

samples and consequently having to multiply the amount of alkali used by 1.63, it is advisable to use a 16.3 cc. pipette, in which case, the reading on the burette denotes directly the percentage of casein.

A very convenient and simple form of acidimeter has been lately put on the market by the author which may be used for both the "acid test" and the "casein test."

A series of comparative tests, using both the new method and the centrifugal method, has been carried on at the Eastern Dairy School, Kingston. The tests were made independently on the same milks by Messrs. Echlin and Cameron. Mr. Echlin did the work with the new test, and Mr. Cameron that with the centrifugal method. The resulting figures, as can be seen from the following table, are in surprisingly close agreement.

Centrifugal method	New method	Centrifugal method	New method	Centrifugal method	New method
2.6	2.64	2.25	2.35	2.2	2.27
2.7	2.69	2.3	2.35	2.4	2.43
2.5	2.44	2.35	2.43	2.4	2.43
2.55	2.61	2.6	2.59	2.4	2.43
2.55	2.61	2.5	2.51	2.5	2.59
2.55	2.61	2.5	2.51	2.45	2.51
2.3	2.36	2.25	2.27	2.4	2.43
2.3	2.36	2.25	2.27	2.4	2.43
2.6	2.61	2.2	2.19	2.45	2.49
2.5	2.53	2.35	2.35	2.5	2.55
2.5	2.53	2.35	2.35	2.3	2.27
2.25	2.28	2.35	2.35	2.35	2.35
2.25	2.28	2.4	2.43	2.4	2.43
2.2	2.20	2.4	2.43
2.5	2.56	2.35	2.35
2.6	2.63	2.35	2.35
2.7	2.67	2.35	2.35
2.3	2.43	2.3	2.27
2.3	2.43	2.3	2.27
2.5	2.43	2.4	2.43
2.45	2.35	2.4	2.43
2.56	2.67	2.4	2.43
2.56	2.67	2.3	2.27
2.55	2.59	2.25	2.27
2.55	2.59
2.55	2.59

The average difference for the above sixty-three determinations is 0.03+.

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I. A NEW ELECTRICALLY CONTROLLED AND TIMED ASPHALT PENETROMETER

II. THE EFFECT ON PENETRATIONS OF VARIATIONS IN STANDARD NEEDLES

By HERMANN W. MAHR

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I

Success in the laying of asphalt pavements is probably more dependent on the proper consistency of the asphalt cement used to bind the mineral aggregate than on any other feature. The varied origins of modern bituminous cements have made the determination of their consistency the most important test applied to these materials. Formerly a few varieties of standard solid bitumens, quite uniform in character, fluxed with definite proportions of petroleum residuums of standard and specified composition, yielding cements of a desired consistency, formed the bulk of the asphaltic cements; but recently many paving bitumens obtained by distilling asphaltic

petroleums to the consistency of cements have come on the market. Proximate chemical analysis is of little value in fixing their origin, and the highway chemist is obliged to rely almost solely on determinations of consistency to ascertain their suitability.

There are several rough methods for determining the consistency of asphaltic cements, but the most generally used scientific determination is by means of penetrometers. The first of these instruments was devised by Bowen, and has been followed by machines working on the same principle by Kenyon, Dow and the New York Testing Laboratory. The two latter are those generally used at present. The penetrometer has made it possible to control the consistency or as usually expressed, the penetration, within narrow limits.

The penetrometers consist essentially of a needle of specified size (Roberts, No. 2) fixed in a rod, the rod and needle being of, or loaded to, definite weights. A clamp of some nature holds the rod with the needle, allowing the latter to penetrate as nearly as possible without friction. A device for measuring the amount the needle has penetrated after it has been released for a specified time and again grasped by the clutch, is also necessary. The penetration is expressed in hundredths of a centimeter.

Penetrations are most commonly made at 25° C. (77° F.) with the needle loaded to 100 grams penetrating for five seconds. In order to ascertain the extent an asphaltic cement will harden when chilled to 0° C. (32° F.), penetrations are frequently made at this temperature with the needle loaded to 200 grams penetrating for one minute. Occasionally it is specified that cements shall not show more than a stated penetration at 37.7° C. (100° F.) or 46° C. (115° F.), the needle being under a weight of 50 grams and released for five seconds.

The Dow penetrometer frame consists of a base to which is fastened a broad upright support with two shelves at different distances from the base. The needle is held in an aluminum rod weighted by a rectangular frame of the same metal. The latter is fixed to the rod at about one-third the distance from its lower end. Weights are placed on the lower part of the frame to load the needle. The above-mentioned framework encloses the lower shelf on which is placed the sample. The rod passes through a hole in the upper shelf and is here grasped by the clamp which is closed on the rod by a spring when not penetrating. The rod and needle are released from the clamp by pressing the spring together with a button-ended rod.

The device for measuring the amount penetrated is fixed above the end of the rod. This consists of a rack, set vertically, the end of which can be brought down to meet the top of the rod. This rack is in gear with a pinion on a horizontal shaft. The latter passes through a graduated dial and an adjustable hand is fastened to it there. A counterweight which hangs from a cord winding up on a small pulley on the pinion shaft allows the rack to be raised or lowered. The divisions on the dial correspond to a movement of 1/100 cm. by the rack.

The Dow instrument has many features which make its use time-consuming. It also requires considerable skill and attention to obtain results checking within three or four degrees. The shelf, on which rests the water bath containing the sample, is fixed in position. This limits the height of the sample container and the bath. The weighted frame limits the width of the vessel used for a water bath, and is an inconvenience in placing the sample on the shelf and setting the needle. When the clamp is opened the space within is quite large. This allows the rod to deviate from a vertical position while penetrating. The weight of the large frame also tends to bring the needle out of the vertical. To avoid this action requires extreme care in setting, and even then it takes place to a small extent. When the rod is again clasped by the clamp, it is moved horizontally, thus bringing a force on the needle other than its weight. On bringing the rack down on the rod, after the needle has been set or penetrated, its weight is liable to drive the rod and needle down into the asphalt. The operator is inclined to avoid this by stopping the rack just above the top of the rod, thus introducing inaccuracies in the determination. The counterweight and its cord often become tangled in the shaft and shelves and are in the way.

These objectionable features of the Dow penetrometer led the New York Testing Laboratory to modify it. This instrument is described by Clifford Richardson in his work on asphalt paving.¹ The fixed shelf of the Dow machine is replaced by one on a screw. This allows the sample to be brought up to the needle very slowly, and the setting can be made with greater accuracy. The weighted frame is dispensed with and replaced by a weight on the lower part of the rod just above the needle. The rod slides in a collar of considerable height and is thus maintained in a vertical position. The clamp holding the rod is fixed in this collar and grasps it more firmly than that of the Dow penetrometer. Instead of a counterweight, the rack is kept in position by a spring pressing against it.

The wide collar, forming part of the clamp of the New York Testing Laboratory instrument, gives rise to considerable friction on the rod. This violates the basic requirement of the ideal penetrometer. The clamp requires the exertion of considerable force to release the rod. This tends to cause the operator to allow the clamp to shut before the expiration of the standard penetrating period. The force required to open the clasp is also liable to disturb the setting of the instrument and thus introduce inaccuracies.

The errors due to the construction of the present machines and to the personal equation of the operator often require a long series of determinations in order to obtain three results which lie within a limit of three or four degrees. Some specifications for asphaltic cement require its consistency to lie within limits ten degrees apart on the penetrometer. It is therefore difficult to interpret the specifications strictly with the results obtained on the present penetrometers. This has given rise to a demand for an instrument which eliminates some of the constructional errors of the present ones, and also some of the personal errors.

¹ "The Modern Asphalt Pavement," John Wiley & Sons

The errors in penetrating, apart from those of the instrument, are quite numerous. The sample, usually contained in a small tin box, must be firmly set in the water bath. This can be very satisfactorily accomplished by means of a glass vessel with a deep layer of fairly hard asphaltic cement.¹ The sample must have been at the standard temperature long enough to have attained it throughout. In this connection, laboratories working with a large number of asphaltic cements will find a good thermostat a great convenience. After a sample has been maintained thirty minutes at constant temperature it can be placed in the penetrometer water bath, filled with water from the thermostat at the same time.

A personal error of considerable magnitude has already been indicated, that of setting the rack on the rod before and after penetrating. In the timing of the penetration period lies probably the greatest error of the determination. The timing is done by either a stop-watch or a metronome. The use of the former is more open to inaccuracies than the latter.

The variation in the size of the standard needles has often been pointed out as a source of variation in results obtained by different chemists or by the same operator from time to time. The personal errors have been so large that no definite conclusions could be drawn as to the extent of this source of difference.

The importance of eliminating the time error and shock, due to the manual control of penetrometers, has long been recognized. With this object in view, Dow and Griffith devised and patented² an electrical limiting-time-interval clamp, to be applied to the then generally used Bowen penetrometer. This clamp clasped the thread supporting the weighted bar which held the needle, and released it for the desired intervals. This electrical limiting-time-interval clamp was cumbersome and complicated in its working and regulation. When Dow devised the penetrometer which bears his name, and which superseded the Bowen instrument, he omitted from it his time-interval clamp.

The advent of the simple, compact, Sieman's intermediate relay with time limits has made it possible to electrically time and control penetrometers of the present type. The penetrating device of the instrument can be directly controlled by the electrically timed magnet, instead of through the intermediary of a clamp, as in the Bowen-Dow-Griffiths electrical penetrometer. This simplifies the operating mechanism and reduces the chances of trouble from its derangement.

A new form of penetrometer, electrically controlled and timed, used for over a year in this Laboratory, is shown in Figure 1. Figure 2 is a sketch of the electromagnetic clutch for holding the rod with its needle. The clutch is on a bracket sliding on the upright rod of the instrument, to which it may be secured by means of a set screw. The weighted rod A, which holds the needle, is of steel, brass-clad. It slides through the openings in the thin German silver plates, C C, and is partly enclosed by the concave poles, B₁ B₂, of the electromagnet. The plates are set so the rod does not come into contact with the poles of the magnet, and are

¹ Bull. 38, Office of Public Roads, U. S. Dept. of Agriculture.

² U. S. Patent 512,687 (1894).

rounded where they touch the rod, thus eliminating all friction. The rod and needle weigh 50 grams and are weighted to 100 and 200 grams by weights of 50 and 150 grams, respectively, slipped over the rod above the needle, and there fastened by set screws.

The penetration is measured, as in other machines, by means of a rack and pinion, the latter being fastened to an adjustable hand on a dial. The counterweight and spring, used in the previous types of instruments for holding the rack in place, are dispensed with. The pinion shaft has an additional gear wheel. This gear is in mesh with a worm on a shaft at right angles to the first, the worm shaft being driven by means of a milled head at one side of the dial. By turning the milled head the rack may be raised or lowered and accurately set on top of the rod. In setting the needle on the surface of the sample, the latter is first raised to within less than a millimeter of the former. The rod is then forced down by the rack until the needle touches the surface of the asphaltic cement. During the setting the rod is held by the magnet, the electro-magnetic force being overcome by that exerted by the

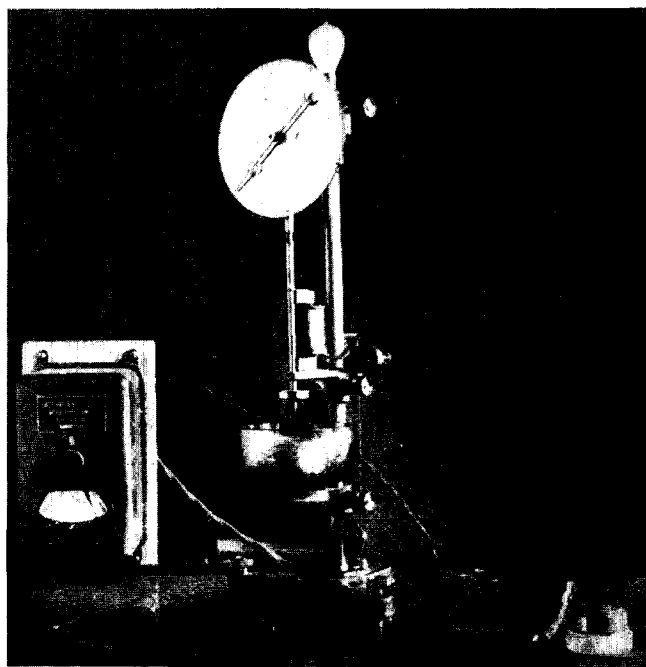


Fig. 1

rack. This device and method of procedure enable very accurate settings of the needle and rack to be made.

The sample is placed on a movable shelf which can be rapidly lowered to allow cleaning of the needle. In order to set the machine in a vertical position, a plumb bob and adjusting screws are attached to the penetrometer. A small electric lamp, fastened to the top of the standard and in series with the electro-magnet, indicates when the current is on. The instrument is wound to be connected to a 110 volt direct current circuit.

The use of the electro-magnet clutch precludes the shock or displacement of the machine, due to a clamp

operated by hand. The circuit is broken by pressing a button. To eliminate the time error of the observer, a Sieman's intermediate relay with time-limit is used. The penetrometer is placed in the circuit with one side of this device, and the lighting circuit passed through a switch and the relay electro-magnet. On breaking the current through the latter, the penetrometer circuit is broken and automatically made at the end of five seconds.

The accuracy of the penetrometer was tested by determining the penetration of three asphaltic cements, using the same needle. The penetrations were made at 77° F., with the needle loaded with 100 grams, penetrating for 5 seconds. After each observation the needle was cleaned with chloroform, dried, and brought to 77° F.

The results are given in the following table:

PENETRATION OF ASPHALTIC CEMENTS. (77° F., 100 g., 5 sec.)

	Oil asphalt cement No. 1	Oil asphalt cement No. 2	Trinidad asphalt cement
Determination No. 1.....	55	53	85
Determination No. 2.....	55	53	84
Determination No. 3.....	55	53	84

II

Richardson¹ has stated that variations in the size of the needles give rise to uncontrollable variations in penetrations determined with them.

Since all errors are eliminated in the machine described, it was possible to ascertain the extent of this variation. Three needles were selected from each of three packages of standard needles. Oil asphalt cement No. 1 was then penetrated, using these needles, taking the precautions outlined above.

PENETRATION OF OIL ASPHALT CEMENT NO. 1, BY DIFFERENT NEEDLES (77° F., 100 g., 5 sec.)

Needle	Package	Penetration	Needle	Package	Penetration
1	1	55	6	2	55
2	1	55	7	3	55
3	1	54	8	3	54
4	2	54	9	3	54
5	2	54			
Average, 54.6					

The greatest deviation from the mean is 0.6 of a unit.

CONCLUSIONS.—A new form of penetrometer, which eliminates the errors due to the construction of previous instruments and permits rapid accurate determinations, has been described.

The personal error in timing penetrations has been eliminated through the use of this penetrometer in connection with an intermediate relay with time-limit.

The variations in determinations of penetration due

¹ Loc. cit.

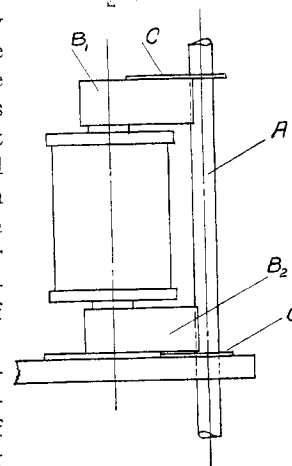


Fig. 2

to variations in the standard needles has been investigated and found to be negligible.

The writer wishes to express his appreciation of the help rendered by Mr. Walter Erlenkotter and Mr. A. D. St. John, of this Laboratory, in connection with this paper.

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AN APPARATUS AND METHOD FOR DETERMINING THE HARDNESS OF BUTTERFAT

By A. E. PERKINS

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The desirability of having an accurate method for determining the hardness or consistency of butterfat and other fats has been felt for a number of years. Several methods and apparatus having a greater or less degree of efficiency have been devised from time to time.

The first methods were proposed for the examination of olive oil. Of these earlier methods the most practical was devised by Serra Carpi.¹ His method was to cool the olive oil down to 20° C., for three hours, and by means of a suitable arrangement, he placed on the solidified fat a cylindrical iron rod, 2 mm. in diameter and 1 cm. long and conical at the bottom. Weights were then put on to the rod until it sank completely into the fat. Thus, for pure olive oil 1700 grams and for cottonseed oil 25 grams were required.

Woods and Parsons² report satisfactory results in determining the hardness of butter by the use of a method which consists essentially of dropping a weighted glass rod through a glass tube 1 meter long, held vertically above the surface of the butter to be tested, and noting the depth of penetration on a scale attached to the glass tubing. The determinations were performed at 15.5° C. after the butter had stood in a cool room for several days. The results were expressed in mm. of penetration.

R. Brulle³ measured the hardness of butter with an apparatus which he called an "oleogrammeter." This is an instrument consisting of a vertical rod on the top of which is fixed a large plate. The rod is allowed to slide in a ring fastened to a stand. The end of the rod is placed on the surface of the solidified fat, and then weights are put on the dish until the rod sinks briskly into the fat. The weights required represent the resistance of the butter to the "oleogrammeter." The butter samples were kept at 21° C. for an hour before testing. This method is essentially the same as that used by Serra Carpi⁴ for olive oil.

Sohn⁵ proposes three forms of apparatus and lays down the following rules, strict adherence to which are necessary:

1. The rod must descend in an absolutely perpendicular direction.

2. It must slide in its bearing with the least possible friction.

3. Conditions of temperature must be constant.

4. Vessels of one diameter must be used for the material under examination.

5. The rod must enter the center of the vessel or at a fixed distance from the circumference.

6. The same depth of material must always be used.

7. The material must be allowed to rest a certain fixed time before testing.

J. B. Lindsey¹ and his associates determined the hardness of butter with an apparatus analogous to that used by Woods and Parsons,² the only real difference being that they dispensed with the large glass tube and the depth of penetration was measured on the plunger. Their determinations were performed on butter which had been in cold storage for some time and then allowed to stand at room temperature for several hours.

Hunziger, Mills and Spitzer³ report results in measuring the hardness or "mechanical firmness" of butter with an apparatus which measured the crushing effect of a plunger, size and weight not stated, on briquettes of manufactured butter. No mention is made of the conditions of temperature under which the tests were conducted.

In some of our experimental work here, we considered it desirable to measure the hardness of butter fat with a reasonable degree of accuracy, and in such a way that results obtained throughout experiments extending over long periods of time would be entirely comparable. After trying several of the above-mentioned methods, none of which proved delicate enough for our purpose, the following apparatus and method were devised.

The apparatus used, as shown in the accompanying cut, comprises a firm support (A) and a separate light frame (B) carrying the penetrating needles and the weights. The support consists of a heavy iron base (c) into which are inserted 2 upright rods (d and e) about 1 meter long, one of which is hollow and contains wires connecting the electro-magnet (g) with (h). These uprights are about 25 cm. apart and are joined together at the top by a piece of hollow iron rod (f): (h) and (h) are binding posts for attaching the batteries to operate the magnet. A key (j) attached to the base serves for making and breaking the current through the magnet: (k) and (l) are millimeter scales reading downward. Attached to the upright rods is an adjustable platform (m) for carrying the sample whose hardness is to be tested. The coarser adjustment of the height of the platform is secured by means of the clamps (n) and (o). This adjustment does not need to be regulated except at rare intervals. A finer adjustment is secured by simply turning (m) which is supported from (n-o) by a 3/4 inch nut and screw. The frame is of hollow brass tubing to get it as light as possible, while still retain-

¹ Serra Carpi, *Z. anal. Chem.*, 1884, **23**, 566.

² Woods and Parsons, *Bull.* **13**, New Hampshire Exp. Sta.

³ R. Brulle, *Compt. rend.*, 1893, **116**, 1255.

⁴ *Loc. cit.*

⁵ Sohn, *Analyst*, 1893, p. 218.

¹ Lindsey and Associates, 13th Ann. Rept. Hatch Exp. Sta., p. 28; 14th Ann. Rpt. Hatch Exp. Sta., p. 167; 16th Ann. Rpt. Hatch Exp. Sta., p. 59; 21st Ann. Rpt. Hatch Exp. Sta., p. 99.

² Woods and Parsons, *loc. cit.*

³ Bull. 159, Purdue University, Indiana Agr. Exp. Sta.