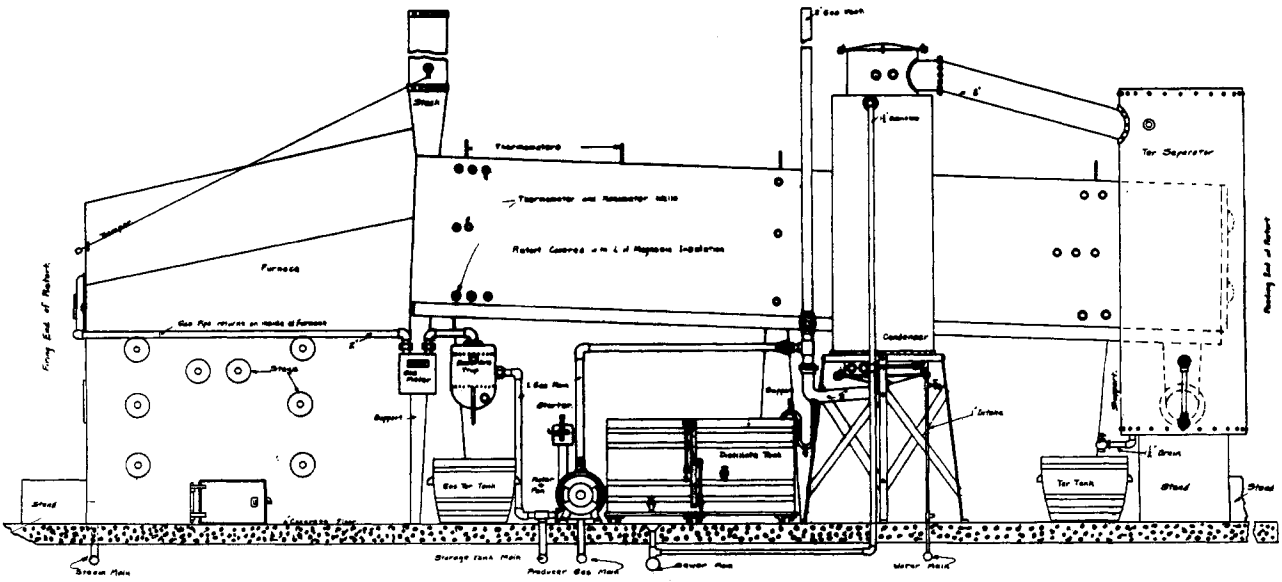

PLATE 5—SIDE VIEW. RETORT AND APPARATUS SET UP

TOTAL CONSUMPTION OF HEAT UNITS

	B. t. u.
(1) To heat and vaporize 150 gallons of distillate	1,430,000
(2) Radiation through insulation	1,512,000
(3) Radiation through exposed door	412,000
(4) To heat buggies and retort 600° F	990,000
(5) To heat wood 300° F	300,000
(6) Heat carried away by non-condensable gases	56,700
(7) Minor losses	100,000
Total heat lost in 20 hours	4,800,700
Total heat lost per hour (roughly)	250,000

plied; A, the total area of the flue surfaces; T, the average temperature of the gases within the flues; t, the average temperature within the retort; r, heat units given off by radiation; and c, heat units given off by convection. Hence,

$$Q = 0.416 \times 3.14 \times 16.6 \times 8\,(1500 + 700/2 - 700)\,(3.63 + 1.06),$$

or 325,000 B. t. u. will be supplied per hour. Accordingly by computation, the heat exchange is shown to be more than sufficient to produce distillation taking place under the conditions as above assumed, and this computation is verified by the results obtained in the operation of the retort, which requires from 18 to 20 hours per run of three-eighths to one-half cord of wood.

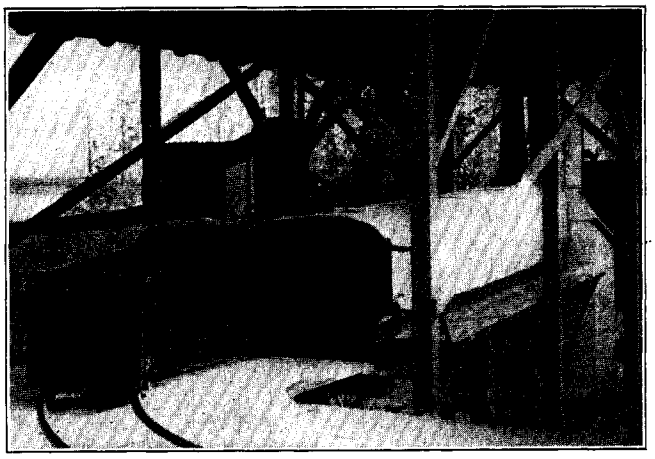

PLATE 6

In Plate 6 is given a general view of the plant as it appeared shortly after its installation.

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AN APPARATUS FOR DETERMINING THE MELTING POINTS OF SUBSTANCES OF INDEFINITE MELTING POINT

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The determination of the true melting point of substances which do not melt sharply at a definite temperature but which gradually soften under the influence of heat and finally become soft enough to be liquid, is one of great difficulty. Indeed it can hardly be said that such substances have any true melting point. Ordinary coal tar pitch such as is used for roofing, etc., is an example of the class of substances under discussion here. As is well known this material at the ordinary atmospheric temperature when struck with a hammer flies to pieces like glass. Nevertheless, if a barrel of it stands for any length of time the pitch will slowly flow out of every crack and crevice or out of the open bung-hole just like a liquid. It may indeed be called a brittle liquid, and this class of substances might be called solid liquids. If coal tar pitch is warmed a little it gets softer and flows somewhat more rapidly; if it is further heated it becomes still softer and flows still more rapidly. Finally, when hot enough it can be made to flow nearly or quite instantaneously like water. When shall we say that coal tar pitch is melted?

Petroleum asphaltums prepared by oxidizing petroleum residuum also belong to this class of bodies, although their characteristics are somewhat different from those of coal tar pitch in that they are softer at ordinary temperatures and require higher temperatures to flow together when exposed for long periods of time to small degrees of heat. They also have a

higher range of melting points as this term is understood in this article.

Experiment shows that an air-blown petroleum asphaltum which when heated rather rapidly becomes liquid at about 135° C., will flow down an inclined plane upon which it may be placed at 40° C. if kept at that temperature a long time, say 18 hours. Shall we say that this substance fuses at 40° C. or at 135° C.?

There appear to be two stages or two temperature points in this gradual softening, which may be of importance in the examination of these substances. *First*, there is that temperature point or approximate point, at which the firm solid or pseudo-solid material just begins to soften, so as to be slightly plastic, or so that if left a long time at that temperature it will flow and finally come to a level in its container. *Second*, there is a temperature at which the substance is so fluid that it can readily and quickly flow to a level in its containing vessel; in other words, it possesses the properties usually ascribed to liquids.

The apparatus now to be described is one for determining the temperature of this second kind of melting. It can scarcely be called a flow point apparatus, although the flow of the sample is measured.

It consists of a block of iron of suitable dimensions, with one face at an angle of 45° to the horizontal. This inclined face is provided with grooves, as further described below. The block is also provided with a small cistern to contain mercury in which is placed the thermometer for reading the temperature of the block. A useful size for this apparatus is about $4^1/_2$ inches wide, 4 inches high and about 5 inches long. This will contain 9 grooves of the size described below. A top view is shown in Fig. 1, and a side view in Fig. 2; here, *A* is the mercury

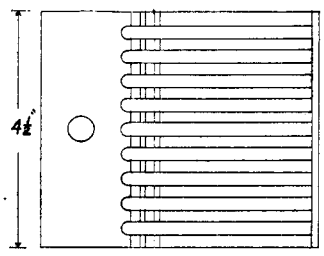

Fig. 1

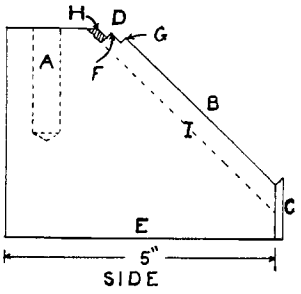

Fig. 2

cistern, *B* the inclined grooved face, *C* is a small stop arranged so that a sheet of glass, or transparent mica laid over the inclined face will be supported and prevented from sliding off, *D* is a series of step-like cuts made across the ribs between the grooves of the inclined face. The top cut *FH* is deeper than the second by one-eighth of an inch, the second cut *FG* is one-eighth of an inch deeper than the top of the ribs on the inclined surface. The length of the top cut is one-quarter of an inch from the angle *H* at the top, down to the step up *F* of the second cut; the length of the second cut is one-quarter of an inch from *F* to *G*.

The grooves are one-quarter inch wide, and their vertical sides are one-quarter inch deep to the beginning of the curved bottom. The latter is of semicircular cross-section with a radius of one-eighth inch. The ribs may be $^7/_{32}$ inch thick. The dotted line *I* represents the bottom of the grooves.

For use, mercury is poured into the cistern *A*, which does not need to be very wide (say $^1/_2$ to $^3/_4$ inch) and the thermometer is inserted. The samples of material to be tested are pressed into the topmost parts of the grooves between *F* and *H* so as to fill the space which is shaded in Fig. 2. About 0.17 gram of asphaltum is needed for this. A sheet of glass or mica is laid on *B* (to shut off cold air drafts), and heat is applied at *E* by means of a Bunsen burner. Each sample will gradually soften and sag down the groove; when it is liquid enough to reach from *F*, its original lower boundary, to *G*, a distance of one-quarter inch, the thermometer reading is taken as the melting point. Marks may be made at intervals down *B*, say at every one-half inch and the behavior of the flowing material as it passes each in succession may be studied as to temperature and time differences.

It is important that the amount of material used in comparative trials should be sensibly the same. Otherwise the melting points observed will differ somewhat according to the relative sizes of the samples. Thus in two trials, certain specimens of air-blown petroleum asphaltum were applied in differently sized pieces and each series tested at the same time with the following results:

Melting Points of Differently Sized Pieces of Petroleum Asphaltum

Sample	No. 1	No. 1	No. 2
Rate of heating for 10° C.....	77 sec.	100 sec.	100 sec.
Small piece.....	184° C.	181° C.	183° C.
Medium piece.....	180° C.	175° C.	178° C.
Large piece.....	178° C.	171° C.	174° C.

It will be seen that sample No. 1 showed in one case a difference of 6° C. in melting point and in the other case of 10° C., depending on whether a large or a small piece was used. The large piece of No. 1 used at rate 77 sec. was not the same in size as that used in rate 100 sec.; the same remark applies to the medium and small pieces. The large pieces were about the size of peas, the small pieces about one-third this size.

In order to overcome this cause of discrepancy the space in the upper part of the groove should, as already said, be filled with the material to be tested, and then the top scraped off even with the surface of the partly cut off rib. By this means sensibly the same sized sample may always be subjected to test, thereby eliminating one of the uncertainties of the melting point of the substances under consideration.

The reason that a larger piece appears to fuse at a lower temperature than a smaller piece seems to be that the action of gravity is relatively greater at the same time that the action of capillarity is relatively less, with the larger piece. The first force tends to pull the mass down the incline, and so to render the apparent melting point lower. The second force tends to prevent the liquefied, or semi-liquefied sample from flowing down and, therefore, to render the apparent melting point higher. These circumstances

necessarily operate in most other methods of determining the melting point of the classes of bodies under consideration. But in this apparatus the variations can be controlled by exactly filling the space as described, whereas in most other methods no such control is as easily obtainable.

As has already been stated material of indefinite melting point will flow at low temperatures if given time enough. Time is therefore an important factor in this determination and must be taken into account.

That the temperature at which substances of indefinite melting point actually fuse depends considerably on the rate of heating is illustrated by the following table:

MELTING POINTS OF AIR-BLOWN PETROLEUM ASPHALTUM FOR DIFFERENT RATES OF HEATING

Rate for 10° C.	43 sec.	65 sec.	68 sec.	75 sec.	113 sec.	126 sec.
Sample No. 1	119° C.	114° C.	114° C.	115° C.	...	112° C.
2	140	134	133	135	128° C.	128
3	157	154	150	149	147	147
4	161	160	156	156	151	150
5	...	170	169	166	157	...

It follows, if comparable results are desired, that the heating up must be conducted at a given rate.

For many purposes, however, where simply relative results are desired, there is no need of heating at a definite rate with this apparatus provided only that the rate of heating is comparatively uniform.

For such relative determinations the several samples to be compared are pressed into the grooves as described, and the several melting points observed are read and compared. Even very slight differences of melting point can be observed in this way.

A rate of about 100 seconds for a rise of 10° C. is the most convenient for use with an apparatus of the size already described. This rate is readily obtained with a good sized Bunsen burner burning so that the flame impinging on the bottom of the block forms a circular disk of about $3^1/_2$ inches in diameter—at least that is the size with the pressure of artificial gas in the city of Cleveland.

All methods heretofore proposed, so far as I am aware, for determining the melting points of substances of indefinite melting point are also subject to some rate of heating factor. This rate is controlled with great ease in the method here described, and may be entirely neglected with this apparatus under the conditions mentioned above.

The rate of heating is without influence on the temperature at which substances of sharp melting point fuse in this apparatus. For example a certain sample of commercial stearic acid, which fused quite sharply at $59^3/_4$° C. by the usual capillary tube method, fused by the iron block method at $59^3/_4$° C. when heated 10° C. in 125 seconds, and at 60° C. when heated 10° C. in 50 seconds. This last rate is about the fastest; it is feasible to use by this method. Substances of sharp melting point do not gradually sag down the incline as the heating progresses. They remain entirely unchanged until the temperature reaches the melting point, and then they suddenly fuse and flow down the inclined groove quite rapidly.

Hence, for determining the melting points of substances of sharp melting point for commercial purposes this apparatus will be found of great use.

Lastly, in at least some cases, the melting point of substances of indefinite melting point is influenced by the amount of working or kneading to which the sample has been subjected. This is exemplified by the following figures; the rate of heating was 10° C. in 77 seconds:

MELTING POINTS OF AIR-BLOWN PETROLEUM ASPHALTUM

	Not kneaded	Well kneaded
Small piece	170° C.	167° C.
Large piece	167° C.	160° C.

Here the kneading has lowered the melting point from 3° to 7° C. This is the most difficult condition to control in the samples submitted to test, because it is impossible to avoid some kneading in pressing the sample into the slot of the machine. The best way to overcome it, and to secure comparable results, is to thoroughly knead each pellet of the samples before pressing them into the slots. Here again the same factor of kneading enters into all methods heretofore proposed for determining the melting points of this class of substances.

As above stated all these conditions operate to cause variations in melting point in every form of apparatus heretofore proposed. But in none of them, so far as the author has tried them, is it possible to satisfactorily overcome the inaccuracies and variations caused by the peculiarities in behavior of these materials of indefinite melting point. But by means of the proper use of the apparatus herein described it has been possible to attain accuracy of results entirely unattainable before this apparatus was put into use.

During the two years in which this apparatus has been in use in this laboratory, the author has found it of great value for lubricating greases of every kind, for pitches, asphaltums, solid insulating compounds, and, in short, for every kind of substance whose melting point is lower than the boiling point of mercury and whose range of fusion is too wide for obtaining definite results by the capillary tube method. For such materials it appears to be as generally useful as the capillary tube is for those of sharp melting point.

The samples are quickly inserted into the slots, it takes but a short time to heat up to the melting point, the temperature is easily and accurately read, many samples can be tested at once, and the apparatus is easily cleaned.

The best way of cleaning this melting-point block is to immerse it while still warm into a can of liquid which is a solvent for the substance tested, for example naphtha, if petroleum pitch or lubricating grease is in the apparatus. After a short soaking the samples can usually readily be scraped or wiped out, and then the slots are easily washed with a small piece of cotton waste or other rag. After that it is rinsed with clean naphtha and allowed to dry, when it is ready for use.

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