

July 24, 1934.

H. G. NEVITT

1,967,424

ANALYTICAL APPARATUS FOR TESTING ASPHALTS

Filed Oct. 17, 1932

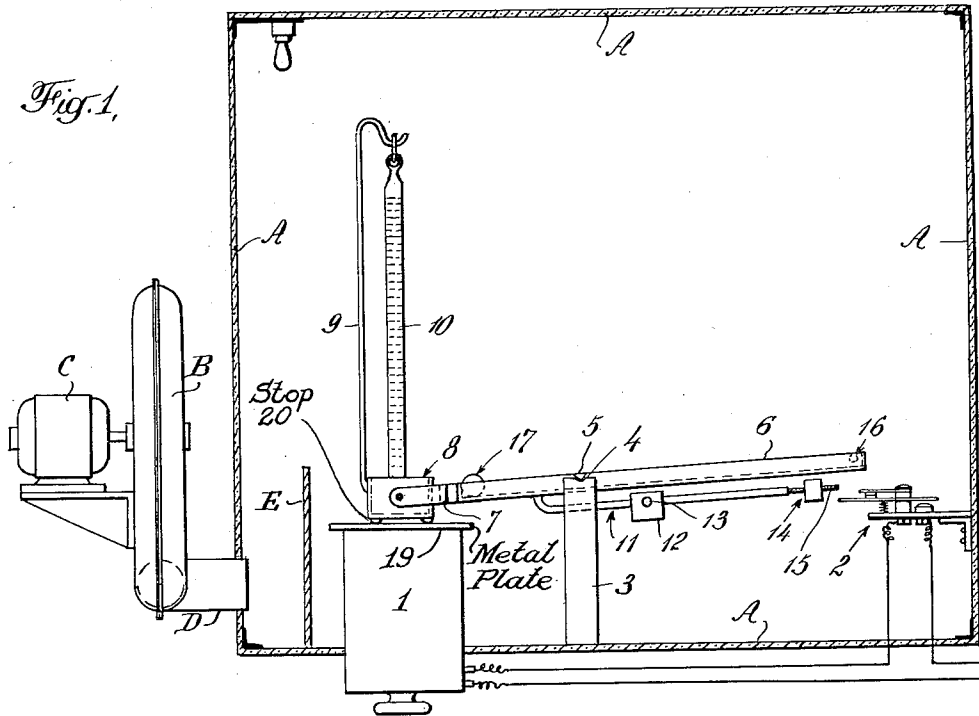


Fig. 2,

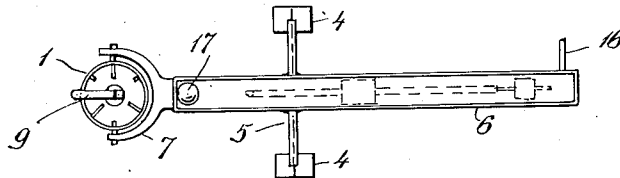
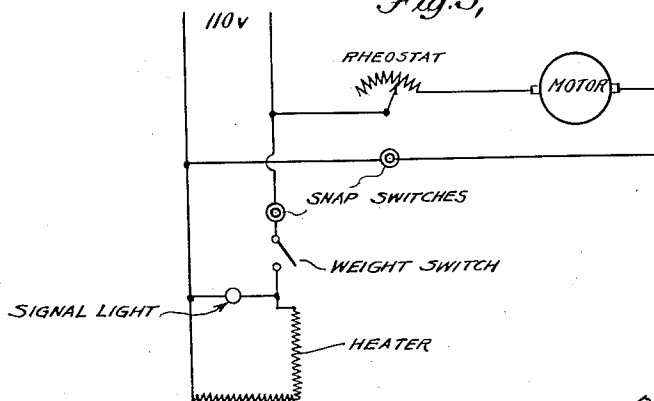


Fig. 3,



HENRY G. NEVITT
INVENTOR

BY
Dallas R. Lamont
ATTORNEY

UNITED STATES PATENT OFFICE

1,967,424

ANALYTICAL APPARATUS FOR TESTING
ASPHALTS

Henry G. Nevitt, Kansas City, Mo., assignor to
White Eagle Oil Corporation, Kansas City, Mo.,
a corporation of Delaware

Application October 17, 1932, Serial No. 638,161

2 Claims. (Cl. 34-19)

In the analytic examination of asphaltic pitches, binders, and fluxes, of asphaltic road oils, and of various materials of varying asphaltic content it is customary to classify the oils by determining the percentage of asphaltic content of specified penetration. To determine the percentage of this content it is necessary that the oils be reduced by evaporation to a residue of the desired consistency and the weight relation which this bears to the original sample be determined. This invention has to do with an apparatus for performing the reduction by evaporation above outlined. It has more specifically to do with provisions for handling the reduction in an automatic manner.

Asphalts are classified by their weight-percentage content of asphaltic residue of a specified consistency. This specified consistency is usually spoken of as, for example, "60 penetration asphalt" and is determined by subjecting a sample of the residue at a certain temperature to test under a needle of specified dimensions which is forced into the residue by a specified weight for a specified length of time. Other tests indicative of the quality of the original material are made upon the residue. One such test is the ductility test wherein a small formed sample of the residue at a specified temperature is pulled apart at a specified rate and the elongation before final breakage noted. Another test usually made upon the residue from such evaporation is that of determining the amount of free carbon or other material insoluble in such solvents as carbon tetrachloride or carbon disulphide. The specified amount of insoluble residue in such tests is usually quite low. It may be seen from the nature of these tests that the manner in which the residue is produced by evaporation from the original sample can be of much influence upon the final outcome of the test. For instance, overheating may cause cracking of a portion of the residue exposed to such overheating, resulting in a destruction of valuable residual material with a corresponding thinning of the remainder by the lighter products of cracking, resulting in a loss of penetration and ductility and also there may be from this cracking a deposit of carbon insoluble in the reagent used in testing.

Another variable in the manner of producing the residue which may affect the test is that of oxidation. The exposure of the heated oil undergoing reduction to the atmosphere results in the oxidation of a portion of it to materials of an asphaltic nature. In other words it creates by the very method of analysis some amount of a

material similar to that which the analysis is calculated to detect. The amount of oxidized material so made is small. It is to a great extent unavoidable, or at least economically unavoidable. It matters therefore, in order that independent workers may check each other, more that the oxidization shall be uniform from sample to sample and from determination to determination than that it be avoided entirely.

Another variable which results in erroneous tests is the method of heat application, as a rapid rate of reduction will give results differing from a slower rate of reduction.

When the amount of the sample is small and the desired amount of residue unknown it is frequently impossible to reduce more than one sample. In such cases it becomes necessary to remove the sample from the reduction apparatus several times and determine the consistency of the residue. In case it is still too thin it must be again heated and reduced. This cooling and reheating may seriously affect the tests on the residue. Even when the desired amount of reduction is known it often becomes necessary to remove, cool, weigh, and reheat the sample several times to determine the exact point of stoppage unless this point is reached the first time by good fortune.

Present methods of performing this reduction vary. The most commonly accepted one is probably that set forth by the American Society for Testing Materials in their 1928 "Book of Tentative Standards" on pages 528-532 and designated as Standard D 243-28 T. A short description of this apparatus to save reference is as follows:

A ring stand is used to support the setup which consists of a hemispherical dish for containing the sample to be reduced, placed in an air bath which is formed of sheet steel in the shape of a truncated cone. The air bath is heated by an ordinary Bunsen burner. Suspended above the sample, with its bulb therein, is a thermometer of certain specified characteristics. This method has several practical objections. The most annoying of these is probably that the container specified is quite unhandy for use in the regular penetration test. More vital defects are that due to the construction, the source of heat, if it is a gas flame as is specified, can come in very direct contact with the sample container resulting in local overheating with its attendant irregular results in the determination. A second objection is that the sample being heated is exposed to the air in varying area and depth, allowing irregularities in oxidation. Other methods of per-

forming the same reduction contemplate the use of sand baths or oil baths for the heating medium. In a sand bath a considerable temperature difference exists between the temperature to which the sample is exposed near the top of the bath and the temperature to which it is exposed near the bottom of the bath with corresponding irregularities in the rate of reduction. Further objection to sand baths lies in the fact that the sample container is in contact with a material of high specific heat and in case of poor control it is quite easy for it to become seriously overheated. In the case of oil baths the material used in the bath must necessarily be an oil of high flash point. Such oils are also of sufficiently high specific heat to give trouble with the control of the rate of heat. As a further defect they are quite tenacious and can only with difficulty be removed from the outside of the sample container when it is removed for weighing. As a further difficulty they have a creeping tendency and are apt to creep up and over the edge of the container or contaminate the sample by condensation within the container of vapors from the oil bath.

It will be noted from the above discussions that in addition to the various analytical defects of the various methods no one of them has any method by which the progress of the reduction may be observed other than visually. Due to the expansion of oils upon heating a visual method is extremely inaccurate. It has therefore been the object of this invention to provide an apparatus for use in the reduction of asphaltic materials to residues of specified consistency which will provide for a uniform method of treatment in order that variations from one determination to another may be minimized; to also provide a method in which the reduction may be automatically stopped when a certain portion of the sample has been evaporated, to provide an apparatus wherein the rate of heating may be so controlled as to be uniform throughout the determination, and to provide a method whereby this analytical determination may be carried out with a minimum of attention from the analyst.

The principle upon which this invention is based is that of placing the sample on one end of a balance arm which is so adjusted that when the weight of the sample has been reduced a specified amount the balance may swing in the opposite direction which movement will result in removing the sample from the zone of heat and which motion may be taken advantage of to cut off the supply of heat at the moment of movement.

To convert an apparatus working on this basic principle into one of commercial characteristics requires a few accessory details. These details are explained in the description of the apparatus which follows:

In order that a commercial workable embodiment of the apparatus may be shown and that its details may be thoroughly made clear I have attached a drawing which is hereby made a part of this specification. In this drawing Figure 1 shows a vertical elevation of the apparatus in operating assembly, partially in section. Figure 2 shows a plan view of a portion of the apparatus. Like numerals are used to designate the same part throughout both figures. Figure 3 is a wiring diagram. In Figure 1, AAAA designates a box or enclosure wherein the apparatus is set up. This enclosure may be made of sheet steel, Transite board or any similar material, its func-

tion being solely to set off the area wherein the determination is made from outside atmospheric disturbances. In order that the atmosphere within this enclosure may be changed to remove fumes and that it may be circulated to insure a constant temperature throughout the enclosure there is provided attached to the enclosure a fan B driven by a motor C or by other suitable means and communicating with the enclosure through a duct D. In order that the current of air from the fan may not play directly upon the apparatus for the determination a baffle E of the same material as the enclosure A is provided. The enclosure with its fan, etc., may be of a size only large enough to contain one reduction apparatus or it may contain a plurality of such machines. Proper methods of releasing air from the enclosure to outside atmosphere or to a stack 18 should be supplied. The apparatus proper consists of an electrical heater 1, the balance mechanism and an electrical switch 2. The heater as shown is of a type in wide use in analytical work in the oil industry and consists of a heating coil which is exposed to the material being heated through an orifice in the top plate of the heater and a rheostat for controlling the amount of heat, which rheostat is adjusted by a knob at the bottom of the heater. This heater may be supported at the proper height in any convenient manner or there may be substituted for it any other manner of heating such as a gas heater with proper air bath or sand bath although the preferred apparatus of my invention contemplates the use of an electric heater to avoid the difficulties inherent in other methods of heating. A short distance removed from the heater is a double support 3 of any convenient form presenting two upper supporting surfaces 4 of exactly the same elevation upon which rests a knife edge 5. Attached to this knife edge is a balance arm 6 arranged in such a manner that one of the extremities is above the orifice of the heating device. This end of the balance arm is formed as a stirrup 7 which may be used to properly support a sample cup of specified dimensions 8. This sample cup is so held that it rests above a metal cover plate 19 and is adjusted to allow about one-quarter inch air space between plate and cup over the orifice of the heater when this end of the beam is depressed. This adjustment is made by means of stop 20. Extending upwardly from the stirrup 7 is a wire 9 which may be used to support a thermometer 10 of the proper type for use in the determination. Running under the balance beam and parallel to and attached thereto is a support arm 11. Slidably mounted upon this support arm is a movable weight 12 with a thumb screw 13 by which its position may be fixed. The end of support arm 11 terminates in a threaded portion 14 upon which end is mounted a weight 15. Adjustment of the positions of weights 12 and 15 will accomplish balancing of the sample with a great degree of accuracy. Attached to the end of beam 6 furthest removed from the heater is an arm 16 and mounted nearby within the enclosure is a pressure switch 2 of such a nature that when its actuating member is depressed by arm 16 the current flowing through the switch is cut off. It may be seen from this description that a weighted sample can be placed in the cup 8 and the weights 12 and 15 so adjusted that when a predetermined portion of the sample has been evaporated the end of the beam most remote from the heater will become heavier and thereby depressed, thus removing the cup 8

from the action of the heat and opening switch 2 through the action of arm 16. To render this action more definite and rapid once the system becomes unbalanced I have constructed beam 6 of a small piece of angle iron open upward, though any other form of channeled beam might be used. In the channel thus formed I have provided a steel ball 17 so mounted that it can roll back and forth, and stops at either end of the channel. As long as the weight relations are such that the sample is heavier the ball will remain at the position shown at the sample end of the beam. As soon as the weights are altered and the sample rises the ball will immediately run to the other end of the beam giving a great discrepancy in weights and thereby effecting a rapidly and definitely actuated functioning of the various portions of the equipment.

Figure 3 is a wiring diagram showing how the heater and motor may be wired in parallel from a source of power with snap switches to control each circuit and the weight switch for controlling the operation of the heater. I have also found it convenient when using a plurality of these machines to install in some convenient place a signal light in the circuit of each heater in order that the conditions of operation may be signaled to an analyst working some distance away.

In the use of this equipment the following details or procedure would be followed. A container would be tared and the sample of oil weighed into it and the combined weight of sample and container noted. From previous experience or by estimate the necessary reduction of the sample would be calculated and a similar container placed in the weighing balance and filled with fine sand, shot, or other material until its weight is that which the first container would have when its sample is reduced the desired amount. This second container is then placed in the stirrup 7 of balance arm 6, the ball 17 is brought to the stirrup end of the balance arm 6 and weights 12 and 15 are adjusted so that the apparatus is just in equilibrium. Then the second container is removed from the stirrup 7 and the first container with the sample is placed therein, the ventilating system is started and properly adjusted, if it is not already running, and the heater 1 is turned on by means of its snap switch and adjusted after working off any foaming which may occur due to water in sample to that point which is known from previous experience to maintain the sample at a temperature of 480-500° F. The apparatus can then be left and receive no further attention. When the sample in the container 8 has been reduced the desired percentage the beam 6, actuated by weights 12 and 15 and assisted after initiation of movement by rolling ball 17 will act to remove the sample from the region of heat to a region wherein it may cool, cut off the current to the source of heat, and extinguish the signal light to notify the analyst that the reduction has been completed. After a suitable time for cooling the sample may then be removed to water bath for temperature equalization and its consistency and other properties noted. In accordance with the usual methods several samples may be run to varying percentage of reduction, the consistency of each reduced sample noted and the percentage of reduction required to gain a given consistency graphically interpolated.

As an example of the operation of this machine in the examination of an asphaltic material which is to be analyzed for its content of asphalt of a specified consistency I wish to present the

following: It is known that the asphalt contains approximately 70% by weight of this residue of specified consistency so three samples are weighed out and one sample is reduced to say 65% residue, one to 70% and one to 75% residue after the method outlined in the general matter of this specification. The consistency of each of these residues by the standard penetration test is noted and a curve is drawn plotting consistency against per cent residue. From this curve the per cent residue having the standard consistency may be determined without actually evaporating to that exact consistency. This method is in nowise different from that at present employed except that the use of this apparatus enables a greater standardization of the conditions of heat application in degree and length of time thereby bringing about a greater consistency of results and a greater exactitude of analytical determination.

It may thus be seen that I have provided an apparatus capable of controlling all of the variables in this particular analytical determination. I have further provided an apparatus capable of conducting the reduction to a specified predetermined extent and then automatically stopping itself and removing the sample from the region of alteration. As a result of these provisions I have not only increased the possibility of accuracy in the determination but I have rendered possible great savings in time and attention. The amount and extent of change will be readily apparent to any one familiar with the analytical procedure at present used.

It is obvious that numerous other mechanical embodiments of this invention might be made without departing from the spirit of the invention and I therefore wish to claim this invention in its widest equivalents except as limited within the following claims:

I claim:

1. In an analytical apparatus for the reduction of asphaltic oil samples to a predetermined percentage of residue an enclosure, pressure ventilating means for such enclosure, and within such enclosure an evaporating device, comprising a source of heat, a support, a balance arm mounted upon said support adjacent said heat source, one end of said arm positioned above said source of heat, this end of said arm being formed to provide a frame wherein a sample cup may be positioned above the source of heat, the weight of the unreduced sample and cup acting to compel approach of the sample to the heat source, stop means whereby the sample cup approach may be limited to avoid contact with the heat source, a weight upon the end of said arm remote from the heat source, said weight being adjustable in position to permit it to overbalance the sample when a predetermined amount has been evaporated therefrom and thereby to remove said sample from said source of heat upon evaporation of said predetermined amount, a channel or trough partially coextensive with said balance arm, a weight free to move therein substantially parallel to the length of said arm, inactive when sample is being heated, acting to accelerate movement of the arm when sample is overbalanced, an electric circuit controlling said source of heat, a switch in said circuit, and means whereby the sample-raising motion of the arm communicated to said switch causes shut off of said heat source.

2. An analytical apparatus for the reduction of an asphaltic oil sample to a predetermined percentage of residue comprising an enclosure, ventilating means to insure uniform circulation

of air therethrough, a means of heat supply, a container for sample and means to maintain such sample container in the heating region but free from contact with the source of heat until the predetermined reduction has been obtained and thereafter to remove the sample to a region away from the source of heat wherein it is subjected to ventilating currents thereby to accurately and rapidly check the reduction, said means by the action of removal acting to shut off the source of heat.

HENRY G. NEVITT.

10	85
15	90
20	95
25	100
30	105
35	110
40	115
45	120
50	125
55	130
60	135
65	140
70	145
75	