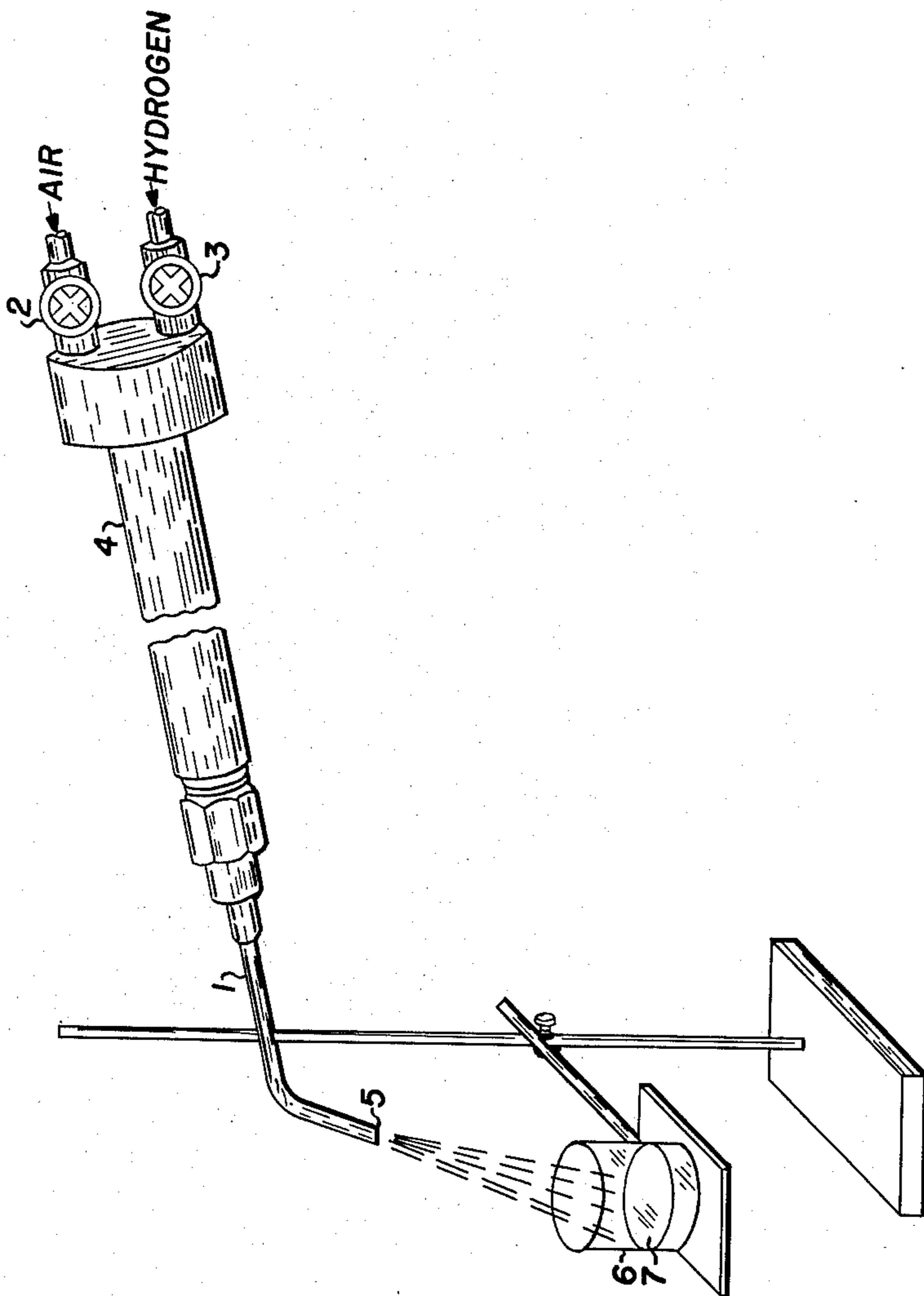


Sept. 4, 1956

A. H. POPKIN
COMBUSTION TEST
Filed May 1, 1953

2,761,766



Alexander H. Popkin Inventor

By *h. f. Mavt* Attorney

1

2,761,766

COMBUSTION TEST

Alexander H. Popkin, Newark, N. J., assignor to Esso Research and Engineering Company, a corporation of Delaware

Application May 1, 1953, Serial No. 352,373

9 Claims. (Cl. 23—230)

The present invention relates to a new and improved method of testing the combustion characteristics of combustible materials. More particularly, the invention is concerned with an improved method for testing the tendency of such materials toward forming combustion chamber deposits when used in the combustion chambers of internal combustion engines.

In brief compass, the invention pertains to a method for testing the deposit-forming tendencies of motor fuels, lubricants, as well as additives for fuels and lubricants by subjecting these materials to combustion under a clean-burning flame, particularly a hydrogen flame in a container having smooth non-absorbing inner surfaces, to form a dry combustion residue on said surfaces. The quantity of total deposit indicates certain properties of the material tested. Thereafter, that portion of the deposit which adheres loosely to the surface may be removed by light mechanical friction and the remaining firmly adhering deposit may be determined. By reference to correlated data it is possible to rate the test material as to its combustion deposit-forming tendencies on the basis of the amount of total deposit and/or the amount of the loosely and/or firmly adhering portions thereof.

The problem of combustion chamber deposits is of urgent concern to the automotive and oil industries. These deposits increase the octane requirements of gasoline engines by about 10 octane units in 3,000 to 5,000 miles. They may also cause preignition, a phenomenon which may lead to knock, and loss of power. As it is becoming increasingly difficult to provide gasoline having the octane quality required by the new high compression engines, it is important to eliminate or reduce the effect of these deposits.

Extensive studies have demonstrated that the formation of combustion chamber deposits may be caused by the fuel, the lubricating oil, as well as by additives used in either of these. A reliable method for predicting the tendency of fuels, lubricants and additives to form such deposits is, therefore, urgently needed. Past field and laboratory studies and the correlation of their results have shown that none of the known laboratory engine tests is suitable for this purpose.

The present invention provides this needed test in an extremely simple and most reliable manner as will appear from the subsequent description read with reference to the accompanying drawing, the single figure of which is a perspective view of equipment adapted to carry out a preferred embodiment of the invention.

In accordance with the present invention it has been found that the combustion residue remaining when burning an oil or fuel sample in a hydrogen flame under controlled conditions may serve accurately to predict the tendency of the sample material toward forming combustion chamber deposits in internal combustion engines. In carrying out the test of the invention, the sample to be tested is placed in an open vessel having smooth non-absorptive inner surfaces, such as a glass beaker, porcelain crucible, heat-resistant metal beaker or crucible, etc. A clean burning hydrogen flame is then directed into the opening of the vessel. The position of the burner tip and the flow rates of hydrogen and oxygen should be so controlled that the flame is directed toward the interior

2

of the vessel. In most cases conditions are so controlled that the flame enters the vessel over a distance of at least about 0.5–2 inches.

Usually within less than 2 minutes of exposure to the hydrogen flame the sample begins to burn. The hydrogen flame is continued until the sample is completely burned and only a dry residue remains. Thereafter, the hydrogen flame is discontinued and the vessel is allowed to cool.

The amount of total combustion residue is now determined by weighing the vessel and deducting from its weight the weight of the empty vessel. This total deposit has been found to correlate satisfactorily with the octane requirement increase determined in actual field tests for a large number of motor fuels.

Particularly in the case of testing lubricants, after determination of the total deposit, the interior of the vessel is carefully wiped with a soft material, such as cloth or soft wiping paper, until no further removal of deposits from the vessel is obtained. Somewhat more effort is required in wiping carbonaceous deposits from the sides of the vessel in the case of lubricant tests than in the case of fuel tests. Such wiping removes carbon deposits. However, varnish which adheres tenaciously to the smooth vessel wall is not removed by this treatment. After wiping, and preferably after a further short standing period for removing static electricity, the vessel is weighed again. The increase in weight of the wiped vessel over the emptied vessel is the amount of varnish formed by the sample and the difference between the total deposit and varnish corresponds to the amount of carbon and a substantial proportion of the ash. The varnish deposits so obtained correlate satisfactorily with the octane requirement increase obtained in actual field tests for a large number of lubricants. The loosely adhering carbon deposit may be readily analyzed to determine the character and proportion of the ash constituents of the combustion chamber deposit.

In accordance with the invention, hydrogen is used in preference to other secondary fuels to obtain a clean flame which itself forms no carbonaceous combustion products. Certain other fuels, such as methane, ethane, propane, etc., may be used provided combustion conditions are such as will provide a clean burning flame. The feed ratio between hydrogen and air to the flame of the invention may vary within wide limits. However, some materials, particularly lubricants, may require a hotter flame than others, in which case relatively high air ratios may be employed.

While any burner type may be used provided hydrogen may be burned under carefully controlled burning conditions, a conventional acetylene blow torch type of burner is preferred. This type of burner affords the greatest ease of control of combustion mixture and flame length. The use of equipment of this type for the purposes of the invention is diagrammatically illustrated in the drawing.

Referring now to the drawing, the numeral 1 designates a conventional blow torch provided with air control valve 2, hydrogen control valve 3 and mixing chamber 4. The torch tip 5 preferably has an opening of about 0.125 in. diameter and is set at an angle of about 10–20° as indicated in the drawing.

At a distance of about 6–7 inches below the tip of the torch, there is placed the rim of a glass beaker 6 or other vessel with smooth non-absorptive inner surfaces, containing the Sample 7 to be tested. As indicated in the drawing, the flame enters the top of the beaker without touching its walls and ending at a distance above the level of the sample.

The application of the equipment illustrated in the drawing for the purposes of the invention will now be explained with the aid of specific examples.

EXAMPLE I

The following is a detailed procedure for the burning of a sample of lubricant in accordance with the invention. A 250 cc. beaker is weighed on an analytical balance to the fourth decimal place. Into the beaker are then added 5.000 grams (5 grams) of lubricant using the same analytical balance. The beaker is then placed under the burning hydrogen torch in the position indicated in the drawing. The tip of the torch is 6½ inches from the top of the beaker and set at an angle as shown. This position and the flow rates of hydrogen (0.166 cu. ft./min.) and primary air (0.116 cu. ft. per min.) are drawn to give a flame length of about 7½ inches, or a flame extending about one inch below the top of the beaker. The flame, moreover, is about ¼ inch from the rear top of the beaker. The flame should not touch the rear portion of the beaker or extend to the bottom of the beaker.

The beaker and contents are placed under the flame in the designated position. Within one minute, vapors and/or slight burning will be obtained. At this time, spattering may also start due to the presence of condensed water formed from the hydrogen-air combustion in the hot oil. It is advisable, if spattering is noted, to lift the beaker slightly from the support surface and to gently stir the lubricant sample by a slow rotary motion of the beaker until a continuous flame is obtained. This may require one-half to one minute of the rotary motion. When the sample begins to burn steadily, the beaker is replaced under the flame and burning continued until a flame is no longer obtained. The beaker is then slowly turned 180 degrees during about 30 seconds to burn the high boiling residual liquid. At this stage, the beaker is removed from flame and the condition at the bottom of the beaker observed by tilting the beaker. If liquid or vapor are noted, the beaker is placed under the flame for an additional 30 seconds and observation repeated. If no liquid is noted, and vapors are not evident in the beaker, the sample is set aside to cool. The total burning time may vary from 5-15 minutes, oils containing heavy ends (i. e., bright stock) requiring longer burning times.

Following the burning, the beaker is allowed to stand at room temperature for at least an hour before weighing. The increase in beaker weight is recorded as the total deposit. The carbon from the sides of the beaker is then removed with several wipings of a suitable soft laboratory wiping paper (e. g., large Kim-Wipe papers). Wiping is maintained until no further removal of deposits from the sides of the beaker is obtained. After wiping, the beaker is allowed to remain at room temperature before weighing again. The increase in weight of the beaker after the wiping operation is recorded as the amount of varnish, and by difference from the total deposit, the amount of carbon is obtained.

As pointed out above, the varnish deposits obtained in this laboratory combustion test correlate satisfactorily with the actual octane requirement increase obtained in engine field tests with eight lubricants evaluated with the same fuel. Data illustrating the correlation are tabulated below in Table I.

Table I

Lubricant	Milligrams Varnish Deposit in H ₂ -Combustion Test	Equilibrium Octane Requirement in Engine Field Test
1.....	2	79
2.....	3.5	78
3.....	4	78
4.....	13	83
5 (Containing Ash-Forming Additives).....	16	83
6 (Containing Ash-Forming Additives).....	17	83
7.....	37	90

The above data show that a substantially linear correlation exists between the varnish deposits as determined by the test of the invention and the equilibrium octane requirement determined in actual engine field tests.

The excellent correlation obtained with lubricants No. 5 and 6 indicates that this test is also satisfactory for evaluating lubricants containing ash-forming additives, a very important consideration in additive screening.

EXAMPLE II

The following is a detailed procedure of the burning of a fuel in the test of the invention. A 400 cc. beaker is weighed on an analytical balance to the fourth decimal place. The beaker is then placed on an accurate laboratory balance (i. e., torsion balance of accuracy better than 0.1 gram) and 200 grams of fuel are weighed into the beaker. The beaker is then placed under the burning hydrogen torch, which is now operated exclusively with secondary air (0.59 cu. ft./min. of H₂), in such fashion that the tip of the torch is 3½ inches from the top of the beaker. The torch tip is at a slight angle (about 15°) above the beaker. The time is noted when the beaker is placed under the hydrogen flame, when the yellow luminous flame of the burning gasoline is gone (approximately 20-25 minutes of burning), and when the beaker is down to dryness (about 1-2 minutes later). Care should be exercised in uniformly judging the time of dryness of the beaker, at which point the beaker is finally removed from the hydrogen flame. This is done by removing the beaker from the hydrogen flame about 15 seconds after disappearance of the yellow luminous flame and noting the presence of liquid or vapor in the beaker. If no liquid or vapor is present, the beaker is judged dry and set aside for cooling and weighing. If liquid or vapor are present, the beaker is placed back into flame for an additional 30 to 60 seconds of burning depending on the amount of liquid present. This operation is repeated until dryness is obtained.

The contents of the beaker at this stage are composed of carbonaceous and lead deposits on the sides of the beaker and varnish deposits (generally reddish-brown) at the bottom. The beaker should be allowed to cool at room temperature for at least one hour after removal from the flame before weighing on the analytical balance. The increase in weight is the total deposit due to fuel.

Satisfactory reproducibility has been obtained for fuels in this test. This is illustrated by repeated determinations conducted on various fuels as shown in Table II below.

Table II

Run No.	Fuel	200 Grams in Hydrogen Combustion Test, Milligrams Total Deposit
1.....	A	58
2.....		62
3.....		65
4.....		54
5.....		71
6.....		64
7.....		69
8.....	B	15
9.....		12
10.....		10
11.....	C	61
12.....		50
13.....		64

The total deposits obtained in the hydrogen combustion test with clear fuels correlates with the equilibrium octane requirement obtained with these fuels in actual engine field tests as shown in Table III.

5
Table III

Fuel	Milligrams Total Deposits in H ₂ -Combustion Test	Equilibrium Octane Requirement in Engine Field Test
D.....	13	78
E.....	13	77
F.....	38	80
G.....	64	82
H.....	88	88

All types of fuels and lubricants including jet fuels, heating oils, diesel fuels, residual fuel oils, etc., may be tested in accordance with the invention for their cleanliness properties upon burning. These fuels are all normally liquid. If it is desired to evaluate fuel or lubricant additives, the same may be incorporated into fuels or lubricants of known deposit-forming characteristics and in this form subjected to the test described above.

This test is also capable of detecting changes in deposit-forming properties of fuels during storage. For this purpose, the contents of the beaker, after determination of the total deposit, are wiped with suitable soft laboratory wiping paper (e. g., large Kim-Wipe papers), to remove the lightly adhering deposits. The varnish deposits are firmly adhered to the glass and are not removed by the wiping. Following the wiping, the beaker is permitted to stand at room temperature for at least 30 minutes to leak-off static charge, and reweighed. This gives the weight of varnish formed in the burning process, and by difference from the total deposit, the amount of carbonaceous deposit (plus lead, if present) is obtained. Newly prepared leaded motor fuel containing unsaturates or such fuel carefully stored under nitrogen has varnish deposits in the order of 3.5 to 5 mgs. The varnish deposits gradually increase during storage in air and handling in air, as shown by the data of Table IV below.

Table IV

CHANGE OF DEPOSIT-FORMING PROPERTIES OF MOTOR FUEL ON STORAGE AND HANDLING IN AIR

Sample Description	200 Grams of Fuel in Hydrogen Combustion Test Milligrams Deposit	
	Total	Varnish
Newly Taken From Drum.....	67	5
Do.....	59	4
Above After 20 Days' Storage on Desk in Clear Bottle.....	64	11

In the foregoing examples the bulk of the sample to be tested was placed in the beaker and subjected as such to the hydrogen flame. If desired, the sample may be supplied continuously or in small increments to the beaker exposed to the hydrogen flame. For example, the sample may be added dropwise from a burette, separatory funnel, or similar overhead reservoir while burning is going on.

It will be understood by those skilled in the art that the invention is not limited to the exact figures given above for flame length, fuel mixture, flame distances, etc. These figures may be different provided they are maintained constant for all tests of fuels or lubricants.

What is claimed is:

1. The process of testing the utility of combustible materials which comprises subjecting a material selected from the group of lubricating oils and normally liquid fuels to combustion under a clean-burning flame in an

6

open vessel having smooth non-absorbing inner surfaces until said material is burned to a dry combustion residue remaining on said surfaces, subjecting said surfaces to light mechanical friction to remove all loosely adhering deposits leaving a firmly adhering deposit not removable by said light mechanical friction, and determining the weight of said firmly adhering deposit, said plane being directed into the opening of said vessel.

2. The process of claim 1 in which said flame is a hydrogen flame.

3. The process of testing the tendency of lubricating oils to increase the octane requirement of internal combustion engines which comprises subjecting a predetermined amount of said oil to combustion under a hydrogen flame in an open vessel having smooth non-absorbing inner surfaces until said amount of oil is burned to a dry combustion residue remaining on said surfaces, subjecting said surface to light mechanical friction to remove all loosely adhering deposits leaving a firmly adhering deposit not removable by said light mechanical friction and determining the weight of said firmly adhering deposit, said flame being directed into the opening of said vessel.

4. The process of claim 3 in which said oil contains ash-forming additives.

5. The process of claim 3 in which said flame is produced by burning a mixture of hydrogen and primary air.

6. The process of testing the deposit-forming properties of normally liquid fuels which comprises subjecting a predetermined amount of said fuels to combustion under a hydrogen flame in an open vessel having smooth non-absorbing inner surfaces until said amount of fuel is burned to a dry combustion residue remaining on said surfaces, subjecting said surfaces to light mechanical friction to remove loosely adhering deposits leaving a firmly adhering deposit not removable by said friction and determining the weight of said firmly adhering deposit, said flame being directed into the opening of said vessel.

7. The process of testing the utility of a material selected from the group of lubricating oils and normally liquid fuels which comprises subjecting said material to combustion under a clean-burning flame in an open vessel until said material is burned to a dry combustion residue remaining on the walls of said vessel and consisting of a loosely adhering and a firmly adhering portion, removing said loosely adhering portion and determining the weight of said firmly adhering portion, said flame being directed into the opening of said vessel.

8. The process of testing the utility of a material selected from the group of lubricating oils and normally liquid fuels which comprises subjecting said material to combustion under a clean-burning flame in an open vessel until said material is burned to a dry combustion residue remaining on the walls of said vessel and consisting of a loosely adhering and a firmly adhering portion, removing said loosely adhering portion and determining the weight of said loosely adhering portion, said flame being directed into the opening of said vessel.

9. The process of testing the utility of a lubricating oil which comprises feeding a predetermined amount of said lubricating oil over an extended period of time to an open vessel, subjecting the material entering said vessel to combustion under a hydrogen flame to burn said oil as it enters said vessel to a dry combustion residue remaining in said vessel, said flame being directed into the opening of said vessel, removing said hydrogen flame after said amount has been burned, subjecting the inner surface of said vessel to light mechanical friction to remove loosely adhering deposits while leaving a firmly adhering deposit and determining the weight of said firmly adhering deposit.

(References on following page)

References Cited in the file of this patent

Fuel Oil Manual by Paul F. Schmidt; 1951, pages 68-71, published by the Industrial Press, 148 Lafayette St., New York 13, N. Y.

ASTM Standards on Petroleum Products and Lubri-
cants, November 1950, pages 65-66.

Hamor: "Examination of Petroleum," McGraw-Hill Book Co. (1920), pages 104-106.

Maillard et al.: Annales Combustible Liquids, vol. 11, pages 1117-26 (1936).