist ignition

(b) the degree to which the fire was propagated, that is, whether or not ignition was extended from the manifold to the fluid collected in the tray below.

Test	Phosphate Ester		Snuffer Fluid		Chloro-silicone	Siloxane	MIL-H-5606
	A	B	C	D			
Closed Flash	2	a	3	4	1	5	6
Open Flash	4	6	3	5	1	2	7
Fire Point	5	6	1	4	2	3	7
SIT	1	1	4	6	3	4	7
Hot Manifold							
Ignition Resistance	2	2	1	4	6	7	4
Flame Propagation	1	1	1	1	1	6	6
Spray							
(a) S/edged nozzle	2	2	1	2	5	5	5
(b) 80 deg nozzle							
4ft to 2ft orifice distance	2	2	1	2	2	6	6
2ft to 3 in. orifice distance	2	2	1	2	5	5	5
Manifold/Spray							
Ignition Resistance	2	2	1	4	6	6	4
Flame Propagation	2	2	1	4	4	6	6
Shrouded manifold/Spray							
Ignition Resistance	2	2	1	5	6	-	4
Hot brake							
High pressure spray	2	2	1	5	7	6	4
Stream	2	2	1	5	7	6	4

a Unobtainable

SUMMARY

The simulated Vanguard hot brake test gave sufficiently similar results to those of the manifold spray test to warrant the latter technique as having significance regarding the specific aspect of fluid sprays impinging solely upon heated brakes. Greater similarity in results was achieved using an annular shroud round the manifold, injecting spray through an aperture in the shroud.

The maximum temperatures at which ignition did not occur were:-

	Hot Brake Temp Deg C	Manifold/ Spray Temp Deg C	Shrouded Manifold/ Spray Temp Deg C
Phosphate Esters A and B	625	700	650
MIL-H-5606	575	550	550
Snuffer Fluid C	700	750	700
Snuffer Fluid D	525	550	525
Siloxane	475	475	---
Chlorosilicone	450	475	450

The maximum non-combustion temperatures achieved on the manifold and the hot brake stream were quite different in contrast to the similarity between the manifold/spray and hot brake spray results.

The fact that the hot brake test showed comparatively little difference in the performance of MIL-H-5606, the phosphate esters, and snuffer fluid D leads to the conclusion that the brake test alone can not have the significance that was expected of it in rating fire resistance performance. However, there must be other factors that should be considered in wheel brake fires. It has become increasingly evident over many years that phosphate esters have had a good fire safety record in aircraft, whereas MIL-H-5606 has not been so successful.

One factor in wheel brake fires could be that a burst tire might not only fracture hoses causing fluid to spray over brakes but also effect ignition of the spray by friction sparks from direct wheel con tact with the runway. Spray ignition tests, especially using 80 deg oil burning nozzles, show up the disadvantage of MIL-H-5606 compared with phosphate esters which may be one cause for this apparent discrepancy. The manifold tests also clearly show MIL-H-5606 to disadvantage in its readiness to propogate flame.

Though snuffer fluid D gave a poorer result than MIL-H-5606 on the hot brake test, the spray ignition test under the more stringent condition of using the 80 deg oil burning nozzle showed that snuffer D had a greater degree of fire resistance than the chlorosilicone, siloxane, and MIL-H-5606. It was also equivalent to the phosphate esters and superior to MIL-H-5606, siloxane, and chlorosilicone in its resistance to propagation of burning.

The flash and fire point results gave no correlation with the hot brake test. Spontaneous ignition tests confirmed the good results of phosphate esters but bore little relationship to the others.

CONCLUSIONS

A simulated aircraft hot brake test revealed that only snuffer fluid C would withstand conflagration at heat pack temperatures of 700C when the fluid was sprayed onto the brake. This is the kind of temperature experienced under heavy braking conditions. Furthermore, reluctance to burn was evidenced by the fluid at a temperature of 775C - the maximum temperature obtainable in the heat pack.

Snuffer fluid C was the only fluid that did not catch fire under the stringent spray ignition conditions using an 80 deg oil burning nozzle.

A most remarkable aspect of the hot brake tests was the comparatively little difference shown in the fire resistance properties of phosphate esters,MIL-H-5606 mineral oil, and snuffer fluid D.

Although most tests showed that all the fluids examined were flammable under certain conditions, snuffer fluids and phosphate esters demonstrated their advantage over the other fluids in displaying a greater degree of resistance to spray ignition and flame propagation.

Of the techniques examined, a combined AMS 3150 C manifold/spray test (high pressure spray directed onto the hot manifold) gave perhaps the most significant results in rating the relative fire resistance properties of the five types of hydraulic fluids examined in an expected order. This test not only showed maximum non-combustion temperatures closely in line with those achieved on the hot brake test but also highlighted the deficiency of MIL-H-5606 and the siloxane in their capacity to propagate flames.

Even closer agreement with the hot brake maximum non-combustion temperatures could be achieved by using the shrouded manifold spray technique described in this paper.

In the main, bulk fluid temperature increase or a difference in pressure (200 to 1000 psi) under which the fluids were sprayed did not significantly affect results.

The use of the straight-edged nozzle in the AMS 3150 C spray ignition test did not usefully differentiate between the fire resistance properties of the fluids. On the other hand, significant differences were achieved with the 80 deg oil burning nozzle.

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REFERENCES

1. Report on Origin of Fires. R.L. Paullin to D.H. Moreton. March 16, 1959.
2. I.P. Standards for Petroleum and its Products. 1961. Page 285.
3. I.P. Standards for Petroleum and its Products. 1961. Page 272.
4. Spontaneous Ignition of Liquid Fuels, by Mullins. Page 14.
5. Aeronautical Material Specification. A.M.S.3150 C Jan. 15, 1960.
6. P.I. Brittain. Hydraulic Fluids - A Comparison of Inflammability. Nov. 30, 1960.
7. Communication from Dunlop Aviation Division, Coventry, England.

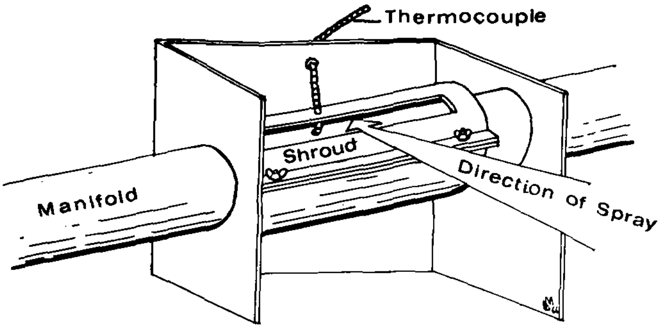

Fig. 1. Shrouded Manifold Spray Test.

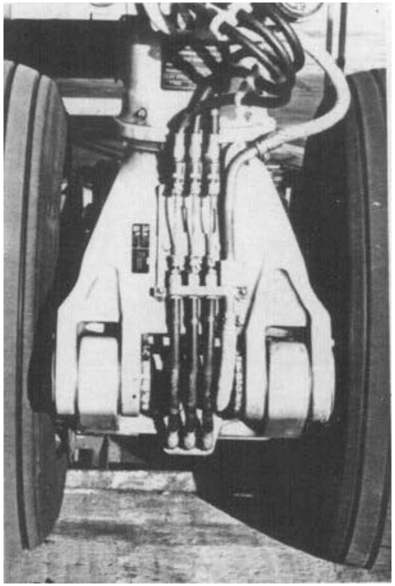

Fig.2. Hawker Siddeley,Trident.
Front view of wheel and brake.

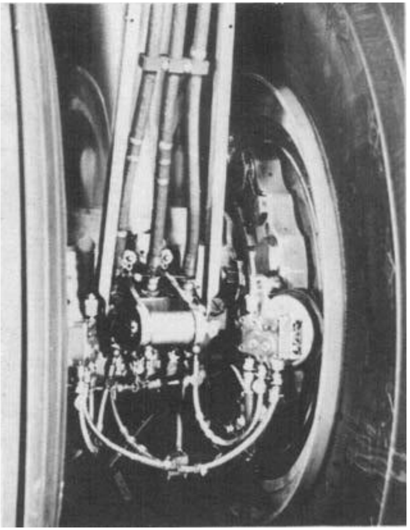

Fig.3. Vickers Vanguard.
Front view of wheel and brake.

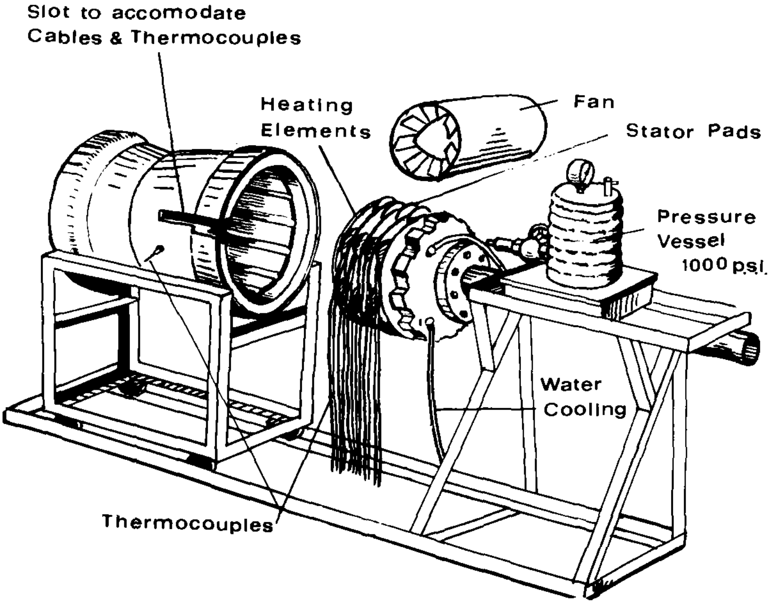

Fig. 4. Vanguard Wheel and Brake Assembly.

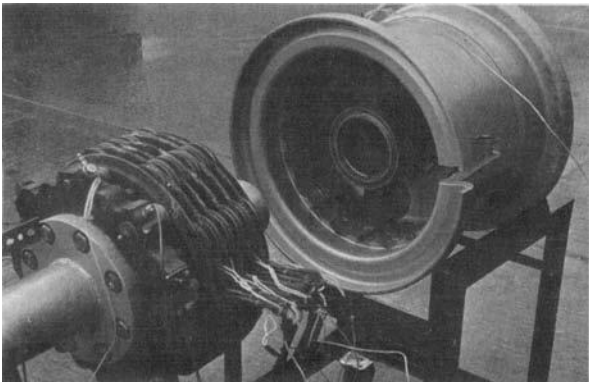

Fig.5. Vanguard Wheel and Brake separated. Shows slot in wheel to accommodate brake heating elements and thermocouples.

Fig.6. MIL-H-5606 burning at 600C brake temperature.