

*Chem. eng'g
thesis cover*



THE EFFECT OF REFLUX IN THE CONTINUOUS SEPARATION OF
HYDROCARBON MIXTURES BY RECTIFICATION

by

L. P. Elliott

B. S. University of California

1922

and

Emil H. M. Lehnhardt

B. S. University of California

1923

Submitted in Partial Fulfillment of the Requirement

for the Degree of

MASTER OF SCIENCE

from the

Massachusetts Institute of Technology

1924

A C K N O W L E D G M E N T.

The authors wish to express their appreciation for the kindly criticism and invaluable suggestions afforded by Prof. W. K. Lewis under whose direction this thesis was prepared, and to extend their gratitude to Mr. Welling of the Research Laboratory of Applied Chemistry whose helpful advice was of assistance in the manipulation of the apparatus.

T A B L E O F C O N T E N T S.

	Page
I. Introduction.	
(1) Purpose of investigation. - - - - -	
(2) Theory of rectification. - - - - -	
(3) State of the art of rectification in the petroleum industry - - - - -	
(4) Previous investigation - - - - -	
II. Summary - - - - -	
III. Experimental work	
(1) Proposed method of attack - - - - -	
(2) Description of apparatus - - - - -	
(a) The continuous still, using reflux - -	
(b) The continuous still, using no reflux -	
(c) The analytical still - - - - -	
(3) Procedure	
(a) The continuous still, using reflux - -	
(b) The continuous still, using no reflux -	
(c) The analytical still - - - - -	
IV. Results - - - - -	
V. Recommendations - - - - -	
VI. Calculations - - - - -	
VII. Sample calculations - - - - -	
VIII. Original data - - - - -	
IX. Bibliography - - - - -	

I I N T R O D U C T I O N

(1) Purpose of investigation

Due to the very large and increasing demand for the lighter fractions obtained in the distillation of petroleum it is essential, from the point of view of the refiner, that all of the light components which are salable as gasoline be removed from the higher fractions, insofar as this is economically feasible.

It is well known that these light components can be practically completely separated by the use of a rectifying column but, unfortunately, the heat requirements for complete separation are so large that it is not economically advisable to carry the rectification to this point. The question the refiner must solve is how far he can carry the separation before its cost becomes greater than the value of the product which he obtains.

In order to determine the optimum conditions for the operation of a rectifying still it is necessary to know the relationship between the heat requirements and the separation obtainable. This relationship has been quite accurately determined for certain binary mixtures but practically no data are available for petroleum mixtures.

Therefore, the primary object of this experimental investigation is to study the heat requirements for distillation, insofar as they influence

the degree of separation of the light components from
the heavy components.

(2) Theory of rectification

The separation of liquid mixtures by distillation is based on the fact that when they are heated, the vapor given off is richer in the more volatile component than is the liquid from which it comes. The ideal distillation process, at constant pressure, consists of allowing vapor and liquid to come into intimate contact in a column. The vapor rising from the still passes upward and meets the descending liquid and, in the ideal column, comes into equilibrium with it. Due to differences in relative volatility the ascending vapor becomes richer in the more volatile constituent whereas the descending liquid becomes poorer in this constituent. A concentration gradient is thereby set up in the column and if the column is sufficiently high the vapor leaving the top will consist of only the more volatile component. The liquid flowing down thru the tower is called reflux. It is formed by condensing part of the vapor. The larger the amount of reflux returned to the still, per unit of product, the greater will be the concentration gradient thru the column and hence the better will be the degree of separation.

In any actual column it is impossible to bring the vapor and liquid into complete equilibrium due to imperfect contacting. The extent to which a true equilibrium may be approached depends largely upon the material with which the column is packed.

Reflux may be obtained either by partial condensation of the vapors at the top of the column or by allowing the column to be cooled by radiation. If the latter method is employed it is easily seen that there will be more reflux at the bottom of the column than at the top. This is undesirable since maximum separation, for a given amount of heat used in producing reflux, will occur when the reflux is a maximum throughout the column.

Furthermore, partial condensation serves to increase the degree of separation, due to the tendency for the less volatile material to be condensed in preference to the more volatile.

The laws governing the separation of simple mixtures have been worked out mathematically and are quite well understood. However, in the case of complicated mixtures such as petroleum it is obvious that a rigid mathematical treatment would become hopelessly involved. For a qualitative examination of this problem it is possible to consider the complicated mixture as if it were a two component system.

In the distillation of such a mixture, in a continuous still, there are five variables;

- (1) Height of column and type of packing, or number of theoretical plates.
- (2) Point of introduction of feed.
- (3) Heat supplied to still and temperature of feed, --- equivalent to reflux.
- (4) Relative volatility of distillate and bottoms.
- (5) Completeness of separation of distillate and bottoms.

In order to investigate the relationship between these variables it is of course necessary to maintain all but two of them constant. The first two; namely, the height of column and the point of introduction of the feed are, for a given apparatus, fixed. The relative volatility of distillate and bottoms will be constant providing that the ratio of distillate to bottoms is constant, and that the composition of the feed is constant. This leaves only the completeness of separation and the heat required for the separation as variables. As previously stated, this is the relationship desired in this investigation.

(3) State of the art of rectification in the petroleum industry.

In many industries in which distillation is an important plant procedure, column apparatus has been developed almost to the exclusion of other types of equipment for separation of liquid mixtures. Curiously, the petroleum industry has, until quite recently, taken but slight advantage of the many desirable points of column distillation. It has developed and used dephlegmating equipment almost exclusively.

Dephlegmating apparatus produces separation by fractional condensation. This condensation must be distinguished from the partial condensation used to produce reflux in the rectifying column. The primary purpose of the reflux condenser is to return a definite amount of liquid to the column in order that separation may take place in the column. But fractionating condensers are intended to produce a vapor richer in the more volatile component than the vapor entering the apparatus.

It appears that in some refineries the first principles of the operation of a rectifying column are not understood. This is shown by the fact that expensive columns are known to have been erected, carefully lagged to prevent heat loss by radiation, and then operated without reflux. This method would of course give maximum amount of distillate per unit of heat but the separation is equivalent only to that obtainable with a simple still.

However, during the past few years the number of rectifying columns used in oil refineries has increased rapidly. Compared with the previous methods of separation they have constituted a tremendous advance in the art, but so little is known about their correct operation that it is doubtful that many of them are being operated in the most economical manner.

(4) Previous investigation.

The available data regarding the separation of hydrocarbon mixtures by rectification are extremely meager. The work which has been done was chiefly for the purpose of developing an analytical method for the determination of the true boiling point curve. In practically all of this work a batch still was used rather than a continuous still such as would be most advantageously used in refinery practice.

However, Reeder and Gordon (M.I.T. Thesis 1922) made two runs on a continuous, plate type, still using "Socony" gasoline fed directly to the still. Their data for these two runs have been recalculated and will be discussed later.

W.T. Davis (M.I.T. Research Lab. of Applied Chemistry 1923) has perfected a method of determining the true boiling point curve of hydrocarbon mixtures. This method is based on the principle that the distillation temperature of the various fractions will very closely approximate the boiling point of the pure components providing that a sufficiently large reflux ratio is maintained and that the rectifying column is of sufficient height. Reflux ratio is defined as the ratio of the amount of liquid condensed and allowed to flow back into the column, to the amount of liquid condensed as product.

Davis found that in order to obtain satisfactory separation of normal gasoline blends with a column packed with 28' of carbon nipples of cylindrical shape, $7/32$ " outside diameter by $7/32$ " long and with a $1/16$ " concentric hole, it was necessary to maintain a reflux ratio of 12:1. With a shorter column it would be necessary to increase this ratio. He also found that the boiling point difference between the fractions being separated influenced the reflux necessary for the separation. For a gasoline blend containing considerable benzol it was necessary to increase the ratio to about 20:1 during the period at which this component was being distilled.

McFarland and McGrath (M.I.T. Thesis 1924) began the investigation considered in this thesis. Lack of time prevented them from obtaining any quantitative results.

II S U M M A R Y.

The effect of reflux on the degree of separation of hydrocarbon mixtures when they are subjected to continuous distillation in a fractionating column has been studied for the case where there is no rectification above the feed plate. The relationship between reflux and separation was found to be hyperbolic in nature, with a limiting value for the separation obtainable in the given column.

The degree of separation obtained in the continuous distillation of a hydrocarbon mixture without reflux was also studied. The degree of separation in this case is surprisingly small.

The degree of separation was found to be a function of the size of the cut.

III EXPERIMENTAL WORK

(1) Proposed method of attack.

In order to investigate the relationship between degree of separation and the heat requirements for the separation of petroleum mixtures in a continuous rectifying column it was proposed that a column be operated with ordinary "Socony" gasoline as feed. In general, the relationships for this mixture should be similar to those of other mixtures of hydrocarbons.

The "bottoms" from the still were to be analyzed by determining their true boiling point curves. From a comparison of these curves with that of the feed the amount of "lights" left in the "bottoms", i.e. the degree of separation, could be determined.

The reflux was to be obtained by partial condensation of the vapor in the still-head by means of oil passing thru a cooling coil. The oil circulating apparatus was to be so constructed that the volume of oil passing thru the system per unit of time, as well as its temperature rise in passing thru the coil could be determined. From this data the heat required for the production of the reflux was to be calculated.

The degree of separation obtainable with zero reflux, i.e. by straight distillation, was to be

determined in a simple continuous still without
a column.

(2) Description of apparatus.

(a) The continuous still, using reflux.

The continuous still used in this investigation was designed and built by McFarland and McGrath (M.I.T. Thesis 1924) and is described in great detail by them. A diagram of the apparatus will be found in this report.

still

The still itself consisted of a section of eight inch standard iron pipe, approximately one foot in length, onto each end of which caps had been screwed and welded to prevent leakage. Into the side of the lower cap a half inch standard iron pipe was screwed and welded. This pipe was for the purpose of drawing off the bottoms from the still and had a slight upward slope in order that the still would not be completely drained by it. The end of this overflow pipe was connected to a twenty inch, water-jacketed condenser. A thermometer well extended from the top to near the bottom of the still.

Column

A four foot section of standard two-inch iron pipe was connected to the top cap of the still by means of a union, a six inch piece of similar two inch pipe, and a 3"x2" standard bushing. A wire screen was forced into the column slightly above the union for the purpose of supporting the column packing which was placed in the four foot section of

pipe.

Column packing

The column was packed for a distance of three feet with sections of 6-8mm glass tubing, the length of which were approximately equal to the diameter.

Partial condenser

The column head consisted of a two inch standard lateral in which a condensing coil was suspended. This coil was constructed from four feet of $\frac{1}{4}$ " copper tubing wound in a spiral. The ends of the coil protruded thru the lateral and were connected thru compression joints to three-eighths inch pipe in which were thermometer wells to enable the operator to determine the rise in temperature of the oil circulated thru the condenser.

Oil circulating system

This system consisted simply of a closed circuit. The oil was pumped from the bottom of a pail, thru a half-inch pipe, to the partial condenser by means of a small rotary pump and was then returned to the pail. A by-pass was constructed around the pump in order that the oil rate thru the condenser might be controlled. As it was sometimes necessary to heat the cooling oil a circular gas burner was placed under the pail.

Head temperature

The temperature at the top of the column was determined by introducing a thermometer into the column head thru a compression bushing. To prevent radiation to the cooling coils this thermometer was shielded by a small lead cone as recommended by W.T.Davis.

Total condenser

The top of the column head was connected to a thirty inch, water-jacketed condenser to condense the product from the column.

Feed reservoir

The feed reservoir was a five gallon galvanized oil can with a sight glass on its side. To its bottom was soldered a short piece of copper tubing which was connected to an eighth-inch needle valve. This reservoir fed into the constant head tank.

Constant head device

The constant head device was constructed from a three inch iron pipe one foot high, with a sight glass on the side. A swivel joint was placed at the bottom and connected to an overflow pipe in such a manner that the height of liquid in the tank could be maintained at any desired point by raising or lowering the overflow pipe. In order to maintain a constant rate of feed to the still an orifice was made by drilling a hole 0.041" in diameter in a piece

of galvanized iron. This was soldered onto the top of a half inch pipe and the pipe was screwed into the bottom of the constant head tank.

Still and column insulation

The still was surrounded with brickwork to prevent cooling by air currents. The column was insulated from heat loss by means of a flue gas jacket consisting of a piece of six inch galvanized iron pipe. This pipe was open at the bottom and so situated that the hot flue gas from the burner under the still passed thru it. When this source of heat was insufficient burner gas from a Bunsen burner was admitted thru a short piece of 2" stove pipe which was riveted to the side of the larger pipe.

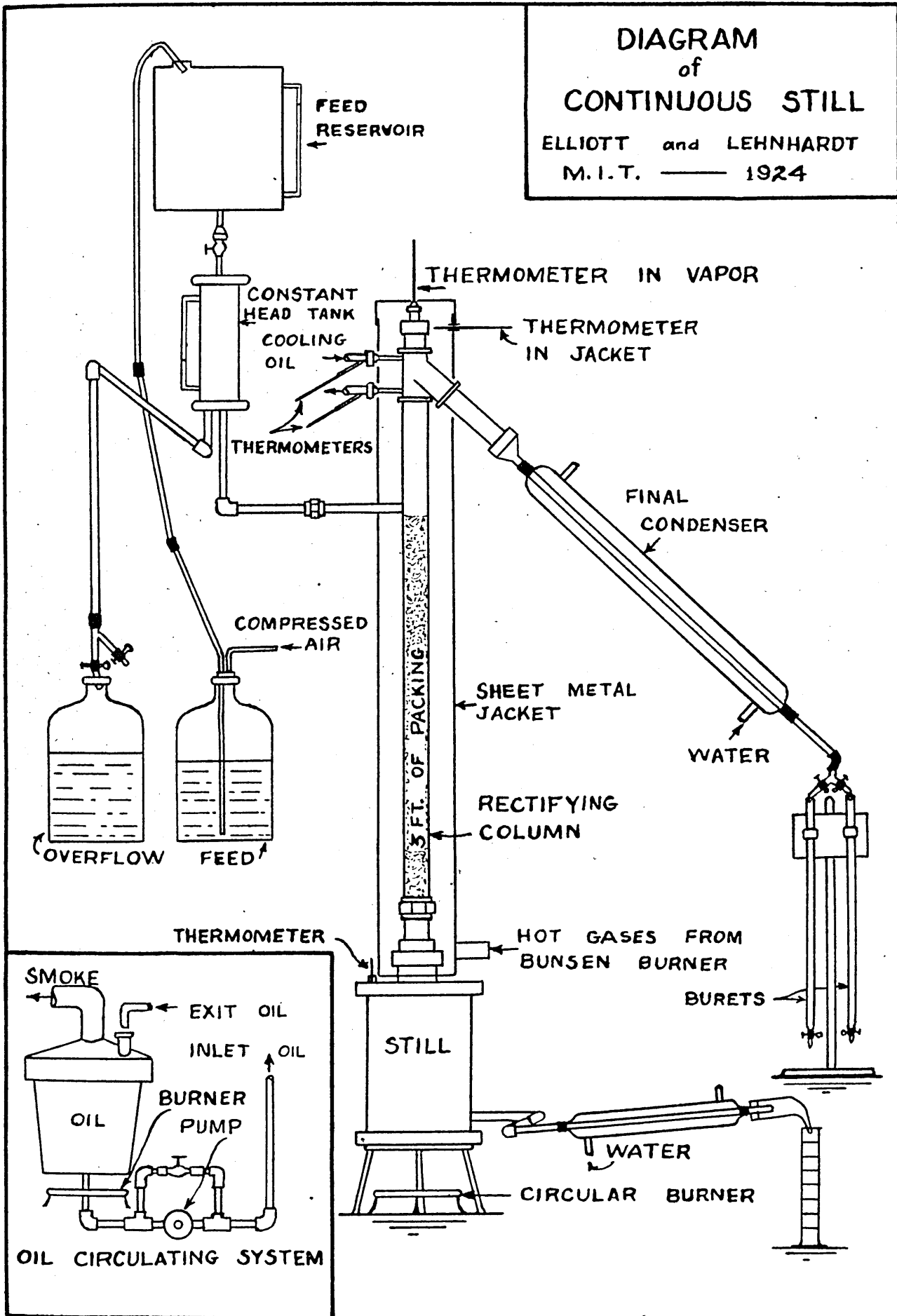
Receivers

The distillate coming from the total condenser was caught and measured in two 50cc burets so arranged that they could be filled alternately.

The bottoms were allowed to overflow into a graduate.

DIAGRAM of CONTINUOUS STILL

ELLIOTT and LEHNHARDT
M.I.T. — 1924



Description of apparatus

(b) The continuous still using no reflux

This still consisted of a two liter Pyrex short necked round bottomed flask. The upper half of the flask, including the neck, was covered with half an inch of magnesia and the entire flask was surrounded with insulating boards in such a manner that the hot flue gas from the burner jacketed the flask and maintained it at a temperature slightly higher than that of the liquid in the flask.

The feed was introduced into a separatory funnel, from a graduate, and allowed to flow into the flask thru a glass tube reaching nearly to the surface of the liquid in the flask.

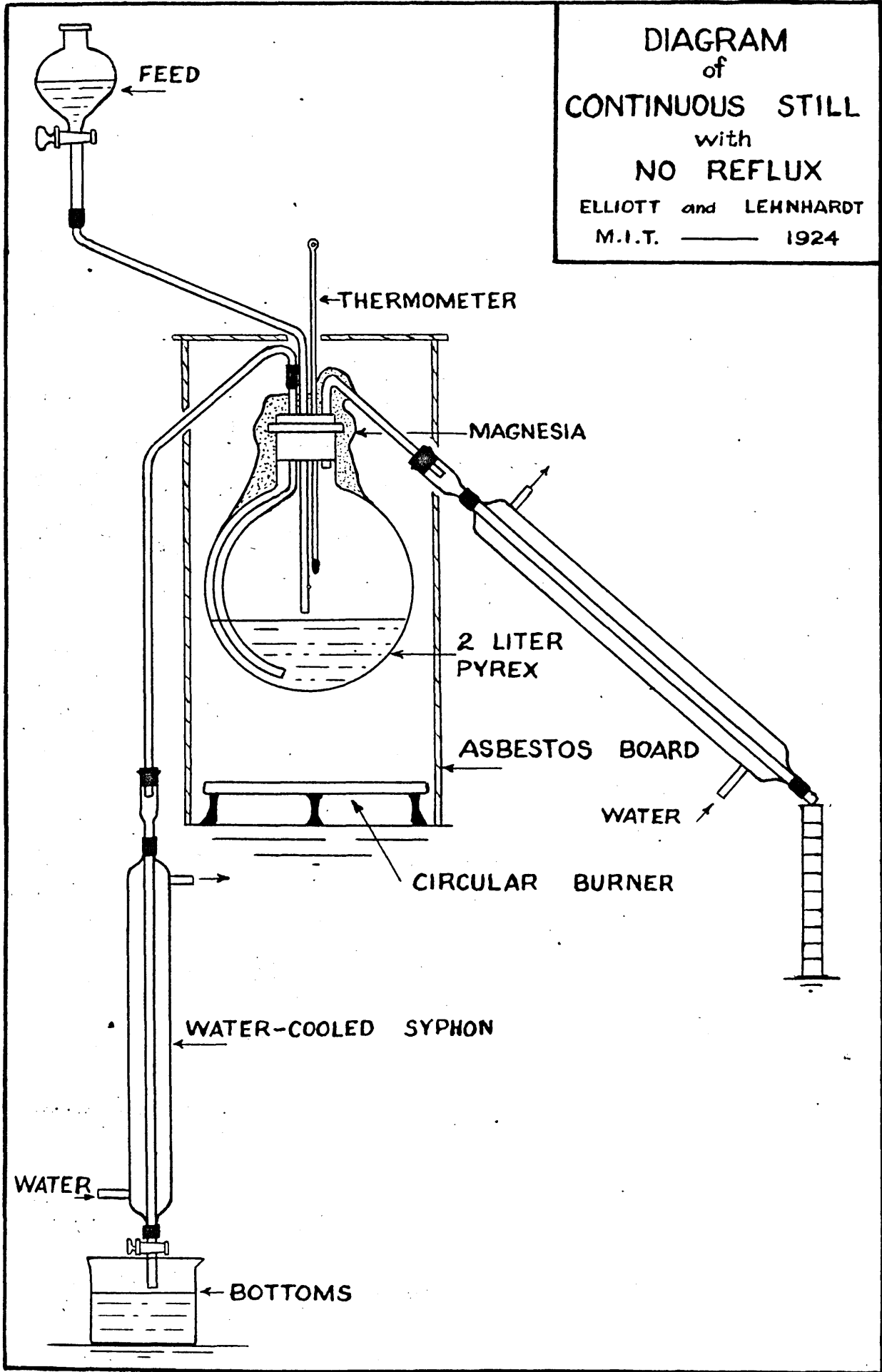
The bottoms were drawn off continuously, at a constant rate, by means of a siphon extending from the bottom of the flask. As shown in the accompanying diagram the bottoms were cooled in a condenser before they were drawn from the apparatus.

The vapor leaving the still was led into a 30" condenser and the resultant distillate was measured in a graduate.

A thermometer was inserted at the top of the flask. This thermometer was not shielded and it is very probable that the temperatures read from it are somewhat erroneous due to radiation from the flame of the gas burner beneath the flask, and from the insulat-

DIAGRAM
of
CONTINUOUS STILL
with
NO REFLUX

ELLIOTT and LEHNHARDT
M.I.T. ——— 1924

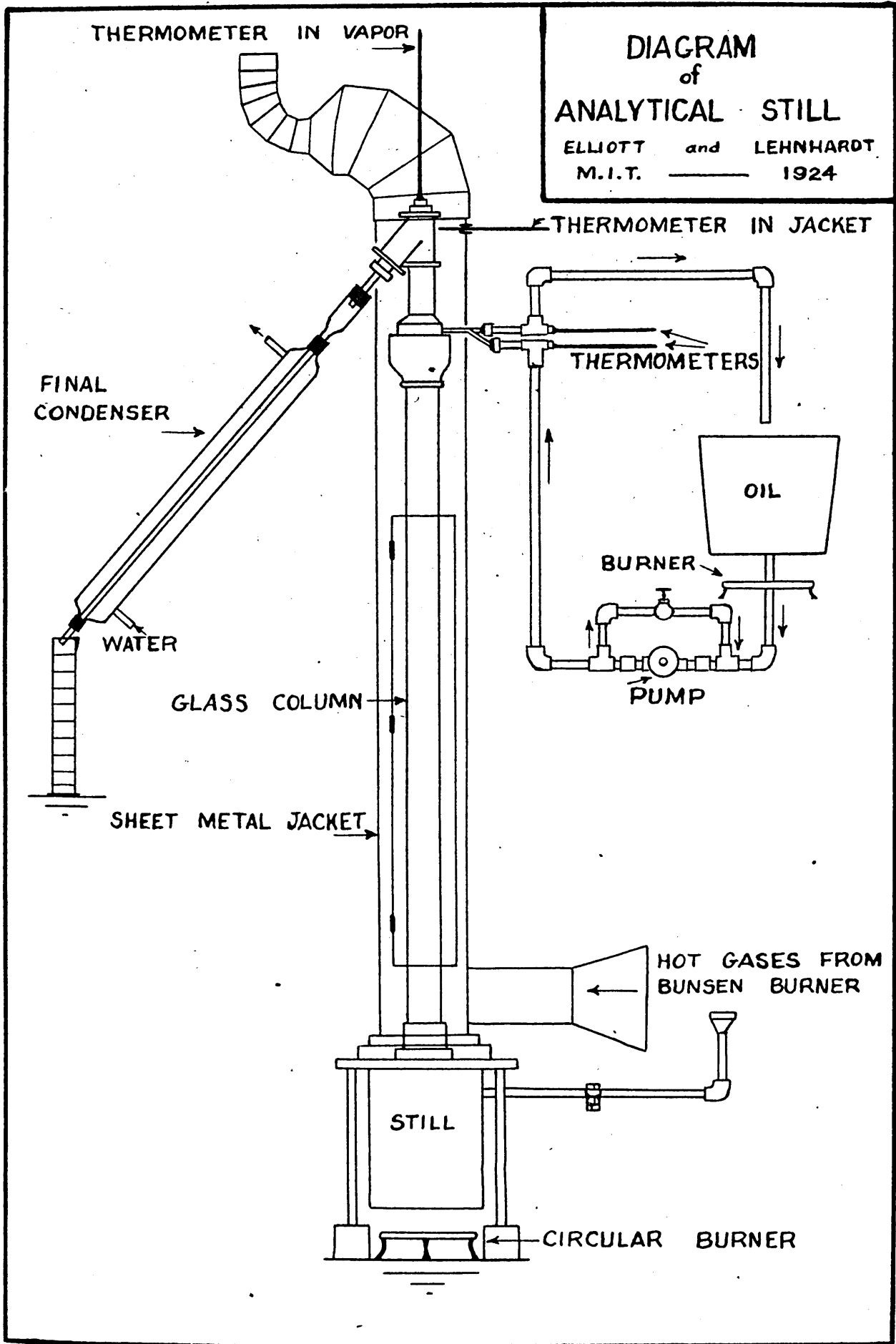


-ing boards which were at a temperature higher than that of the still.

(c) The analytical still

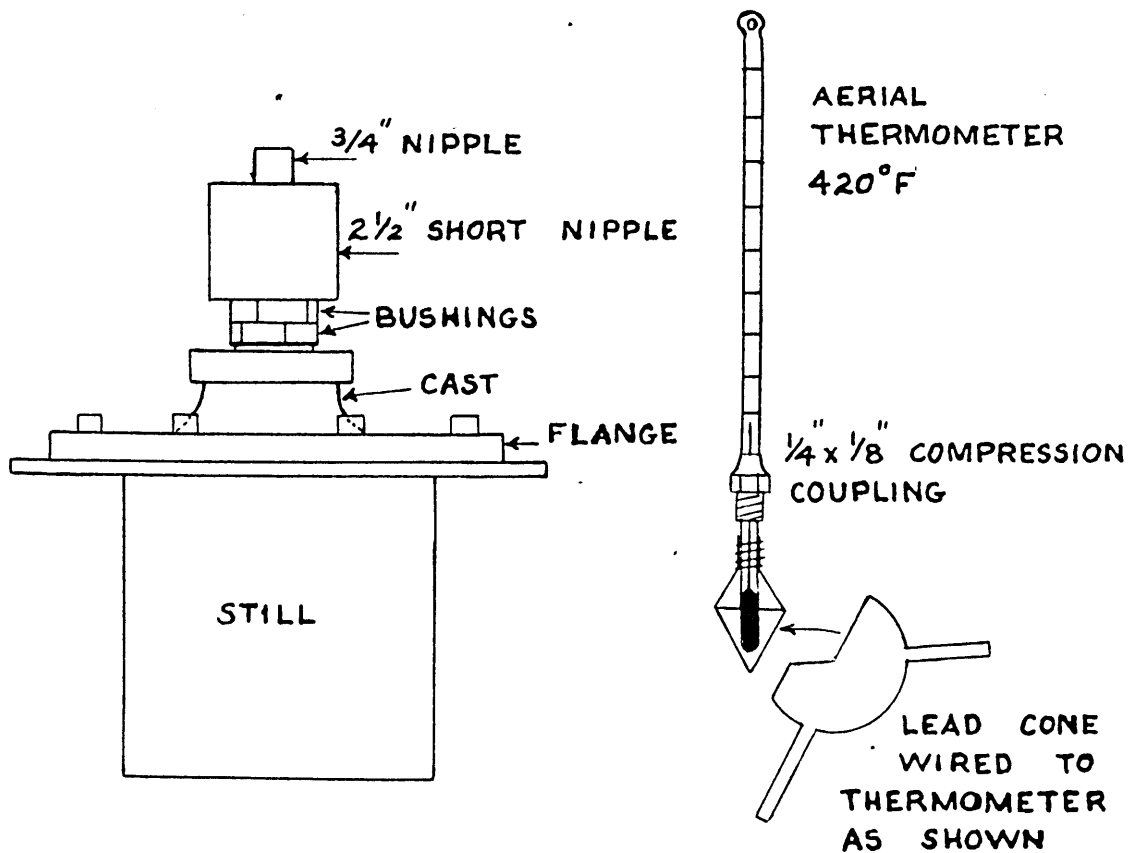
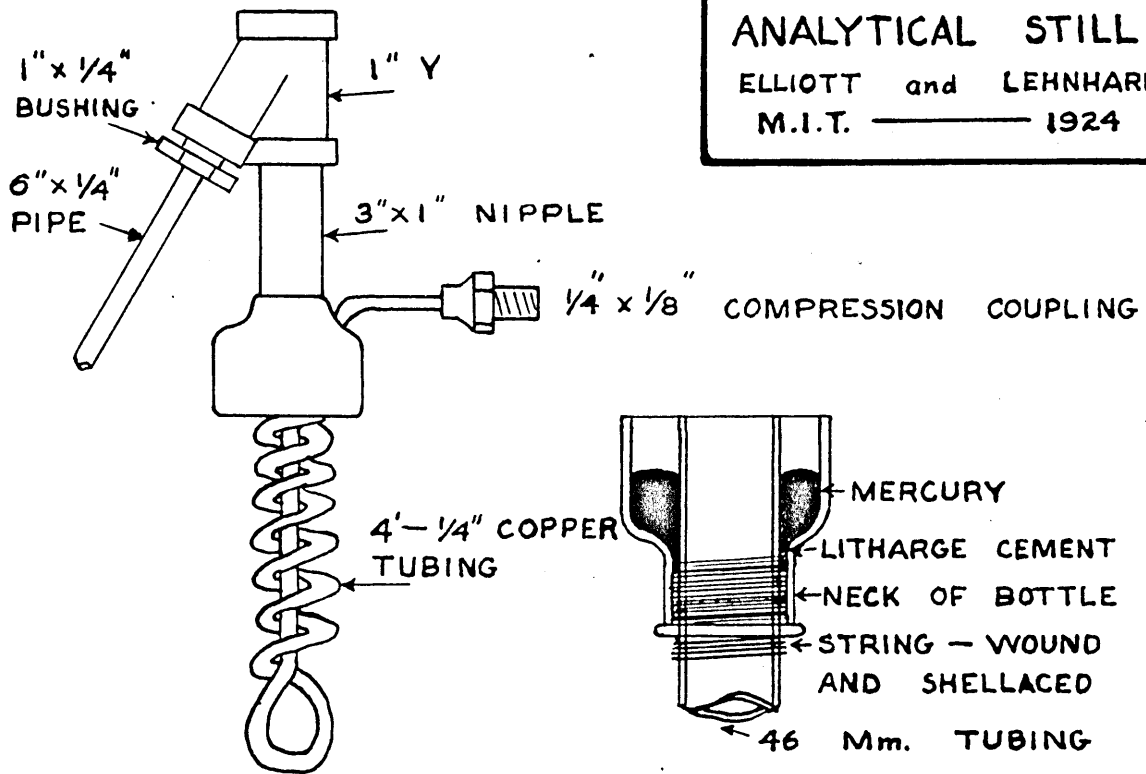
The still used for the determination of the true boiling point curves was constructed by Mackey (M.I.T. Thesis 1924) and was very similar in design to that recommended by W.T. Davis. A diagram of the apparatus is shown in this report.

This apparatus consisted essentially of a 1.5 gallon still, 40" still head with 28 inches of carbon nipple packing, a partial condenser to secure proper amount of reflux, and a final condenser. The partial condenser consisted of about four feet of $\frac{1}{4}$ " copper tubing in the form of a coil. A metal cone was fixed below the thermometer which gave the temperature of the vapor going to the final condenser, to shield it from radiation to the cooling coil. Radiation from the column was prevented by surrounding it with a flue gas jacketing chimney.



DETAILS
of
ANALYTICAL STILL

ELLIOTT and LEHNHARDT
M.I.T. ——— 1924



(3) Procedure

Continuous still, using reflux

Before a run was started the feed reservoir was filled with gasoline. This could be done either by pouring the gasoline directly into the tank thru a funnel or by means of compressed air as shown in the diagram of the apparatus. The latter method is much more convenient but it takes considerably more time. This method has the further disadvantage that it contains an element of danger. Glass bottles will withstand only small pressures, and if the air should happen to be turned on too fully there is a possibility that the bottle will break before the stopper is blown out. In this case there is not only the danger of the flying glass but also the possibility of a serious fire. Altho this method of elevating liquids is very convenient it must be used with discretion as serious accidents have resulted from its use.

After the feed reservoir had been filled, the valve beneath it was opened to allow the gasoline to flow into the constant head device. Any desired level of liquid was maintained in the head box by raising or lowering the overflow pipe. The valve mentioned above was so adjusted that the overflow from the head box amounted to a steady drip. As the liquor level in the feed reservoir decreased it

was necessary to open the valve somewhat more.

The gasoline flowed thru the orifice beneath the head box and then entered the column. After a small amount had reached the still the gas burner beneath the still was lighted and fully turned on in order that the still and column might be brought up to heat as soon as possible. When the temperature at the top of the column had reached approximately 150 F. the oil circulating pump was started if the desired reflux was greater than that furnished by the cold feed. If a large reflux was desired the oil was not heated, but in order to obtain a somewhat smaller reflux it was necessary to heat the oil. The amount of heating was controlled by adjusting the gas burner beneath the oil reservoir.

It was very difficult to bring the still to constant operating conditions. This was particularly true if it was desired to operate at predetermined conditions. In general it was found that the best method was to adjust the burner beneath the still and allow it to remain unchanged. The oil rate was then adjusted in such a manner that the product of its rate by the temperature difference at the inlet and outlet of the cooling coil was constant. The temperature at the top of the column would then either rise or fall until it would eventually become constant. The distillate rate would then be constant also.

After the column had been operating uniformly for approximately an hour a run was started. During the run the distillation rate was watched continually. If it very suddenly decreased the cause was usually plugging of the orifice in the feeding device. In such a case the pipe containing the orifice was struck a sharp blow with a hammer. This usually removed the obstruction and allowed the rate to almost immediately return to normal. If the distillation rate showed a tendency to either decrease or increase, the reflux remaining unchanged, it was usually due to a change in the amount of heat under the still resulting from a fluctuation of gas pressure. In this case a very slight adjustment of the burner was made.

In case it was desired to operate under predetermined conditions it was necessary to adjust the heat under the still and the temperature and rate of the cooling oil until the desired conditions were obtained. It was almost impossible to do this with the apparatus used in this investigation.

In order to prevent radiation and consequent condensation in the column it was necessary to maintain the jacket surrounding the column at very nearly the same temperature as the column itself. This was accomplished quite easily by adjusting the amount of hot flue gas entering

the bottom of the jacket from the space surrounding the still. In case the heat supply from this source was insufficient it was augmented by inserting a Bunsen burner in the sidearm provided for that purpose.

The distillate was caught and measured in two burettes connected by a Y in such a manner that one could be emptied while the other was being filled. With the use of a watch the distillation rate could be determined quite accurately. These rates were very valuable as an indication of the uniformity of operation. The total volume and the temperature of the distillate collected during a run was recorded. Its specific gravity was measured at 60 F. with a hydrometer.

The bottoms were allowed to overflow from the still thru the overflow pipe provided for that purpose. Ordinarily this worked quite smoothly but on a few occasions pressure fluctuations in the still caused it to operate very erratically. The exact cause of these fluctuations was not discovered. When they occurred the bottoms were forced from the still at a very rapid rate and the continuity of the still operation was disrupted. During the run occasional rates of flow were taken on the bottoms to see that it was at least reasonably constant. There were minor fluctuations but over a period of

a few minutes the rate was ordinarily quite constant.

McFarland and McGrath in operating this still determined the rate of feed when the still was cold and assumed that this rate would be the same when the still was in operation. They measured the distillate collected each minute, subtracted it from the volume of feed for the same length of time, and then drew off the difference as bottoms. This method of determining the rate of feed was checked up and found to be erroneous due to the fact that the pressure in the column is not the same during the run as when the still is not in operation.

The total volume and the temperature of the bottoms from a run were measured and recorded. The specific gravity at 60 F. was determined with a hydrometer. The length of a run was determined by the bottoms rate since it was necessary to have at least two liters of bottoms for the determination of the true boiling point curve.

At intervals of five minutes during a run the column temperature; the temperature of the inlet and outlet oil; and the oil rate, were recorded.

The oil rate was determined by measuring, in a graduate, the oil returned from the cooling coil during a period of one minute. Some difficulty was encountered in maintaining this rate constant, on account of the fact that the pump was belt driven.

Oil on the belt caused it to slip a great deal, especially when a high oil rate was being used.

Considerable technique which can be obtained only by actual operation of the still must be acquired before the column can be operated altogether successfully.

Certain structural changes should be made before this apparatus is used for further work. These are discussed under "Recommendations".

(3) Procedure

Continuous still, using no reflux.

The operation of this still was quite simple. Before a run was started the size of the cut to be made was decided. About 400cc of feed was then run into the still and the burner under the still was lighted. A definite amount of feed was then measured in a graduate and added to the separatory funnel each minute, the stopcock on the funnel being so adjusted that a minute was required to empty it. This gave a practically uniform rate of feed. The bottoms were drawn off thru the siphon at a rate sufficient to maintain a constant level in the flask. The heat under the still was carefully adjusted until the rate of distillation corresponding to the desired cut was obtained. After constant conditions had been established for approximately half an hour the run was started. During the run the rate of distillation was watched continually and any tendency toward a change was compensated by a very slight adjustment of the burner. The total time; total feed; total distillate; temperature at the top of the flask; and the density of feed, distillate, and bottoms at 60 F. were recorded.

The length of a run was determined by the time required to collect somewhat more than two liters of bottoms.

(3) Procedure

The analytical still

This still was a very unwieldly thing to charge. The top of the jacket had first to be removed, using great care not to break the thermometer which protruded thru it. The condenser was then disconnected from the stillhead. The stillhead, containing the partial condenser, was then lifted straight up until the cooling coil had been removed from the column. The column could then be removed from the jacket by lifting it until it was disconnected from the mercury seal at the top of the still, and then taking it out thru the door in the jacket. When this had been done the jacket was lifted high enough to clear the mercury seal at the top of the still, and the still was lifted from its position under the jacket. The mercury was removed from the seal and the still was turned up side down to remove any residue from it. Since all of the liquid remaining in the still could not be removed by this method it was necessary to insert a glass tube into the still and draw out the remaining residue.

A two liter charge of the gasoline, or bottoms, which it was desired to analyze was measured in a liter graduate and poured into the still. The reverse of the operations described in the previous paragraph was then carried out to restore

the apparatus to operating conditions. The gas burner beneath the still was then lighted and the still brought up to heat. The jacket temperature was then adjusted to approximately the temperature at which the first fraction was expected to come over. The oil circulating pump was started and so adjusted that when the column was operating a reflux ratio of 15:1 to 25:1 was maintained. As the column temperature increased it was necessary to heat the oil to prevent total condensation by the reflux condenser. The heat under the still was carefully adjusted until it was as high as could be maintained without causing the column to prime. In general, the distillation rate was maintained at from 1cc to 3.5cc per minute.

The volume of distillate was measured in a 50cc graduate. Readings of total distillate over; temperature at the top of the column; jacket temperature; temperature of the inlet and outlet oil; and the oil rate, expressed as seconds per 200cc, were taken at frequent intervals and as nearly simultaneously as possible.

The distillation of the bottoms was continued until the column temperature reached a point somewhat higher than the temperature on the true distillation curve of the feed corresponding with the same percent cut as that taken off in the

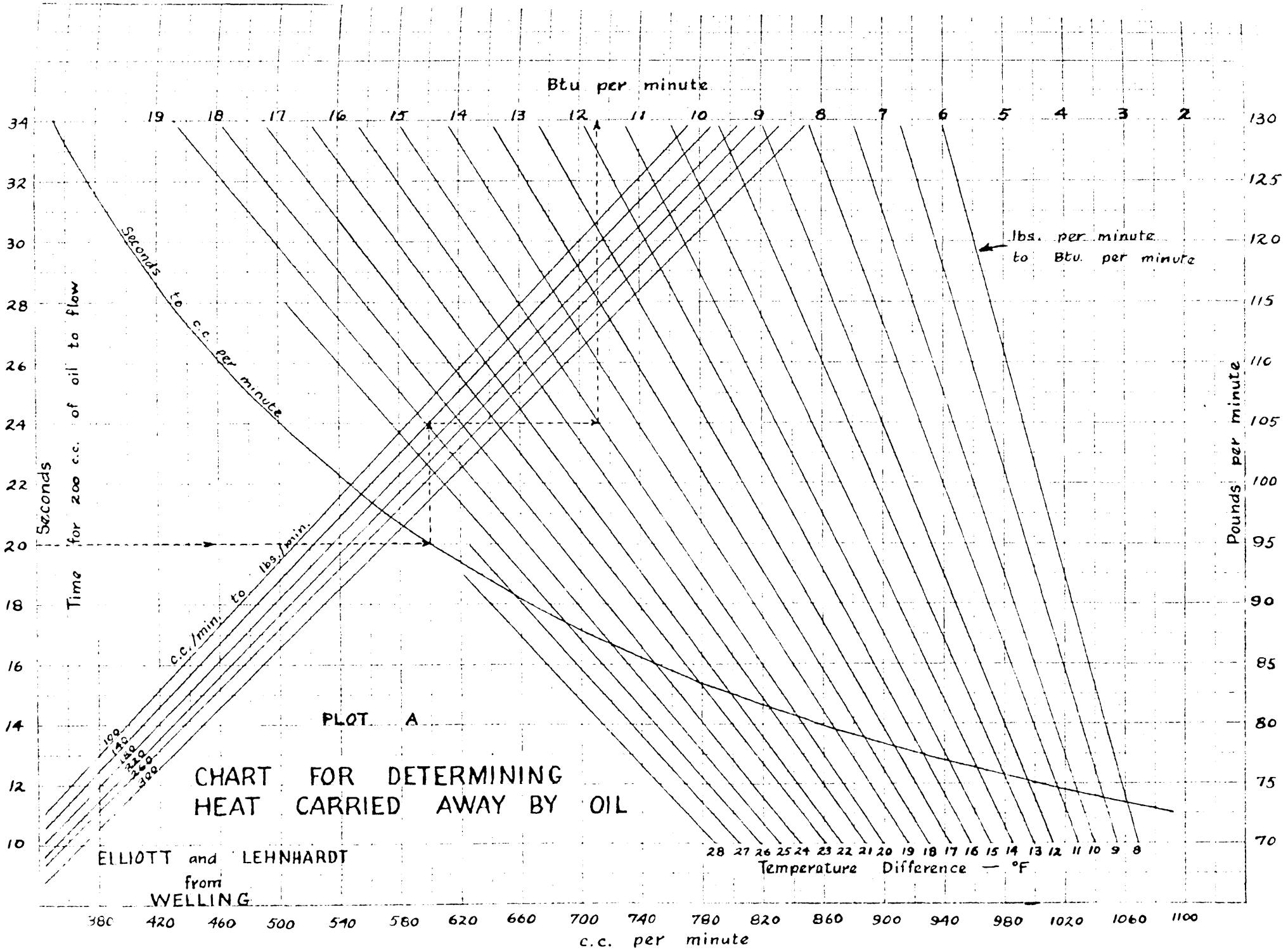
continuous still.

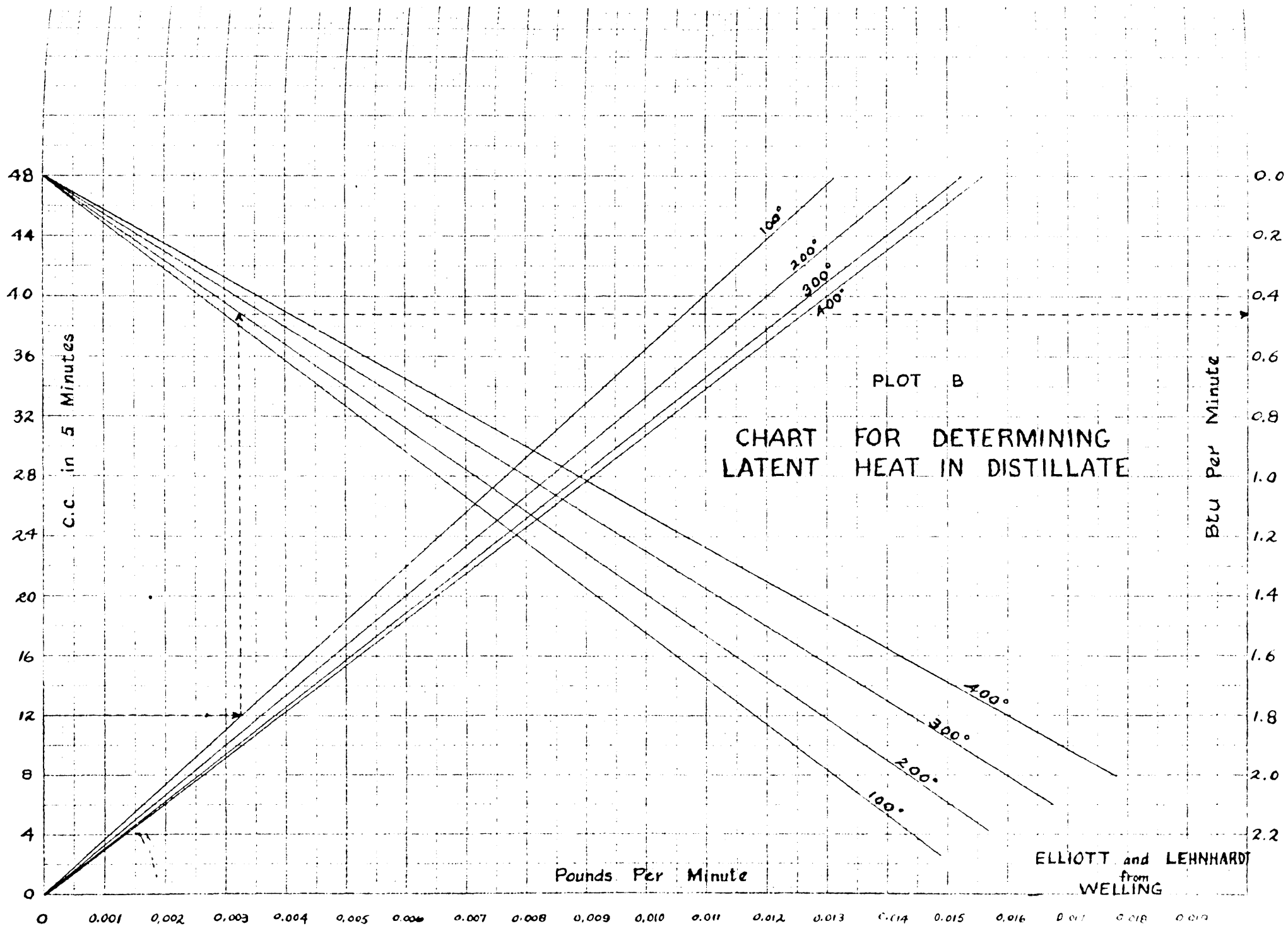
The reflux ratio was obtained from charts drawn by Welling, (M.I.T. Research Lab. of Appd. Chem.). With their aid, the reflux could be calculated in a very few minutes. Copies of these charts are contained in this report.

The method of using can best be explained by an example:

Consider a case where the time read for 200c.c. of oil to flow thru the cooling coil is 20 sec. and where the temperature of the inlet oil is 130 F and that of the exit oil is 150 F.; the rate of distillation being 12 c.c. in five minutes, and the column temperature 200 F. From chart A, the number of c.c. of oil per minute corresponding to 200c.c. per 20 sec. is 600. The number of pounds of oil per minute is read from the line corresponding to the average temperature, and in this case is 1.05# /min. For a temperature difference of 20 F., this gives 11.7 B.T.U. per min. carried away by the cooling oil. Turning now to chart B, the number of pounds of distillate per minute for a rate of 12c.c. in five minutes, with a column temperature of 200 F. is .00323. The corresponding number of B.T.U.'s, required to vaporize the distillate, is 0.46. The reflux ratio is then 11.7 divided by 0.46 or 25.4.

The data for the construction of these curves was obtained from "The Physical Properties of the Paraffin Hydrocarbons" (R.E. Wilson, W.H. Bahlke. Journal of Ind. and Eng. Chem. Feb. 1924). The specific heat of the oil and of the distillate was taken as 0.5.





IV R E S U L T S :

(1) Presentation of Results

Since this problem is one on which very little previous work has been done there was considerable doubt as to the best method of calculating the results.

The original plan was to determine the per cent cut made in each run and read, from the distillation curve of the feed, the temperature which would have been reached had the same cut been made under conditions complete rectification. Then, from the distillation curve of the bottoms, the per cent of material distilling below this temperature could be determined.

This per cent of bottoms distilling below the temperature mentioned above was plotted against B.T.U. per pounds of bottoms, required to give the reflux used in the run on the continuous still. Plot I shows the curve obtained by plotting Runs 5,10,11,12, and 13 in the above manner. These particular Runs were used because it is essential that, in order to be comparable, the cuts be very nearly equal. The initial point on this curve was interpolated from Plot V which will be discussed later.

It will be noted that one point appears to be much too low. This is undoubtedly due to insufficient reflux during the determination of the distillation curve of the bottoms, since this curve for Run 13 is much closer to the Engler curve than it is in the case of the other runs.

Altho this method of determining the degree of separation does undoubtedly give an indication of the effect of reflux on separation, it is impossible to say that it is an exact measure of the separation. The difficulty encountered is in the definition of what is meant by degree of separation. In the case of a binary mixture, it is perfectly possible to determine this quantity since, if the bottoms contained, for example, 20% of the lower boiling constituent it would be obvious that the degree of separation was 80%. However, in dealing with a mixture such as herein encountered, it is necessary to arbitrarily define what is meant by degree of separation. If the lights left in the bottoms are defined as the amount therein which distills below the temperature which would have been attained had the same cut been made under conditions of complete rectification, then the above method of calculation is permissible. It is obvious that, on the basis of feed, the per cent lights in the bottoms must equal the per cent of heavy ends in the overhead or distillate. It must be realized that it is impossible to obtain complete separation in a tower in which the feed is introduced at the top of the column, since in such a column there is no section provided for the purpose of stripping the heavy ends from the distillate.

It was found to^{be} impossible to obtain points on the left hand portion of this curve. The lowest value of heat was obtained using only feed as a means of producing reflux. It was suggested that a still lower re-

flux could be obtained by preheating the feed. However, had this been done, some of the more volatile portions of the feed would have been vaporized, thus changing the composition of the gasoline actually introduced as feed, and thereby making the results incomparable. (See also McCready M.I.T. Thesis 1924)

Plot II shows the effect of the per cent cut vs. lights left in the bottoms. The straight line is for continuous distillation with zero reflux whereas the curve shows the relationship when operating with reflux. In order to be comparable the amount of reflux should either be constant or have no effect on either of the variables plotted. It so happens that the reflux was such that, within the limits used, the degree of separation, as determined by the method previously discussed, did not vary greatly with change in reflux. The points on this curve are therefore comparable. The curve is hyperbolic in nature. Its equation may be derived as follows:

EQUATION FOR THE RELATIONSHIP BETWEEN WEIGHT PER CENT OF
 BOTTOMS vs. PER CENT CUT FOR A HIGH REFLUX.

Plot II

Call:

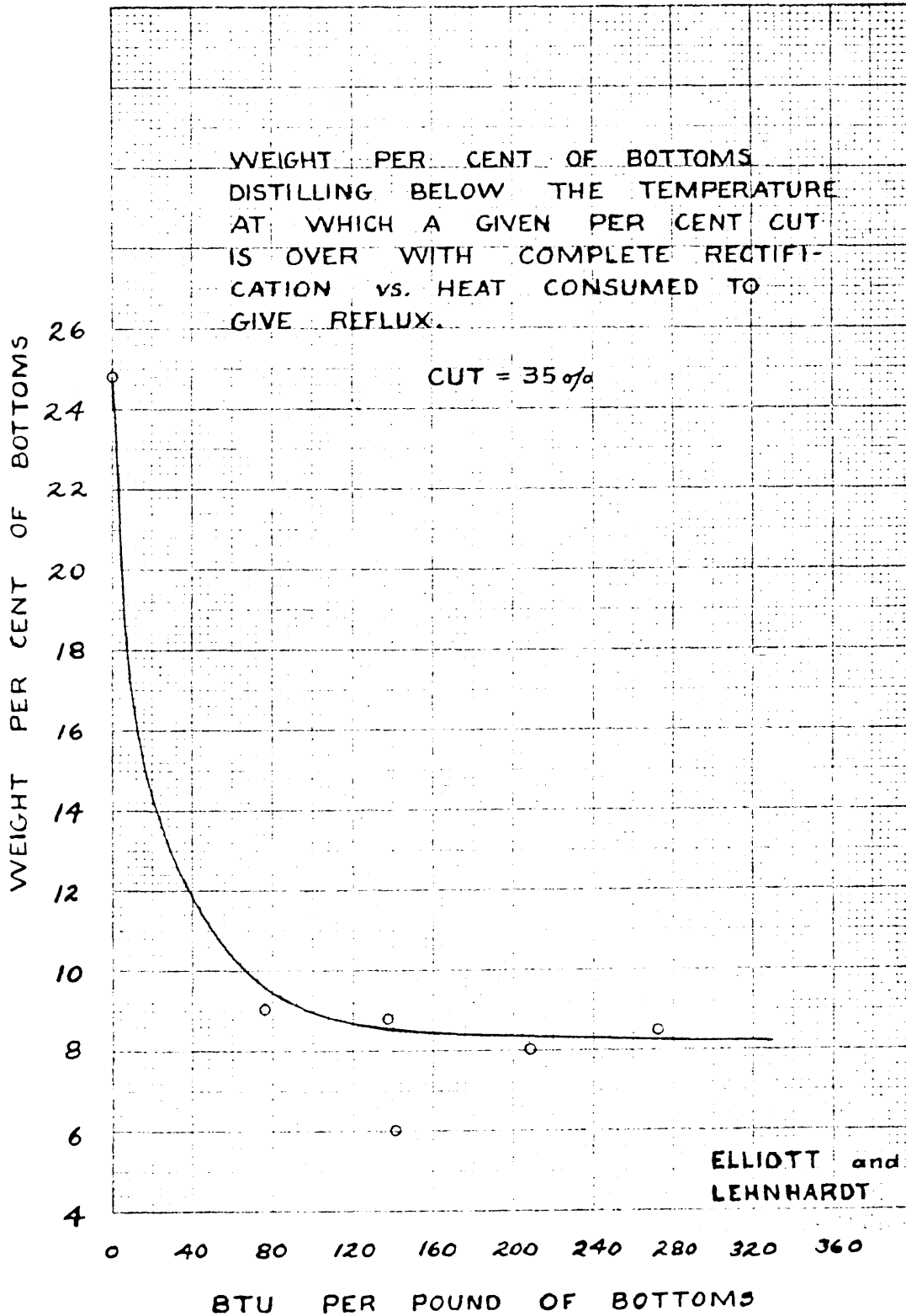
x Per cent cut.

y Weight per cent of bottoms distilling below the
 temperature at which the given per cent cut is o
 over with complete rectification.

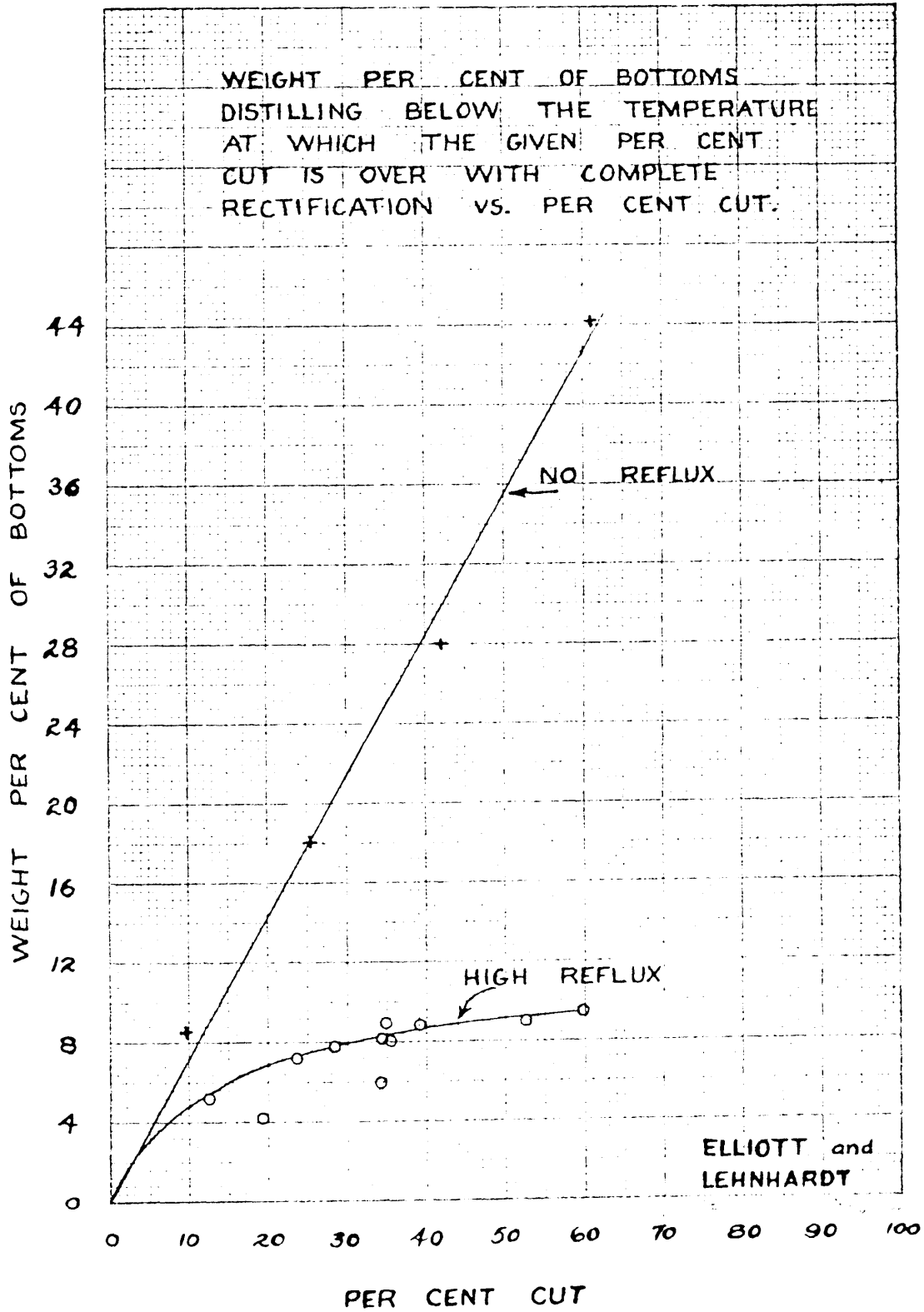
A Plot of $\frac{x}{y}$ vs. x has been made and is found to be a
 straight line. (Plot IIa). This line was drawn using
 the method of least squares, omitting the two points
 which are apparently in error, judging from a compari-
 son of the true b. pt. curve of the bottoms with the
 Engler for these runs.

Run	x	y	$\frac{x}{y} = y_1$	
1	28.2	7.8	3.62	The equation of this straight line is $y_1 = 1.22 + 0.085 x$ from which we get
2	23.7	7.2	3.29	
3	12.7	5.2	2.44	
4	19.3	4.2	4.6	
5	39.0	8.8	4.44	$x = \frac{1.22}{\frac{1}{y} - 0.085}$
6	52.6	9.0	58.5	
7	59.8	9.4	6.36	
10	35.5	8.0	4.44	
11	34.5	8.5	4.06	
12	35.0	9.0	3.89	
13	34.6	6.0	5.76	

PLOT I



PLOT II

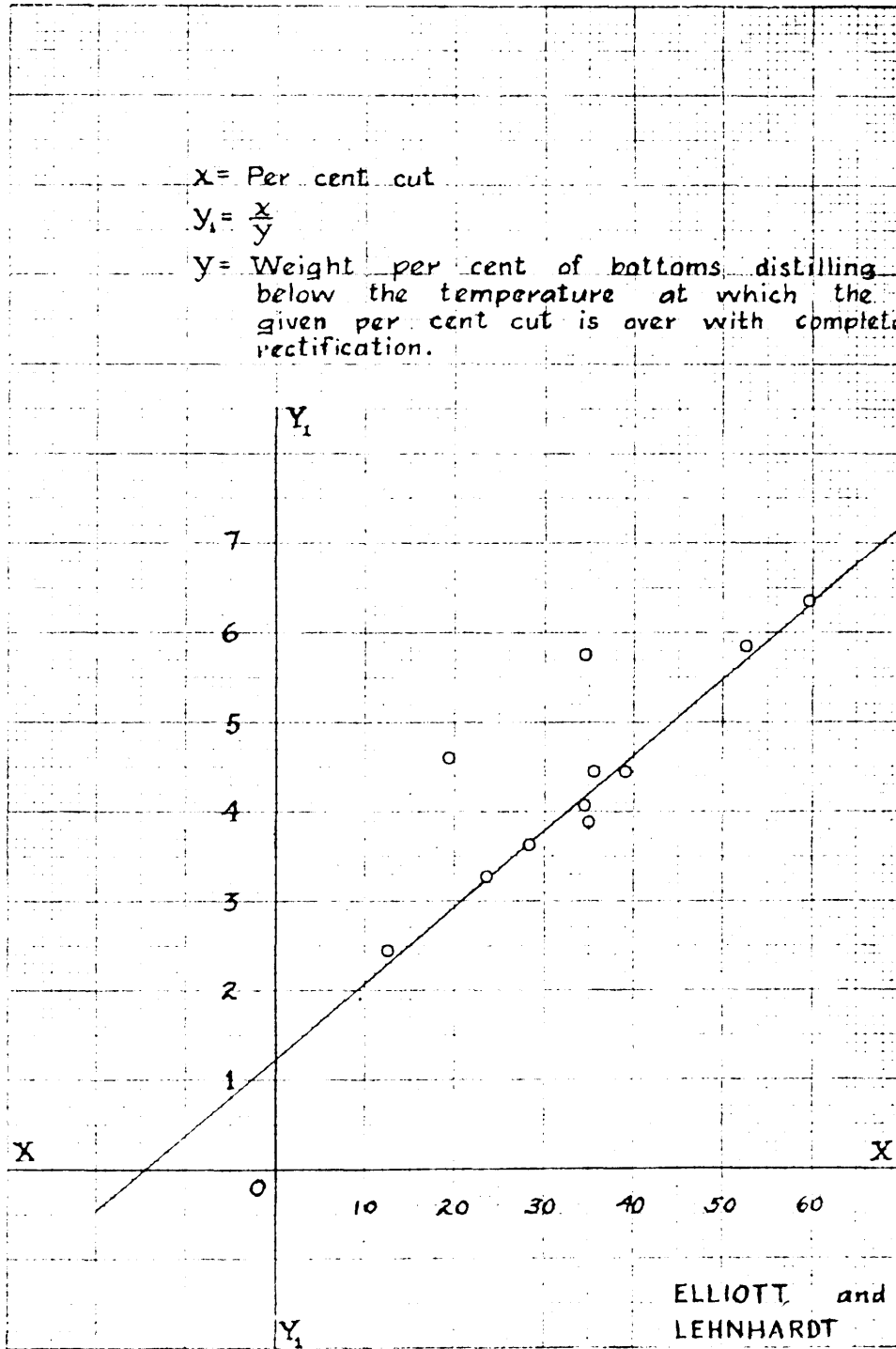


PLOT IIa

x = Per cent cut

$$y_1 = \frac{x}{y}$$

y = Weight per cent of bottoms distilling below the temperature at which the given per cent cut is over with complete rectification.



Perhaps a better method of calculation is that used in the determination of Plot III. In this case the initial boiling point of the bottoms was considered as the determining factor in calculation^{na} the per cent of lights in the bottoms. From the true boiling point curve of the feed the per cent which should distill below the initial boiling point of the bottoms was determined. This volume per cent was then calculated to weight per cent, and subtracted from the per cent cut. The basis of this method is as follows. The per cent lights in the feed is arbitrarily defined as being equal to the per cent cut. If when the bottoms are subjected to complete rectification it is found that the initial boiling point has been raised, it follows that the amount of feed distilling between the initial boiling point of the feed and the initial boiling point of the bottoms has been removed. Then, since the per cent lights in the feed was defined as equal to the per cent cut, the per cent of lights in the bottoms must be equal to the difference between the cut and the amount distilling between the temperatures mentioned above. It is obvious, from a material balance, that regardless of the degree of separation obtained, the pounds of lights in the bottoms per pound of feed, must be equal to the pounds of heavy in the overhead per pound of feed.

The initial point on this curve was interpolated from Plot V.

The curve obtained by this method of calculation is hyperbolic in nature. Its equation may be derived as follows:

EQUATION FOR THE RELATIONSHIP BETWEEN LIGHTS IN BOTTOMS
AND HEAT REQUIRED.

Plot III

Call:

x Btu. per lb. of bottoms

y Pounds of lights in bottoms per 100 lbs. of feed.

Assuming a hyperbolic relationship between x and y, the following equation may be set up.

$$(x+a)(y-b) - c \quad \text{where } a, b, \text{ and } c \text{ are constants.}$$

From the experimental data it is known that when $x = 0$,
 $y = 33.5$

Now,
$$y = \frac{c}{x-a} + b$$

And
$$33.5 = \frac{c}{a} + b$$

Subtracting,

$$33.5 - y = \frac{c}{a} - \frac{c}{x+a}$$

$$\frac{x}{33.5-y} = \frac{a}{c} (x+a)$$

Which shows the left hand member of the equation to be a linear function of x. The accompanying plot of $\frac{x}{33.5-y}$ vs. x gives a straight line (Plot IIIa) showing the assumption of a hyperbolic relationship between x and y to be justified.

Run	y	x	$\frac{x}{33.5-y} = y,$
10	19.2	209.4	14.65
11	19.1	272.7	18.94
12	22.3	75.9	6.78
13	20.6	140.8	10.91
--	33.5	0	-----

From the straight line plot, when $x = 0$, $y_1 = 2.3$ and when $x = 280$, $y_1 = 19.2$.

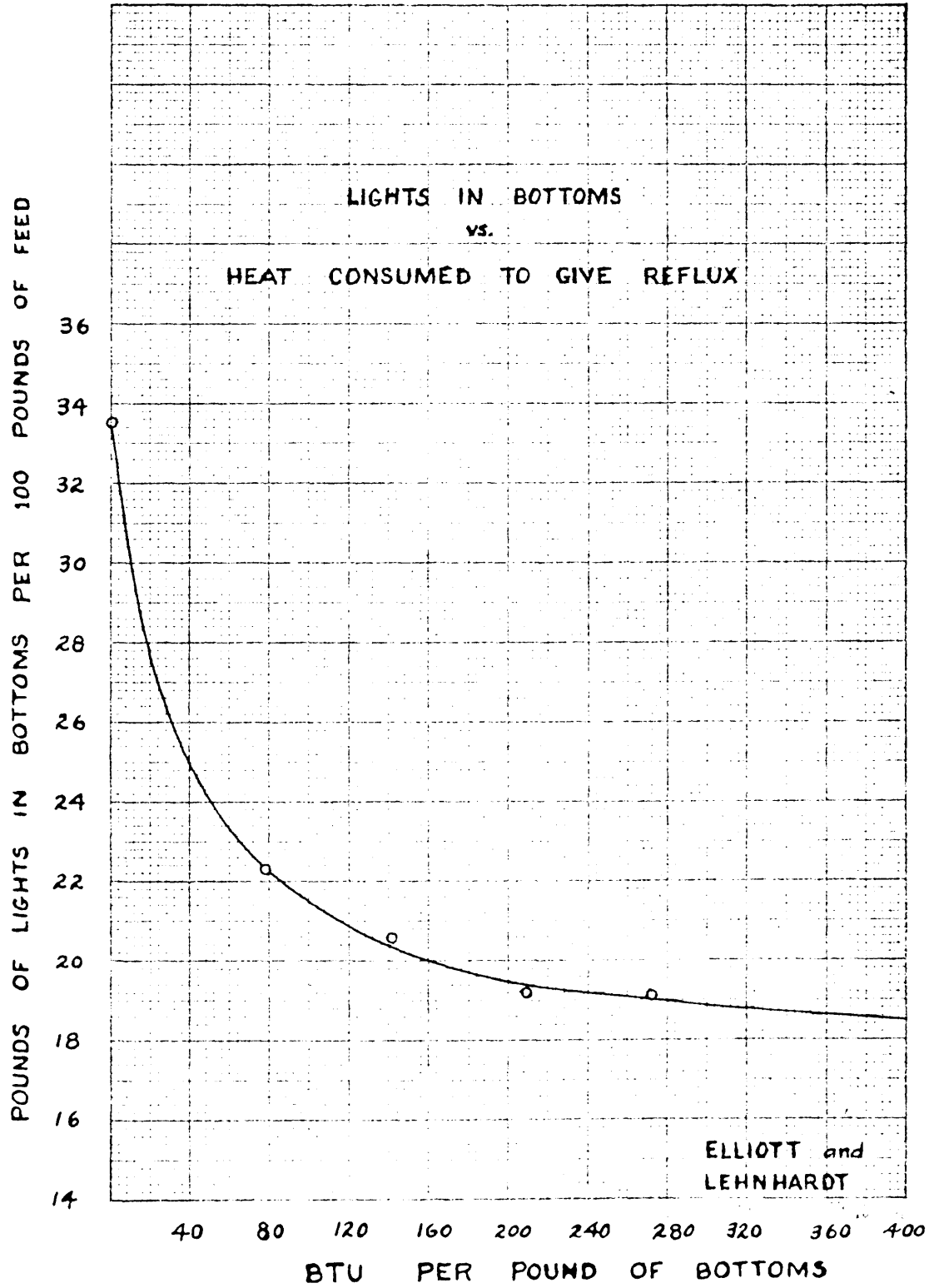
The slope of the line, $\frac{a}{c}$, is then $\frac{19.2-2.3}{280} = 0.0604$

When $x = 0$, $\frac{a}{c} = 2.3$ Then $a = \frac{2.3}{0.0604} = 38$

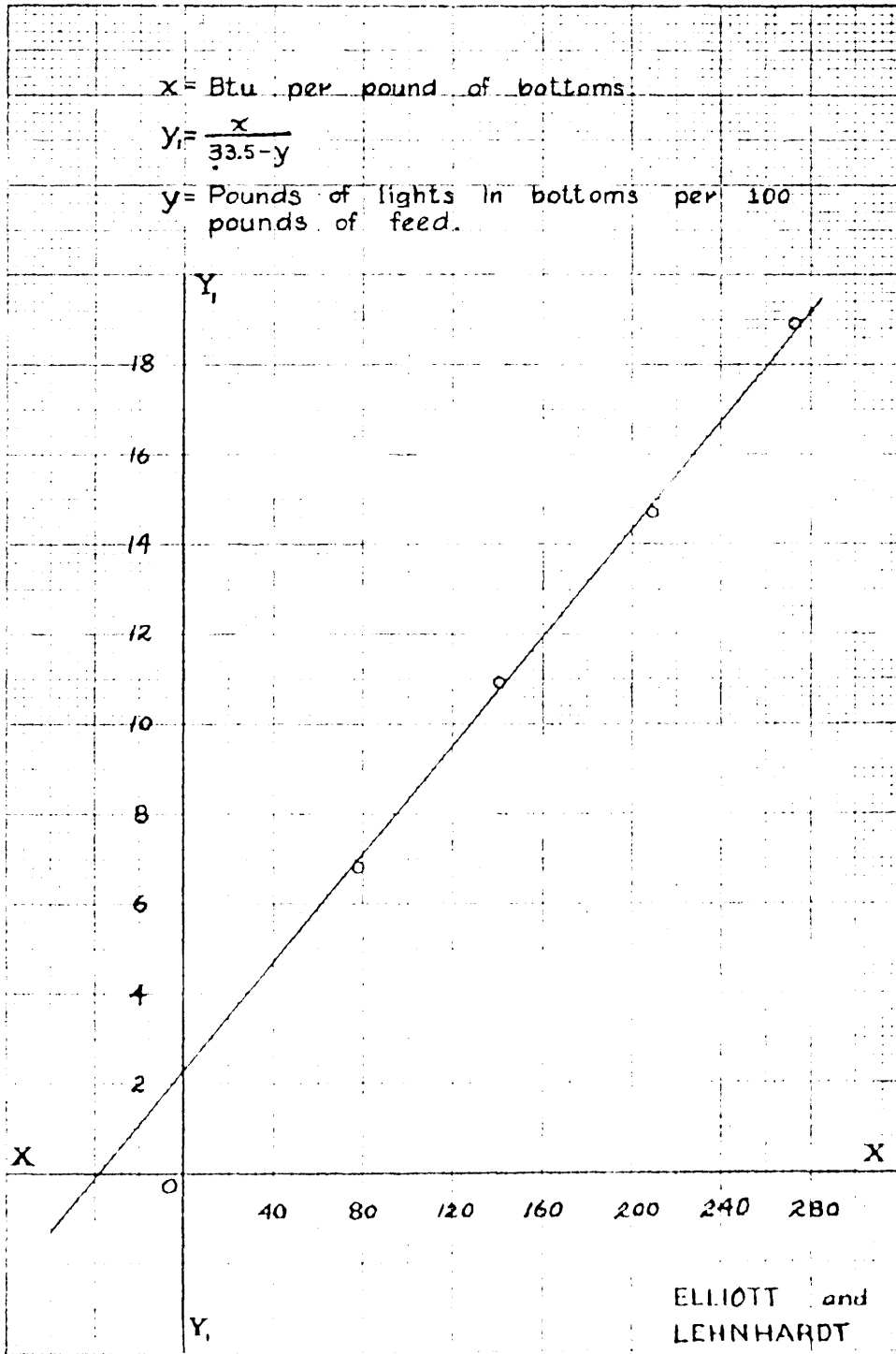
Then $c = 630$ And $b = 16.9$

$$y = \frac{630}{x+38} + 16.9$$

PLOT III



PLOT III a



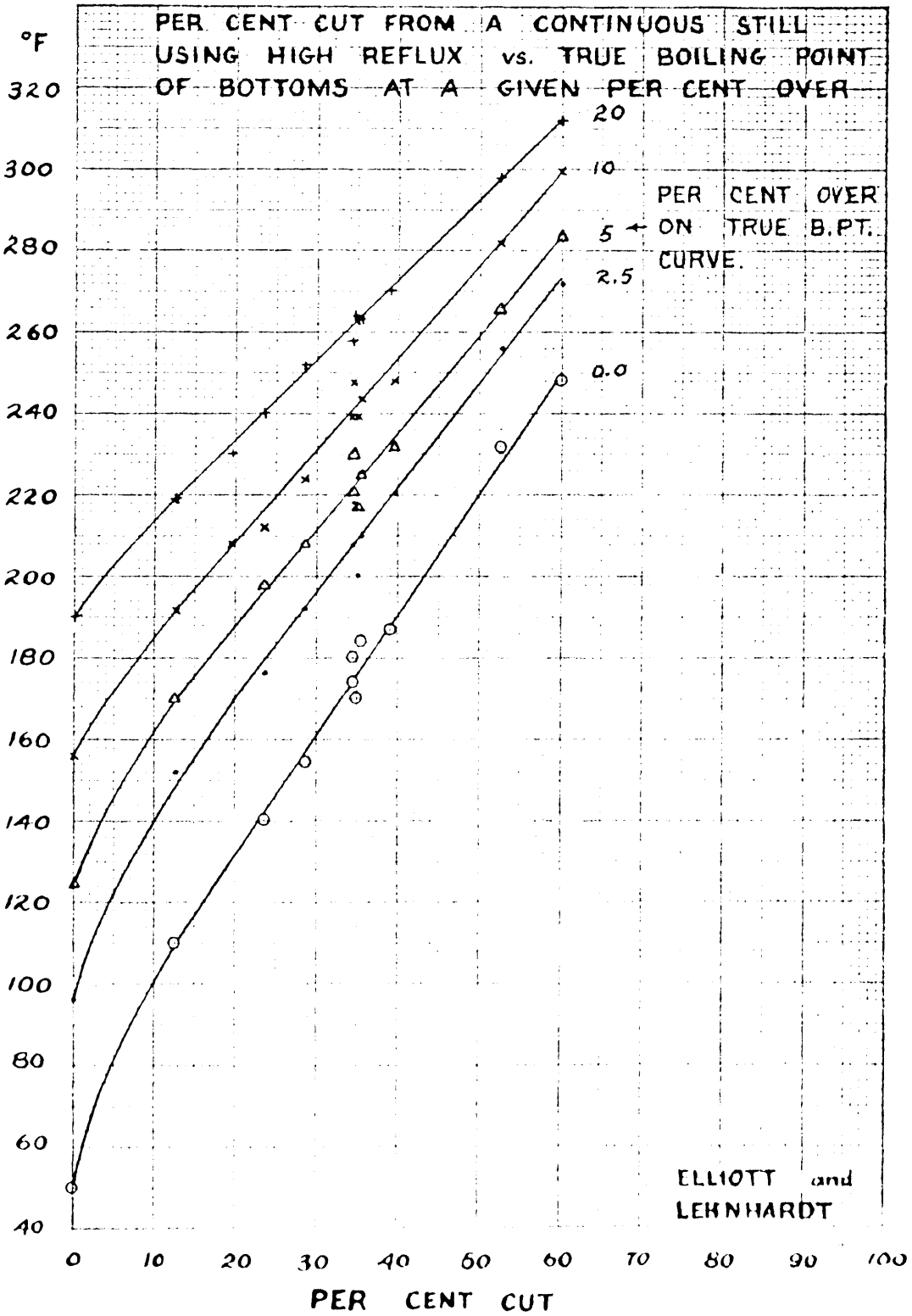
A comparison of Plots IV and V shows very clearly the effect of rectification. In these plots the per cent cut is plotted against the temperature at which a given per cent is over in the distillation of the bottoms. In general these points, for different reflux ratios, would not fall on the curves as they do. But due to the fact that most of the runs were made under conditions where a change in reflux ratio made only a slight difference in the degree of separation the points fall reasonably close to the curves. It is interesting to note the very low initial points obtained when no reflux was used.

Plot VI was constructed by reading the initial points for various cuts from Plots IV and V and from them determining the amount of lights in the bottoms, by means of the "initial point method" previously discussed. The similarity between this plot and Plot II should be noted.

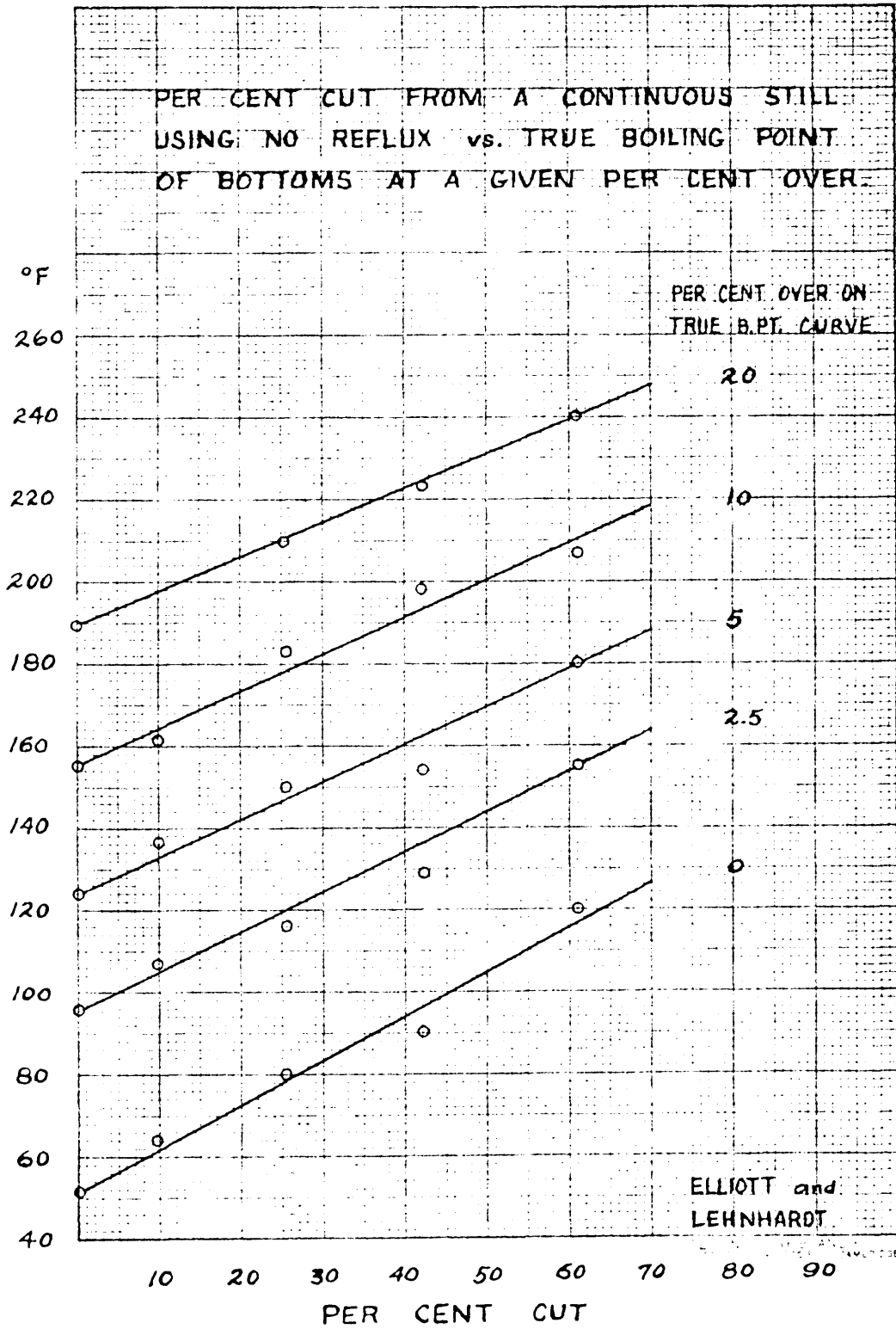
Plot VII shows the effect of rectification. Had complete rectification taken place, the center curve would have been identical with the upper curve. These curves show that in the case of Run 12 no bottoms boiling above 290 were taken over in the overhead. The lower curve shows that when no reflux was used a large amount of high boiling material was carried over in the overhead.

Plot VIII was determined directly from Plot VII. It shows the ratio of the per cent over on the true boiling point curve of the bottoms from a continuous still using no reflux, to the per cent over at the same temperature on the true boiling point curve of the bottoms from a contin-

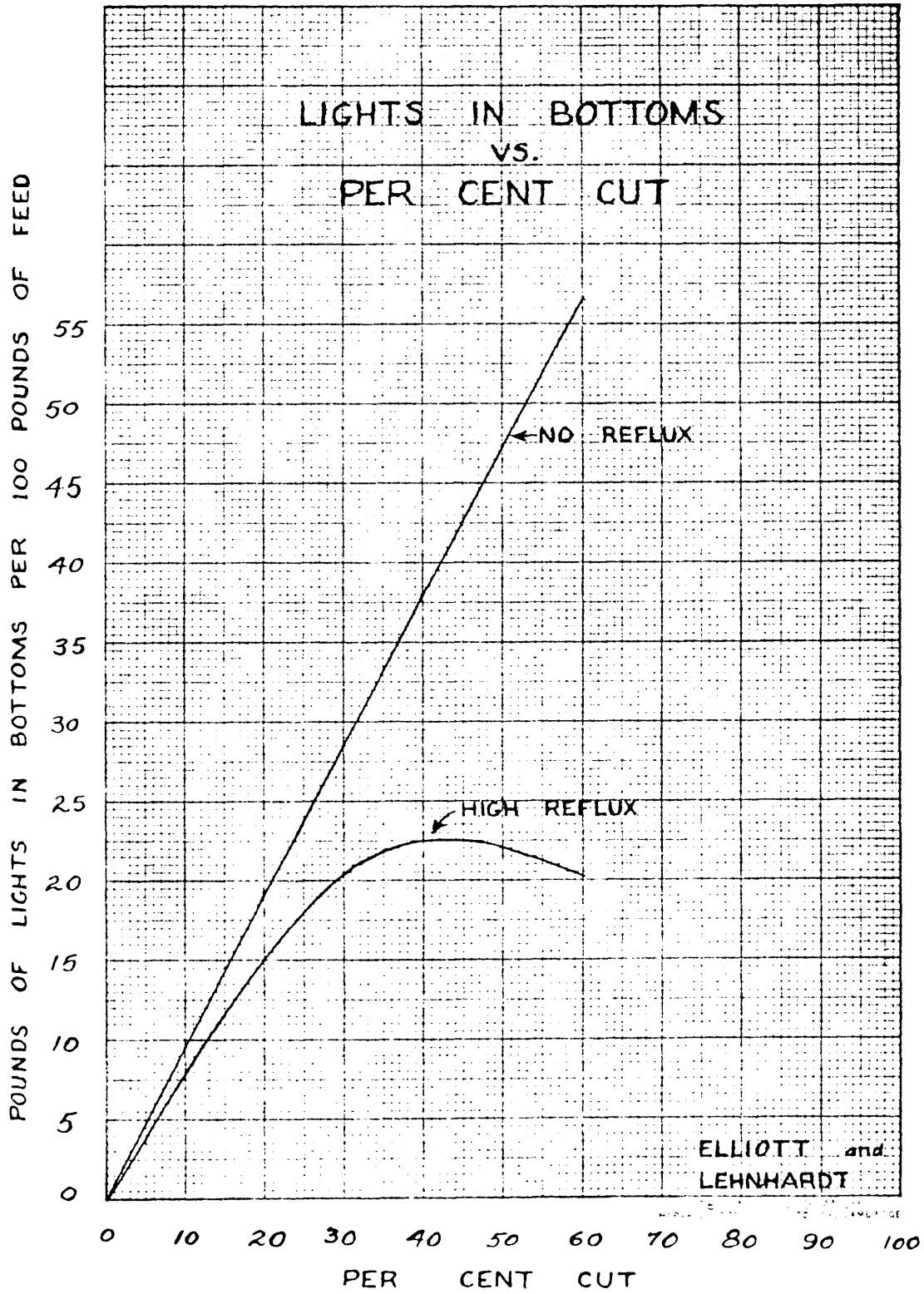
PLOT IV



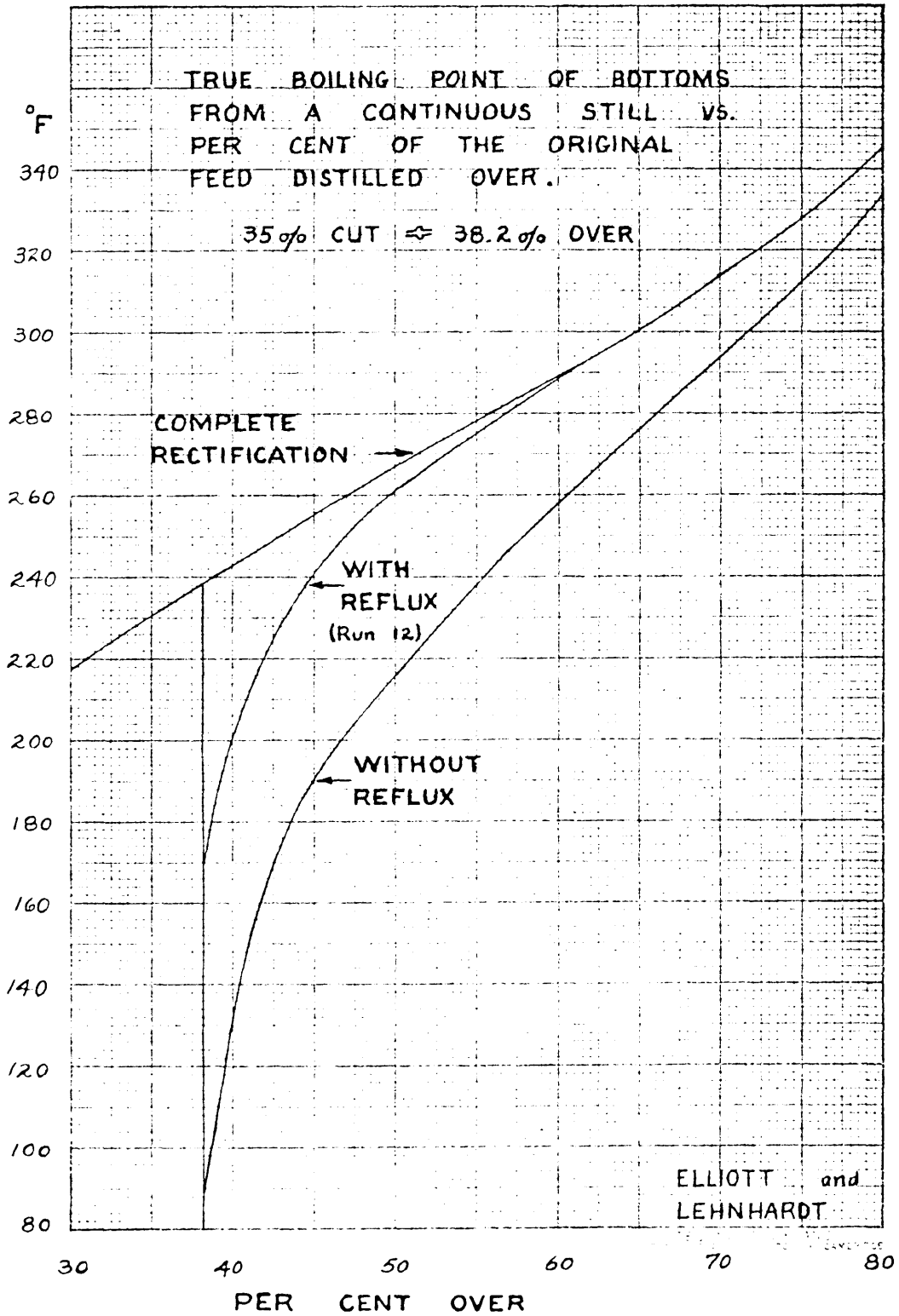
PLOT V



PLOT VI



PLOT VII

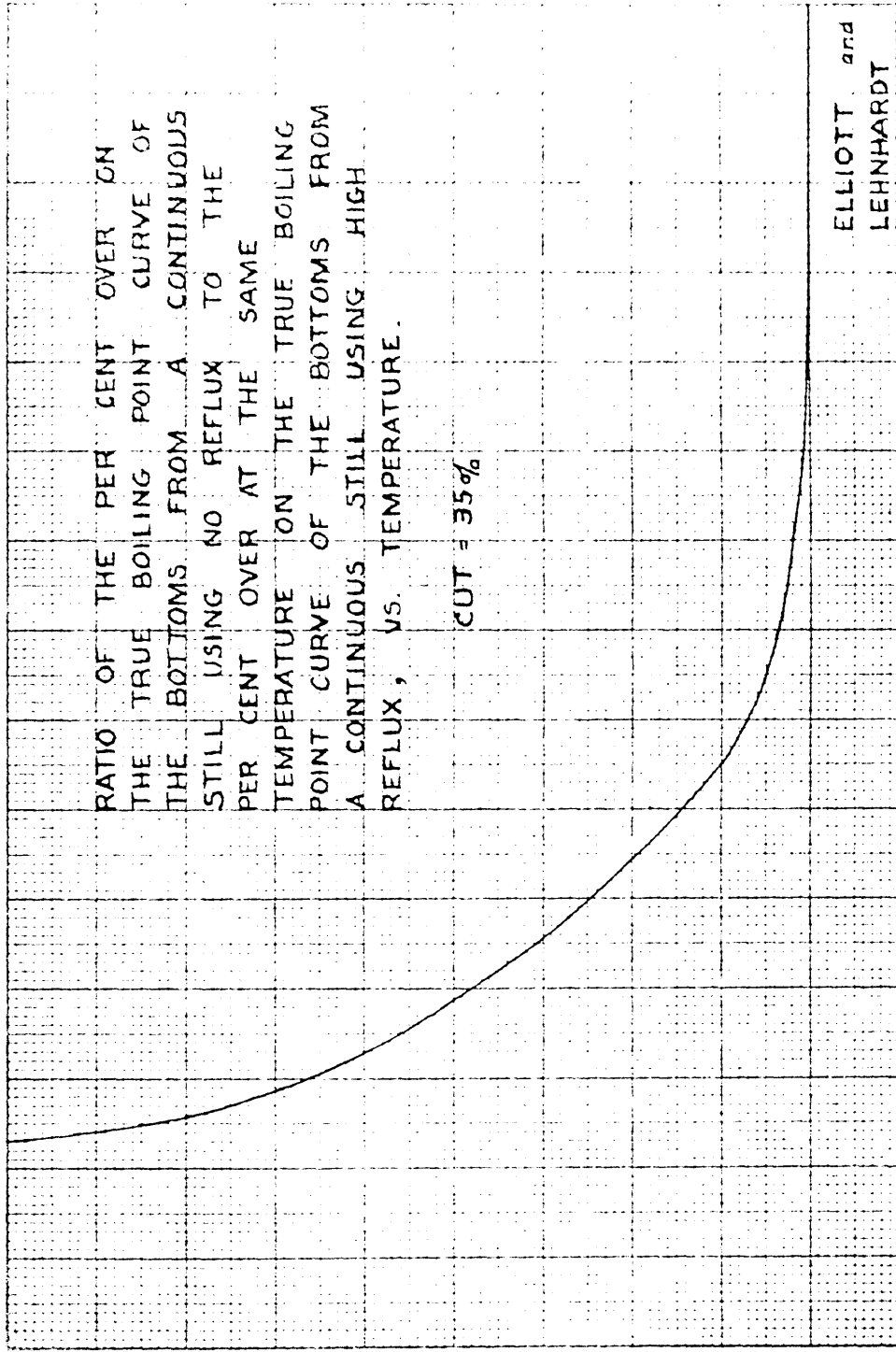


PLOT VIII

RATIO OF THE PER CENT OVER ON THE TRUE BOILING POINT CURVE OF THE BOTTOMS FROM A CONTINUOUS STILL USING NO REFLUX TO THE PER CENT OVER AT THE SAME TEMPERATURE ON THE TRUE BOILING POINT CURVE OF THE BOTTOMS FROM A CONTINUOUS STILL USING HIGH REFLUX, VS. TEMPERATURE.

CUT = 35%

ELLIOTT and
LEHNHARDT



170 190 210 230 250 270 290 310 330 350 370 390 410 430

TEMPERATURE ~ °F

uous still using reflux, vs. temperature.

The next four curves show the relationship between the true boiling point of the bottoms from the continuous still and the per cent of the original feed distilled over. The per cent of the feed distilled over includes that taken over both in the continuous still and in the analytical still.

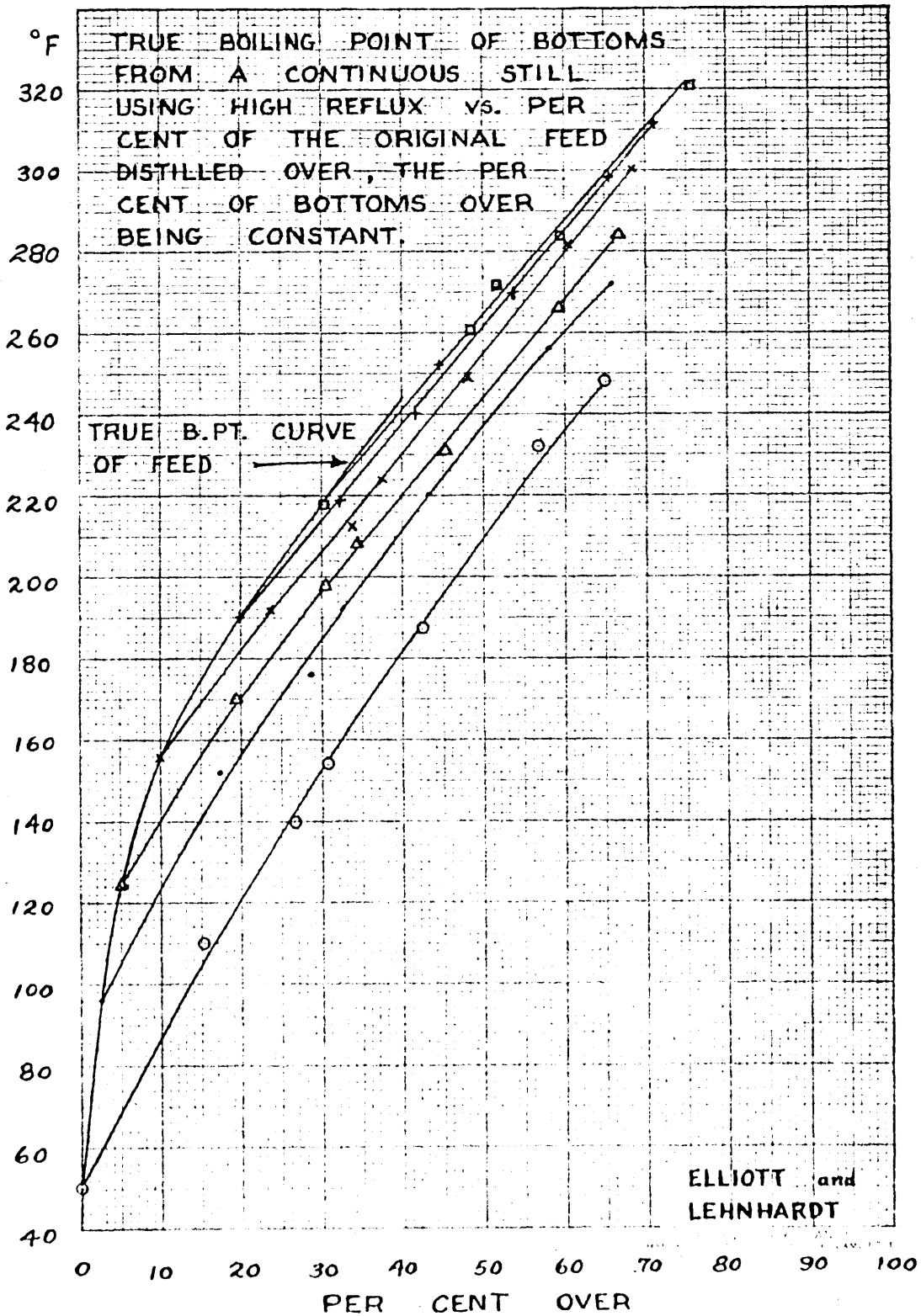
Plots IX and X show this relationship for a constant per cent of the bottoms distilled over. Each curve corresponds to a definite per cent of the bottoms distilled over in the analytical still, the value of which is given by the intersection of the curve with the true boiling point of the feed. The true boiling curve of the feed is, in this case, a plot of the true boiling point vs. per cent over for a zero cut. On this curve the per cent over is, therefore, taken over only in the analytical still and must equal the per cent of bottoms over since feed and bottoms are identical.

Consider the case where high reflux has been used in the analytical still. An example will probably render these points more clear.

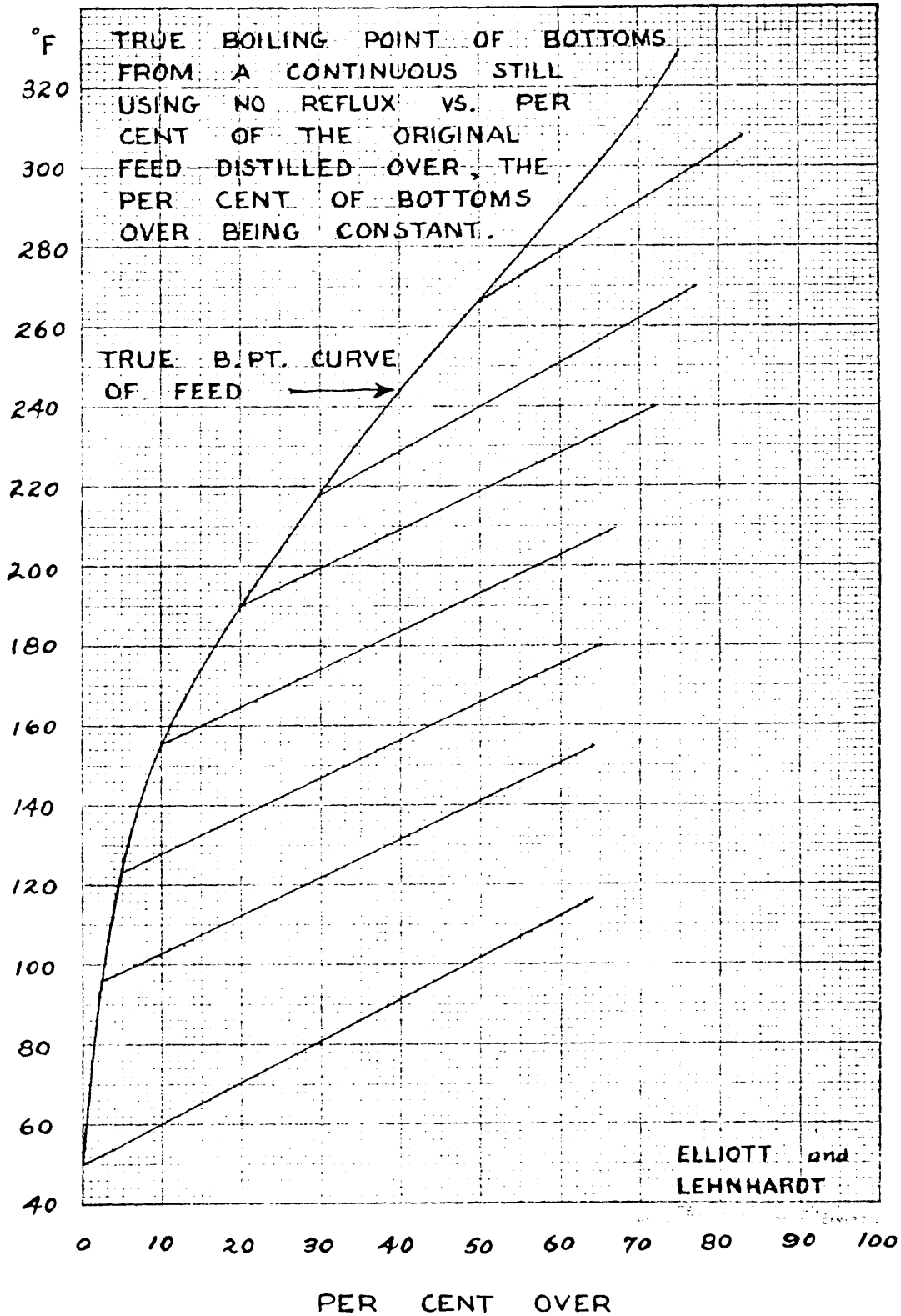
Suppose that when 5% of the bottoms has been taken over in the analytical still, the boiling point is 200 F. Following the curve intersecting the true boiling point curve at 5% over, we find that a temperature of 220 F. corresponds to 40% of the original feed distilled over.

The lowest curve shows the relationship between the initial boiling point of the bottoms and the volume per cent cut.

PLOT IX



PLOT X



The effect of rectification is clearly shown by a comparison of the two plots. Reflux tends to increase the slopes of the curves and causes them to more nearly approach the true boiling point curve of the feed. In fact, if complete rectification were obtained in the continuous still, the curves would all coincide with the true boiling point curve of the feed. When reflux is used, the curves corresponding to higher per cents of bottoms actually do manifest this coincidence.

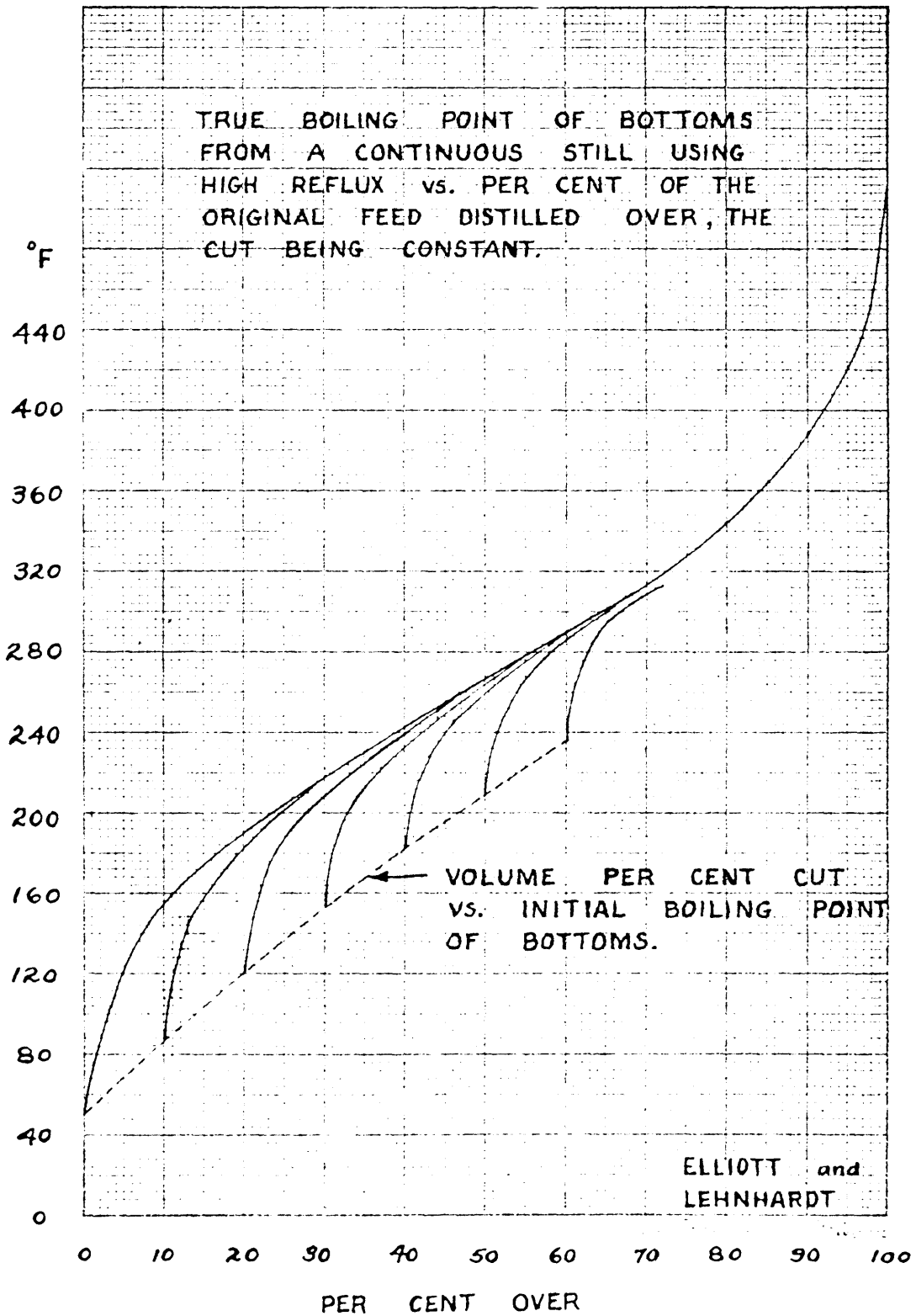
Plots XI and XII are merely true boiling point curves of bottoms for various cuts, the initial point being shifted to coincide with the volume per cent cut and the scale reduced to a basis of original feed. These curves have been calculated from Plots IX and X in order to get curves corresponding to cuts at 10% intervals.

When reflux is used, these curves approach and ultimately coincide with the true boiling point curve of the feed except in the case of the higher cuts. In this case, the amount of reflux has not been sufficient to maintain all of the heavy ends in the bottoms so that the per cent of lower boiling constituents has been increased accordingly and the curves fall below that for the feed.

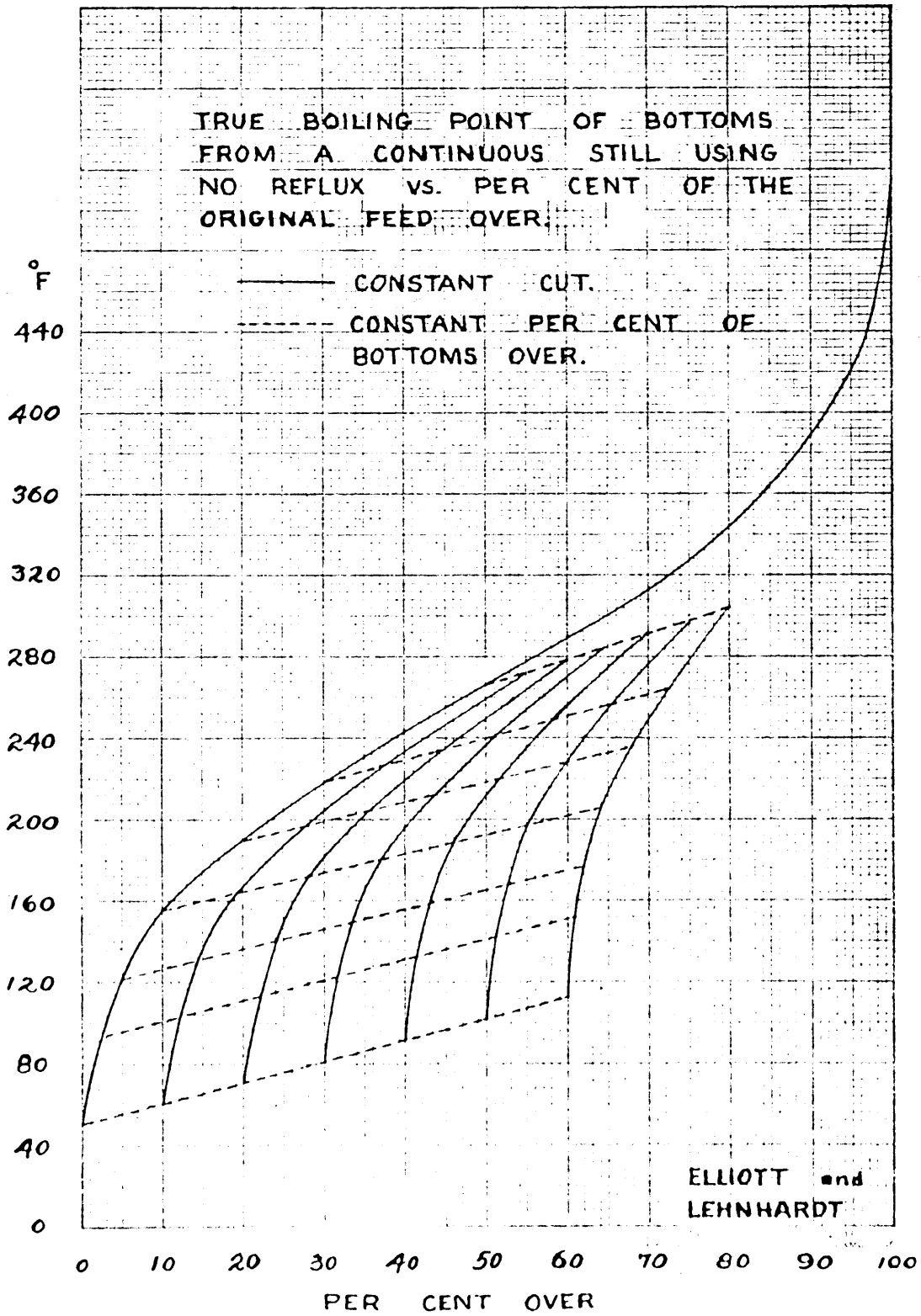
When no reflux is used, so many of the heavy ends are taken over that the divergence from the true boiling point curve of the feed is very much greater and, even in the case of the lower cuts, partial coincidence with it is not obtained.

The true boiling point curves of the bottoms from runs No. 19 and 20 of Reeder and Gordon (see page __) have

PLOT XI

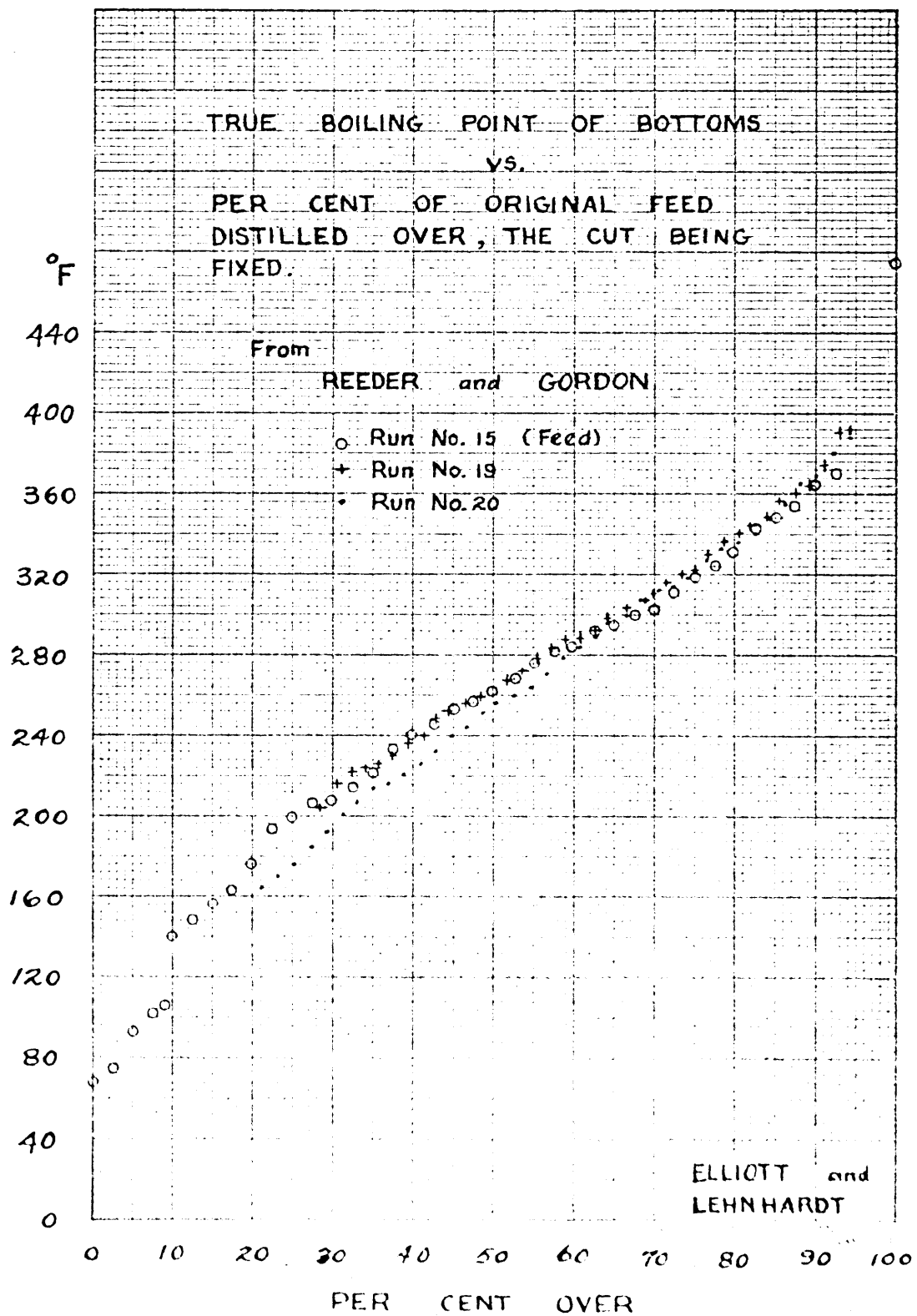


PLOT XII



been calculated on the basis of the original feed. In the case of run 19, it is seen that complete rectification has been obtained, while in the case of run 20 where a somewhat smaller reflux was used the separation is not quite perfect. In these runs, the reflux ratios were 19:1 and 16.5 respectively. These ratios are very much larger than the ones considered in the present thesis, and it will be remembered that Reeder and Gordon used a plate type column with the feed introduced directly into the still. These two runs are, therefore, examples of rectification above the feed plate, whereas the present thesis deals with rectification below the feed plate.

PLOT XIII



V RECOMMENDATIONS

(1) If further work is done with this continuous still, the overflow for the bottoms should be so arranged that they flow into a closed vessel with a by-pass to the vapor space in the still. This will prevent uneven flow due to fluctuations in pressure.

(2) The distillate from both the continuous and the analytical still should flow into receivers equipped with reflux condensers to minimize evaporation losses.

(3) The distillate and bottoms should be weighed so that all calculations could be made on a weight basis.

(4) A cooling coil should be installed in the oil circulating system of the continuous still in order to obtain better control of the reflux.

(5) The oil circulating pump on the continuous still should be equipped with a chain drive in order to obtain more uniform operation of the pump.

(6) A feed reservoir which would allow accurate calibration should be installed so that the feed rate could be determined more accurately.

VI C A L C U L A T I O N S

Since the true distillation curves obtained were run on a volume per cent basis, certain of the results which depend upon their use had to be calculated on a volume basis rather than upon the more preferable weight basis.

To avoid confusion, the term "per cent over" shall refer to volume per cent and the term "per cent cut" to weight per cent unless explicitly modified to mean otherwise.

The data calculated for the construction of the plots embodied in the results follow.

DATA FOR HEAT REQUIRED TO PREHEAT THE FEED

Run	gm./min.			c.c./min. Feed	% Wt.	Cut Vol.
	Dist.	Bott.	Feed			
1	10.0	25.5	35.5	47.5	28.2	30.6
2	7.6	24.5	32.1	43.0	23.7	26.6
3.	4.5	31.3	35.8	48.0	12.6	15.1
4	6.6	27.6	34.2	46.0	19.3	22.4
5	12.5	19.6	32.1	43.0	39.0	42.2
6	15.0	13.5	28.5	38.2	52.6	56.9
7	17.0	11.5	28.5	38.5	59.8	65.0
8	15.5	12.3	27.8	37.3	55.8	58.0
9	11.7	20.5	32.2	43.3	36.3	39.4
10	11.4	20.7	32.1	43.0	35.5	38.0
11	11.1	20.8	32.2	42.9	34.5	37.8
12	11.3	20.9	32.2	43.1	35.0	38.2
13	11.1	20.9	32.0	42.8	34.6	37.6
14	11.8	16.0	27.8	38.0	42.5	45.0

Runs No. 7 to 13 inclusive are computed using the value for the feed rate obtained by direct measurement.

DATA FOR HEAT REQUIRED TO PREHEAT THE FEED
(Continued)

Run	Col. T.	Feed T.	T. Diff.	Sp. Ht.	BTU./Lb.	Bott.
1	182 F	78 F	104	0.53	76.7	
2	168	76	92	0.53	64.2	
3	119	76	43	0.52	25.6	
4	144	76	68	0.53	44.6	
5	180	76	104	0.53	90.3	
6	199	75	124	0.53	138.7	
7	212	75	137	0.53	136.2	
8	199	72	127	0.53	152.0	
9	168	75	93	0.53	77.3	
10	167	76	91	0.53	74.8	
11	165	77	88	0.53	72.2	
12	166	73	93	0.53	75.9	
13	167	73	94	0.53	76.2	
14	268	73	-----	-----	-----	

(no reflux)

DATA FOR HEAT REMOVED IN THE COOLING OIL

TOTAL HEAT REMOVED AND REFLUX RATIO

Run	Lat. Ht.	Av. T. Diff Oil--F	c.c./min.	BTU./lb.Bott. In Oil	Total BTU. Lb.Bott.	Reflux
1	146	-----			76.7	1.34
2	142	-----			64.2	1.46
3	150	22.2	92	23.5	49.1	2.28
4	146	3.06	400	16.0	60.6	1.73
5	141	7.75	333	47.4	137.7	1.53
6	138	-----			138.7	0.905
7	137	-----			136.2	0.673
8	138	-----			152.0	0.875
9	142.5	-----			77.3	0.950
10	142.5	25	310	134.6	209.4	2.67
11	142.5	5.75	2015	200.5	272.7	3.59
12	142.5	-----			75.9	0.985
13	142.5	27	139	64.6	140.8	1.86

Sp. gr. of oil taken as 0.8 and sp. ht.
as 0.45 or (sp. gr.)(sp. ht.) = 0.36

DATA FOR THE WEIGHT PER CENT OF BOTTOMS
DISTILLING BELOW THE TEMPERATURE AT WHICH
THE GIVEN PER CENT CUT IS OVER WITH COMPLETE
RECTIFICATION.

(Plot II)

Run	Wt. % cut	Vol. % cut	Temp. F.	Vol. % Bottoms	Wt. % Bottoms
1	28.2	30.6	220	8.8	7.8
2	23.7	26.6	209	8.2	7.2
3	12.6	15.1	173	5.7	5.2
4	19.3	22.4	197	4.7	4.2
5	39.0	42.2	248	9.8	8.8
6	52.6	56.9	282	10.0	9.0
7	59.8	65.0	301	10.4	9.4
10	35.5	39.0	241	9.0	8.0
11	34.5	37.8	238	9.5	8.5
12	35.0	38.2	239	10.0	9.0
13	34.6	37.6	237	6.5	6.0

DATA FOR PER CENT CUT
vs.
POUNDS OF LIGHTS IN BOTTOMS PER 100 POUNDS OF FEED

With Reflux

Data read from Plot IV

PLOT II

Cut	Initial B. Pt.	Vol. % Over	Wt. % Over	Lbs. Lights/100	Lbs. Bottoms
10	100	3.0	2.3	7.7	
20	132	6.0	5.0	15.0	
30	161	11.5	9.6	20.4	
40	190	20.2	17.5	22.5	
50	220	30.9	28.5	21.5	
60	250	43.0	39.8	20.2	

Without Reflux

Data read from Plot V

Cut	Initial B. Pt.	Vol. % Over	Wt. % Over	Lbs. Lights/100	Lbs. Bottoms
10	62	0.5	0.4	9.6	
20	72	1.0	0.8	19.2	
30	83	1.9	1.5	28.5	
40	94	2.5	2.0	38.0	
50	105	3.4	2.7	47.3	
60	116	4.3	3.4	56.6	

DATA FOR LIGHTS IN BOTTOMS vs. HEAT REQUIRED FOR REFLUX.

(Plot III)

Run	Initial B.Pt.-F	% Cut	Vol. % Over	Wt. % Over	Lights per 100 lbs. Feed
5	187	39.0	19.0	17.2	21.8
10	184	35.5	18.0	16.3	19.2
11	180	34.5	17.0	15.4	19.1
12	170	35.0	14.0	11.7	22.3
13	174	34.6	15.5	14.0	20.6
---*	89	35.0	----	1.5	33.5

* From Plot V.

DATA FOR CURVES OF PER CENT CUT vs. TRUE BOILING POINT
OF THE BOTTOMS.

With Reflux.

(Plot IV)

Run No.---	1	2	3	4	5	6	7
% Cut---	28.2	23.7	12.6	19.3	39.0	52.6	59.8

% Over

0	154	140	110	---	187	232	248
2.5	192	176	152	---	220	256	272
5	208	198	170	---	231	266	283
10	224	212	192	208	249	282	299
20	252	240	219	230	270	298	312

(True Boiling Points----^oF)

Run No.---	10	11	12	13	
% Cut---	35.5	34.5	35.0	34.6	0

% Over

0	184	180	170	174	52
2.5	216	208	200	216n	96
5	227	225	221	228	124
10	244	239	239	248	155
20	263	257	263	264	190

(True Boiling Point----^oF)

DATA FOR CURVES OF PER CENT CUT vs. TRUE BOILING POINT
OF THE BOTTOMS.

Without Reflux

(Plot V)

Run No.-----	14	15	16	17	
% Cut-----	42	10	25.5	61	0
% Over					
0	90	64	80	120	52
2.5	129	107	116	155	96
5	154	136	150	180	124
10	198	161	183	207	155
20	223	---	210	240	190

(True Boiling Point----^oF)

DATA FOR TRUE BOILING POINT OF BOTTOMS vs. PER CENT OF THE ORIGINAL FEED DISTILLED OVER.

Run No. 1

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	154	192	208	224	252	272
Bottoms Over / 100 Feed	0.0	1.7	3.5	6.9	13.9	20.8
Distillate / 100 Feed	<u>30.6</u>	<u>30.6</u>	<u>30.6</u>	<u>30.6</u>	<u>30.6</u>	<u>30.6</u>
Per Cent of Feed Over	30.6	32.3	34.1	37.5	44.5	51.4

Run No. 2

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	140	176	198	212	240	261
Bottoms Over / 100 Feed	<u>0.0</u>	<u>1.8</u>	<u>3.7</u>	7.3	14.7	22.0
Distillate / 100 Feed	<u>26.6</u>	<u>26.6</u>	<u>26.6</u>	<u>26.6</u>	<u>26.6</u>	<u>26.6</u>
Per Cent of Feed Over	26.6	28.4	30.3	33.9	21.3	28.6

Run No. 3

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	110	152	170	192	219	
Bottoms Over / 100 Feed	0.0	2.1	4.2	8.5	17.0	
Distillate / 100 Feed	<u>15.1</u>	<u>15.1</u>	<u>15.1</u>	<u>15.1</u>	<u>15.1</u>	
Per Cent of Feed Over	15.1	17.2	19.3	23.6	32.1	

Run No. 5

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	187	220	231	249	270	284
Bottoms Over / 100 Feed	0.0	1.4	2.9	5.8	11.6	17.3
Distillate / 100 Feed	<u>42.2</u>	<u>42.2</u>	<u>42.2</u>	<u>42.2</u>	<u>42.2</u>	<u>42.2</u>
Per Cent of Feed Over	42.2	43.6	45.1	48.0	53.8	59.5

Run No. 6

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	232	256	266	282	298	304
Bottoms Over / 100 Feed	0.0	1.1	2.2	4.3	8.6	12.9
Distillate / 100 Feed	<u>56.9</u>	<u>56.9</u>	<u>56.9</u>	<u>56.9</u>	<u>56.9</u>	<u>56.9</u>
Per Cent of Feed Over	56.9	58.0	59.1	61.2	65.5	69.8

Run No. 7

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	248	272	284	300	312	321
Bottoms Over / 100 Feed	0.0	0.9	1.8	3.5	7.0	10.7
Distillate / 100 Feed	<u>65.0</u>	<u>65.0</u>	<u>65.0</u>	<u>65.0</u>	<u>65.0</u>	<u>65.0</u>
Per Cent of Feed Over	65.0	65.9	66.8	68.5	72.0	75.7

Run No. 10

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	184	210	225	243	263	276
Bottoms Over / 100 Feed	0.0	1.6	3.1	6.2	12.4	18.6
Distillate / 100 Feed	<u>38.0</u>	<u>38.0</u>	<u>38.0</u>	<u>38.0</u>	<u>38.0</u>	<u>38.0</u>
Per Cent of Feed Over	38.0	39.6	41.1	44.2	50.4	56.6

Run No. 11

Per Cent of Bottoms Over	0	2.5	5	10	20	30
Temperature F.	180	206	220	239	257	268
Bottoms Over / 100 Feed	0.0	1.6	3.1	6.2	12.4	18.6
Distillate / 100 Feed	<u>37.8</u>	<u>37.8</u>	<u>37.8</u>	<u>37.8</u>	<u>37.8</u>	<u>37.8</u>
Per Cent of Feed Over	37.8	39.4	39.9	44.0	50.2	56.4

Run No. 12

Per Cent of Bottoms Over	0	2.5	5	10	20 $\frac{1}{2}$	30
Temperature F.	170	199	216	238	263	280
Bottoms Over / 100 Feed	0.0	1.6	3.1	6.2	12.4	18.6
Distillate / 100 Feed	<u>38.2</u>	<u>38.2</u>	<u>38.2</u>	<u>38.2</u>	<u>38.2</u>	<u>38.2</u>
Per Cent of Feed Over	38.2	39.8	41.3	44.4	50.6	56.8

Run No. 13

Per Cent of Bottoms Over	0	2.5	5	10	20	25
Temperature F.	174	216	230	248	264	270
Bottoms Over / 100 Feed	0.0	1.6	3.1	6.2	12.5	15.6
Distillate / 100 Feed	<u>37.6</u>	<u>37.6</u>	<u>37.6</u>	<u>37.6</u>	<u>37.6</u>	<u>37.6</u>
Per Cent of Feed Over	37.6	39.2	40.7	43.8	50.1	53.2

From Plot V For A 35% Cut

Per Cent of Bottoms Over	0	2.5	5	10	20	30	50
Temperature F.	89	129	156	187	218	247	290

DATA FOR THE RATIO OF THE PER CENT OVER ON THE TRUE BOILING POINT CURVE OF THE BOTTOMS FROM A CONTINUOUS STILL USING NO REFLUX TO THE PER CENT OVER AT THE SAME TEMPERATURE ON THE TRUE BOILING POINT CURVE OF THE BOTTOMS FROM A CONTINUOUS STILL USING HIGH REFLUX.

CUT = 35 %

Data read from Plot VII

PLOT VIII

Temp. F.	% Over With Reflux	% Over Without Reflux	Ratio
170	0	4.3	
200	1.8	8.3	4.62
220	3.8	12.6	3.32
240	6.8	17.3	2.54
260	11.3	22.3	1.97
280	18.6	27.8	1.50
300	26.8	33.4	1.25
320	34.3	38.8	1.13
330	37.3	41.3	1.11

VII S A M P L E C A L C U L A T I O N S.

PER CENT CUT

Run No. 3

Volume of distillate = 450 c.c. at 72 F

Volume of bottoms = 2588 c.c. at 90 F

Length of run = 62 min.

Column temperature = 119 F

Feed temperature = 76 F

Sp. Gr. Feed = 0.745 at 60 F

Sp. Gr. distillate = 0.63 at 60 F

Sp. Gr. bottoms = 0.77 at 60 F

The decrease in specific gravity per degree F. temperature rise is taken as 0.001.

$$\frac{(450)(0.62)}{62} = 4.5 \text{ gm. distillate / min.}$$

$$\frac{(2588)(0.75)}{62} = 31.3 \text{ gm. bottoms / min.}$$

35.8 gm. feed / min.

$$\frac{(35.8)}{0.745} = 48.0 \text{ c.c. feed / min.}$$

$$\frac{(4.5)}{(35.8)} = 12.6 \text{ weight \% cut}$$

$$\frac{(450)(100)}{(62)(48)} = 15.1 \text{ volume \% cut}$$

HEAT CONSUMED TO GIVE REFLUX

PLOTS I AND III

Run No. 3

$$\frac{(119 - 76)(0.52)(35.8)}{(31.3)} = 25.6 \text{ btu / lb. bottoms}$$

To preheat feed

$$\frac{(92)(0.36)(22.2)}{454} = 1.62 \text{ btu / min. removed in oil}$$

$$\frac{(1.62)(454)}{(31.3)} = 23.5 \text{ btu / lb. bottoms removed in oil}$$

$$25.6 + 23.5 = 49.1 \text{ btu / lb. bottoms for reflux}$$

$$\frac{(49.1)(31.3)}{(150)(4.5)} = 2.88 \text{ reflux ratio}$$

WEIGHT PER CENT OF BOTTOMS DISTILLING BELOW THE TEMPERATURE AT WHICH A GIVEN PER CENT CUT IS OVER WITH COMPLETE RECTIFICATION.

PLOTS I AND II

Run No. 12

Weight per cent cut = 35.0

Volume per cent cut = 38.2

On the true boiling point curve of the feed, the temperature corresponding to 38.2 per cent over is 239 F. On the true boiling point curve of the bottoms, the per cent over at 239 F is 10.0 per cent (by volume) which is equivalent to 9 per cent by weight, or .09 pounds of material per pound of bottoms.

LIGHTS IN BOTTOMS

PLOT III

Run No. 12

The initial boiling point of the bottoms is 170 F. Turning to the distillation curve of the feed, we find that at 170 F there is 14.0% over (by volume) which is equivalent to 12.7% over by weight.

The cut is 35.0%. Therefore, 35 pounds of distillate per 100 pounds of feed has been distilled over in the continuous still to carry over all material boiling below 170 F. But with complete rectification, it is necessary to distill over only 12.7 pounds of distillate to carry over this material. Therefore, $(35.0 - 12.7) = 22.3$ lbs. of heavy in the overhead / 100 pounds feed which, from a material balance, equals the lights in the bottoms per 100 pounds of feed.

PER CENT CUT vs. TRUE BOILING POINT AT A GIVEN PER CENT
OVER

PLOTS IV AND V

Run No. 1

Cut = 28.2%

On the true boiling point curve of the bottoms, 2.5% is over at 192 F. Therefore on Plot IV, 28.2% is plotted against 192 F, falling on the curve marked 2.5 per cent over on true boiling point curve.

LIGHTS IN BOTTOMS vs. PER CENT CUT

PLOT VI

The lights in the bottoms for these curves are calculated as in Plot III. The necessary data are furnished from Plots IV and V using the curves "0 per cent over on true b. pt. curve."

TRUE BOILING POINT OF THE BOTTOMS vs. PER CENT OF THE ORIGINAL FEED DISTILLED OVER.

PLOTS VII AND XIII.

Run No. 12

35.0 weight per cent cut

38.2 volume per cent cut

2.5 per cent of the bottoms is equivalent to

$$\frac{(2.5)(100 - 38.2)}{100} = 1.6 \text{ parts of bottoms over per } 100 \text{ parts of feed.}$$

$38.2 + 1.6 = 39.6$ per cent of the original feed over.

The temperature on the true boiling point curve of the corresponding to 2.5% over is 199° F.

Plot 39.6% vs. 199° F.

In the above calculation the effect of change in specific gravity has been neglected. Errors introduced by this assumption are compensating so that the resultant error is small.

The "per cents" in the above calculation are all volume per cents.

PLOT VII-----Without Reflux.

From Plot V, on the curve marked 2.5 % over on the true B.Pt. Curve, the temperature corresponding to a cut of 35.0% is 129° F. Plot 39.6% over vs. 129° F.

RATIO OF THE PER CENT OVER ON THE TRUE BOILING POINT CURVE OF THE BOTTOMS FROM A CONTINUOUS STILL USING NO REFLUX TO THE PER CENT OVER ON THE TRUE BOILING POINT CURVE OF THE BOTTOMS FROM A CONTINUOUS STILL USING HIGH REFLUX , vs. TEMPERATURE.

PLOT VIII.

From Plot VI at 200°F the per cent over with reflux is

$$(40 - 38.2) = 1.8$$

The per cent over without reflux is

$$(46.5 - 38.2) = 8.3$$

The ratio $\frac{8.3}{1.8} = 4.62$ is plotted against 200°F .

TRUE BOILING POINT OF THE BOTTOMS vs. PER CENT OF THE ORIGINAL FEED DISTILLED OVER , THE PER CENT OF BOTTOMS OVER BEING CONSTANT.

PLOTS IX AND X.

The calculation is the same as for Plot VII. The point calculated in the example falls on the curve intersecting the true boiling point curve of the feed at 2.5% over.

All points on the curve intersecting the true boiling point curve of the feed at 2.5% over correspond to 2.5% over on the true boiling point curve of the bottoms.

TRUE BOILING POINT OF BOTTOMS vs, PER CENT OF THE ORIGINAL
FEED DISTILLED OVER , THE CUT BEING CONSTANT.

PLOTS XI AND XII.

With Reflux.

Points on these curves are read from Plots IX and X.

Consider on Plot IX the curve corresponding to 2.5%
of bottoms over. As in the case of Plot VII

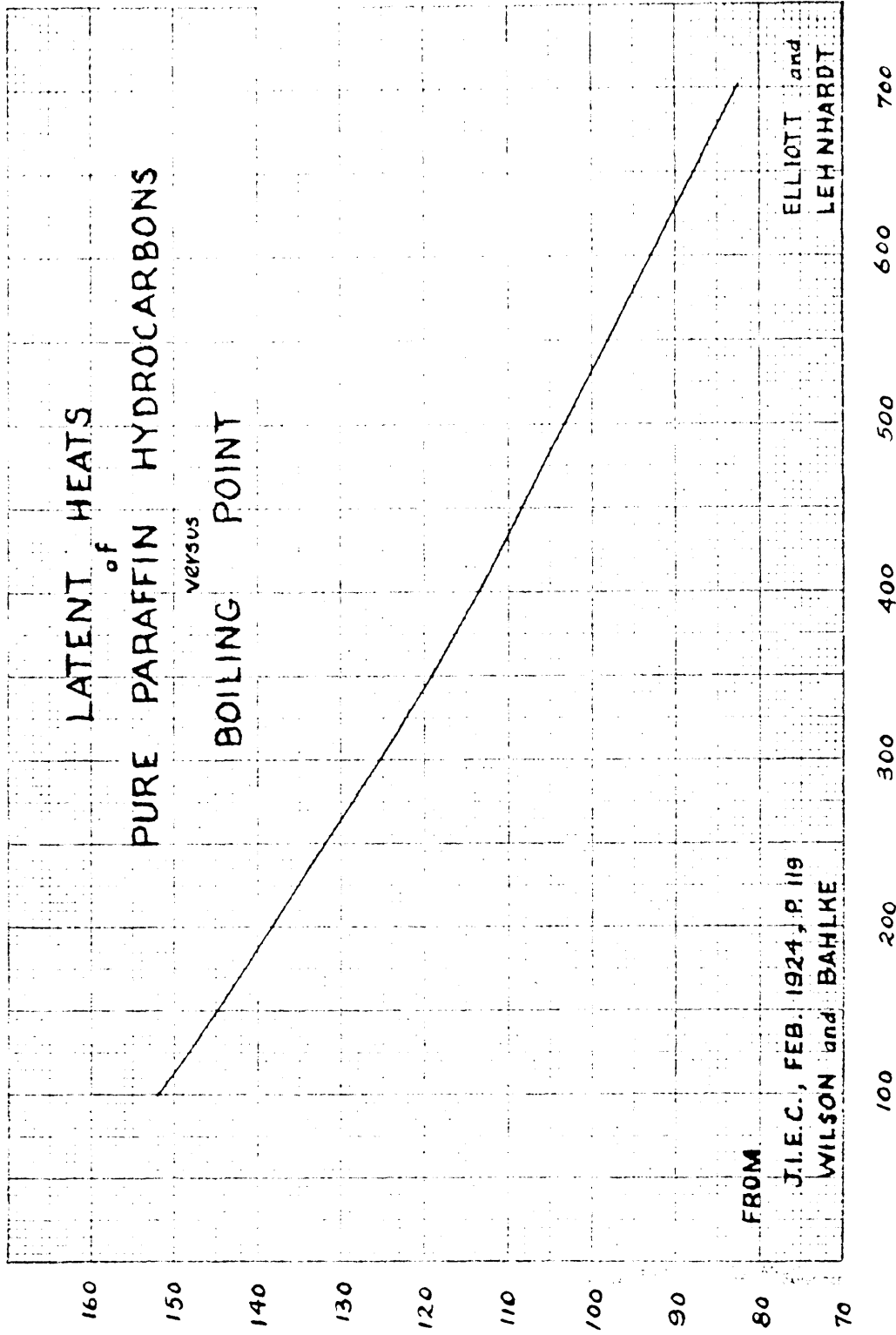
$$\frac{(2.5)((100 - \text{Vol. \% Cut})}{100} + \text{Vol. \% Cut} = \% \text{ over}$$

Therefore for a 30% cut by volume , the per cent over
is $(2.5)(0.7) + 30 = 31.8\%$ over.

The temperature corresponding to 31.8% over, on Plot IX,
is 189^o F.

Plot 189^o F vs. 31.8% over , falling on the curve
intersecting the dotted line at 30% over.

LATENT HEATS
of
PURE PARAFFIN HYDROCARBONS
versus
BOILING POINT



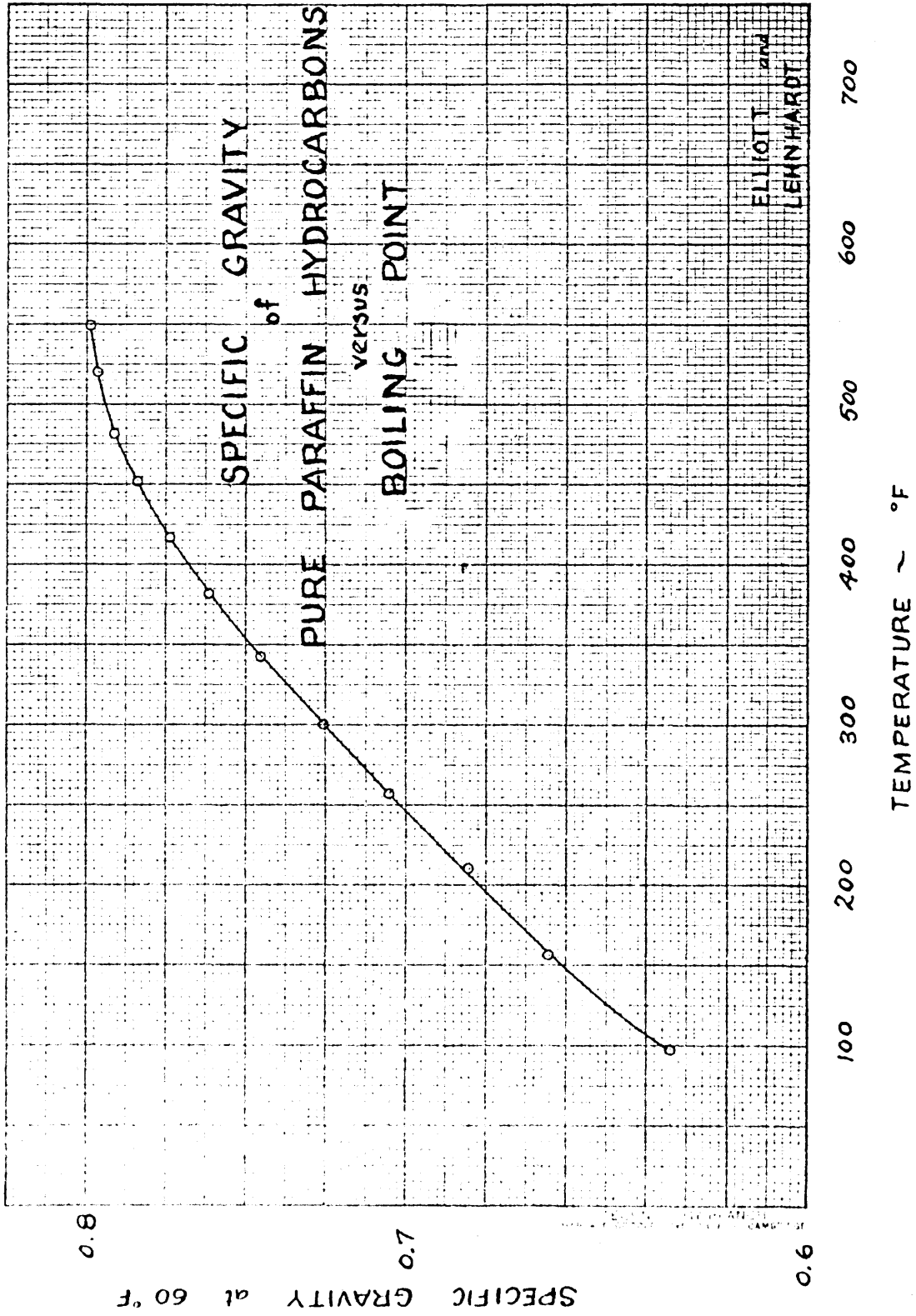
FROM

J.I.E.C., FEB. 1924, P. 119
WILSON and BAHLKE

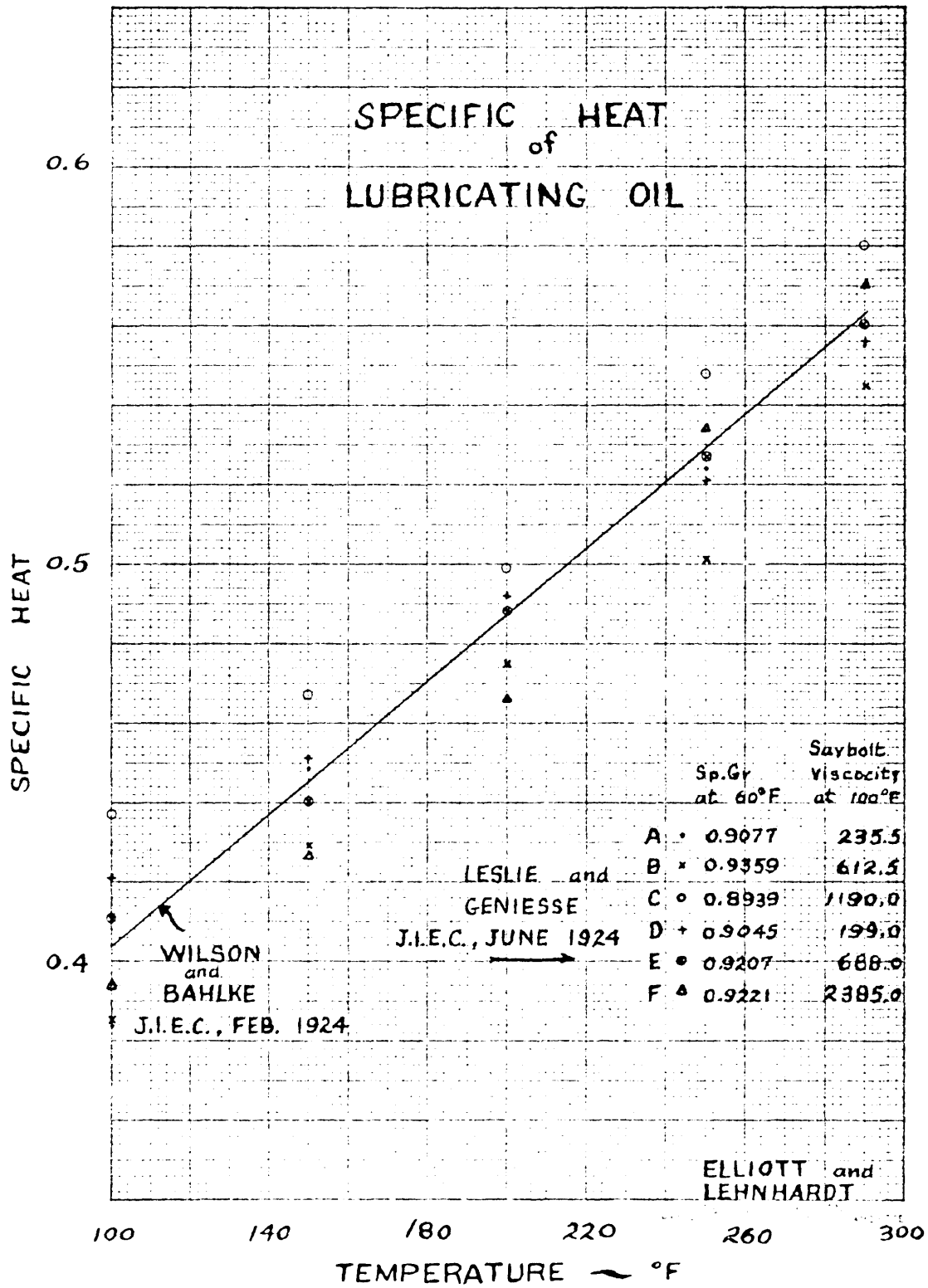
ELLIOTT and
LEHNHARDT

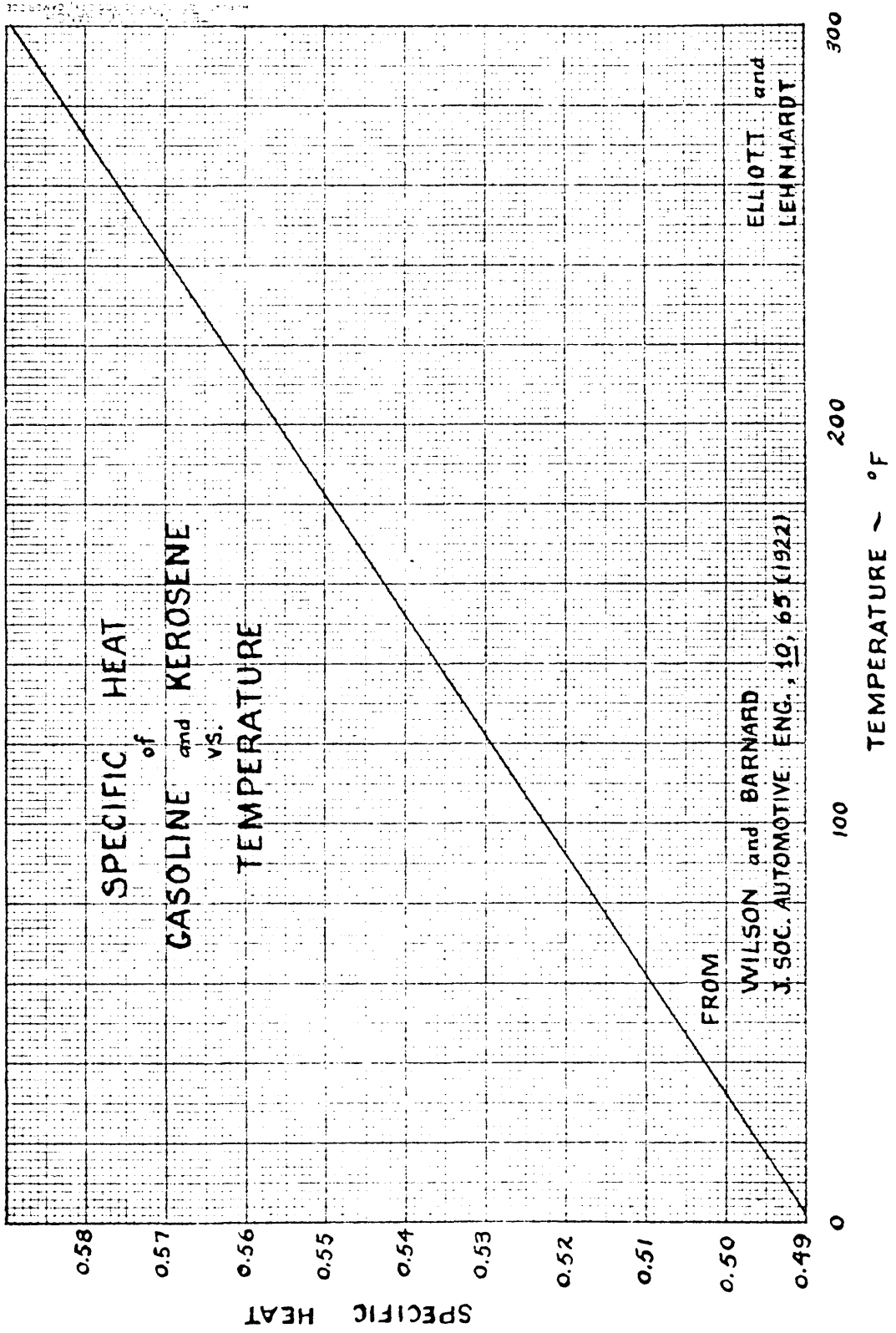
LATENT HEAT ~ BTU PER POUND

BOILING POINT ~ °F



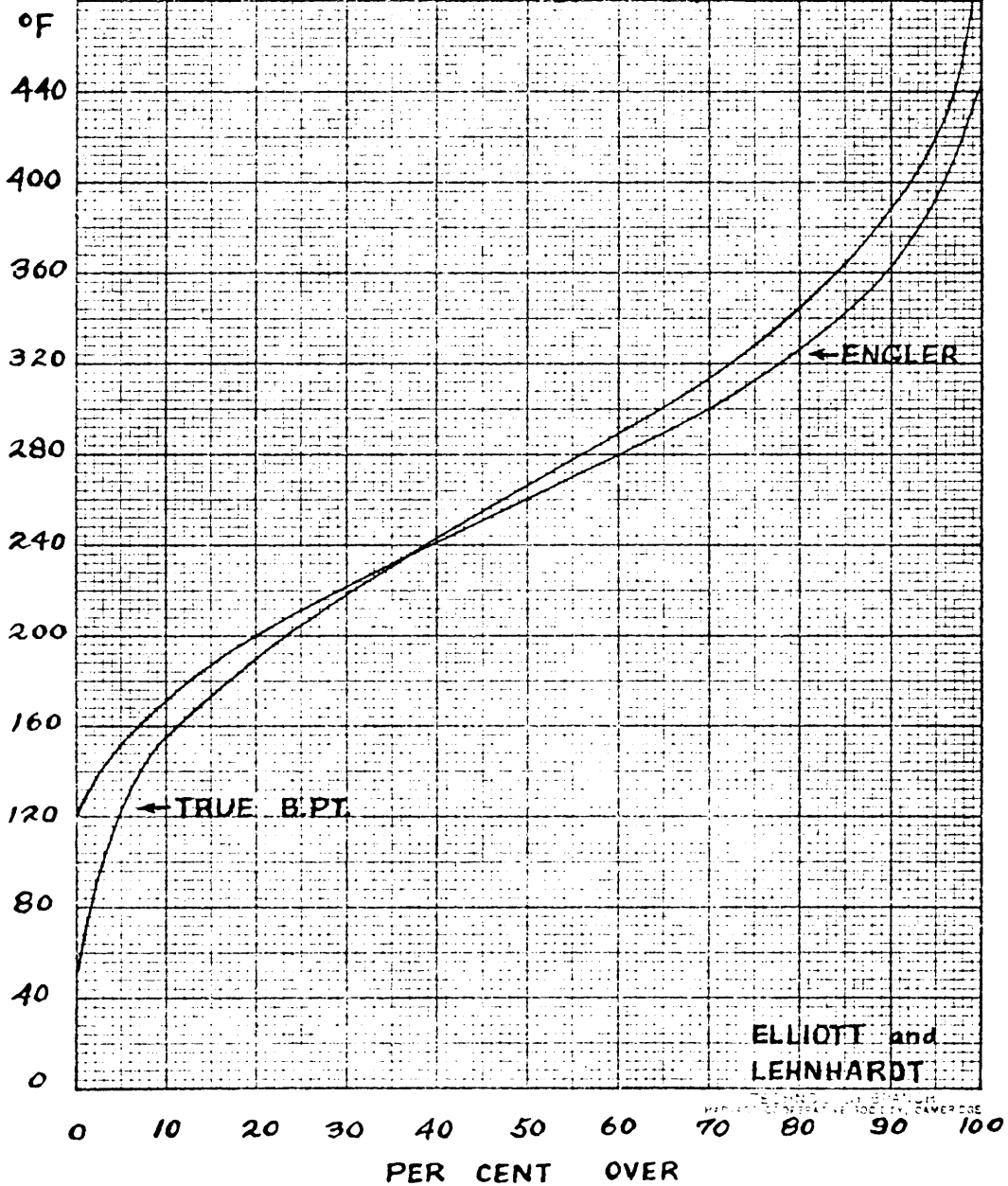
SPECIFIC HEAT of LUBRICATING OIL



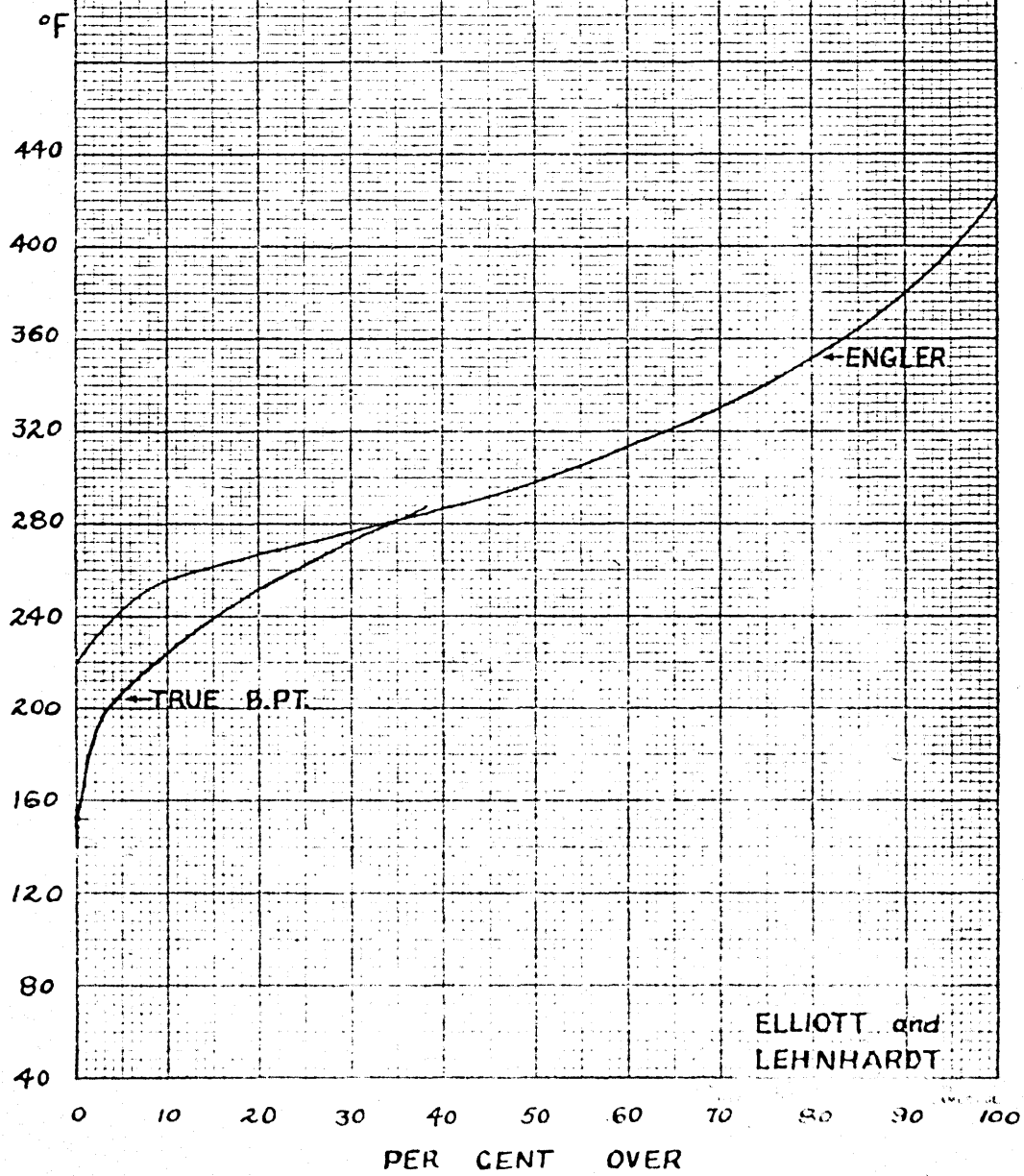


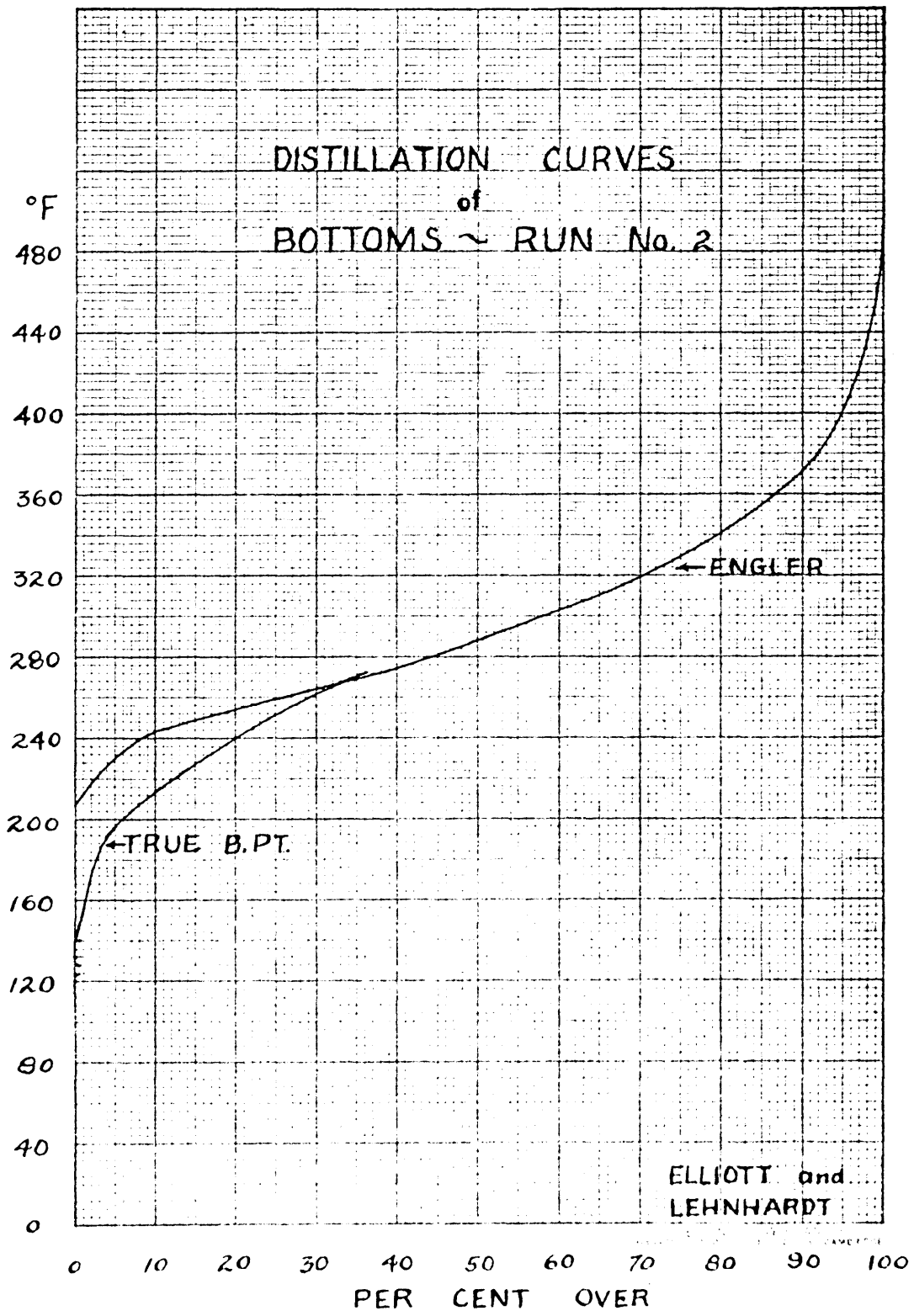
VIII O R I G I N A L D A T A .

DISTILLATION CURVES of SOCONY GASOLINE



DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 1

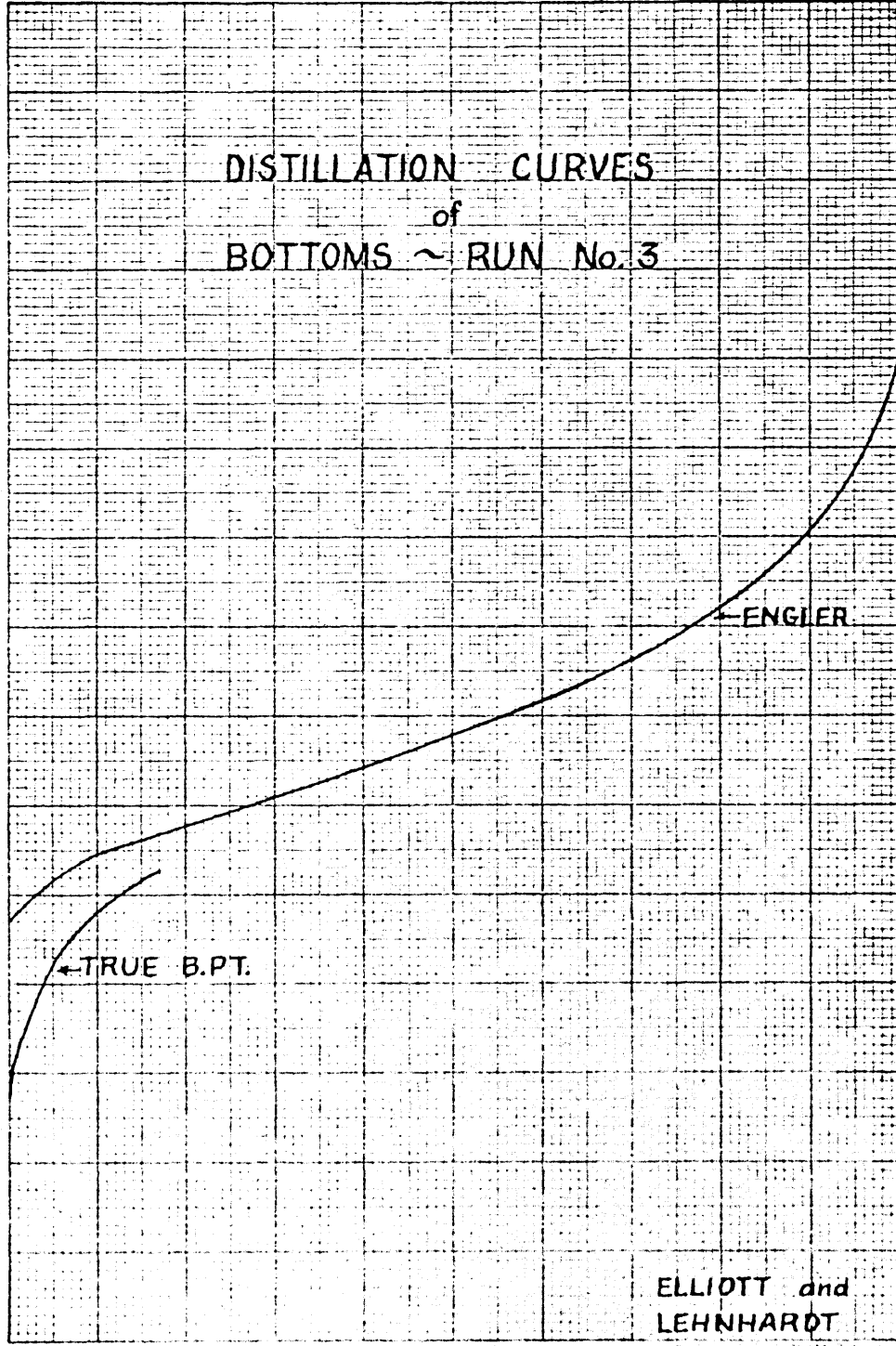




DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 3

°F

440
400
360
320
280
240
200
160
120
80
40
0



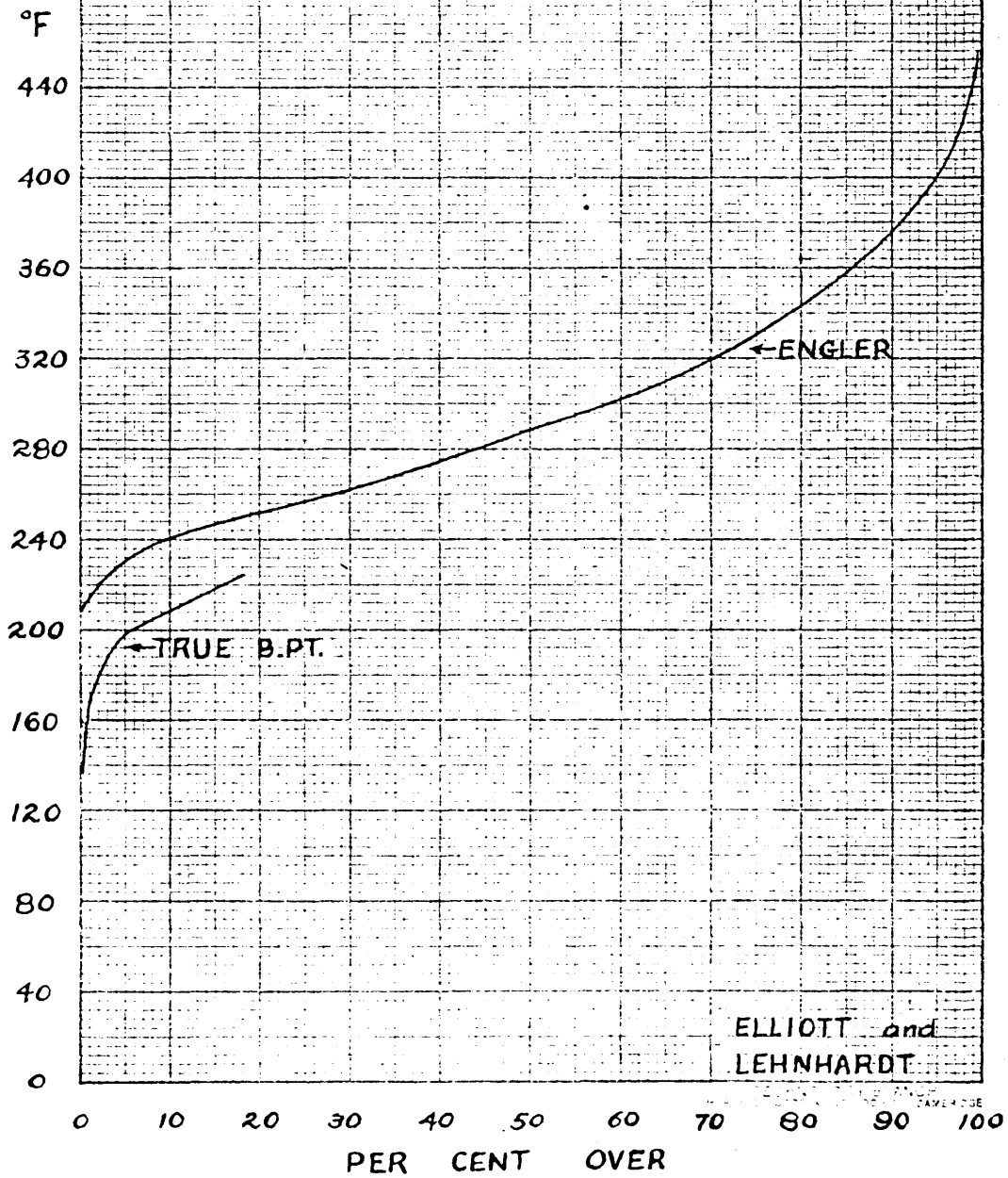
← TRUE B.P.T.

← ENGLER

ELLIOTT and
LEHNHARDT

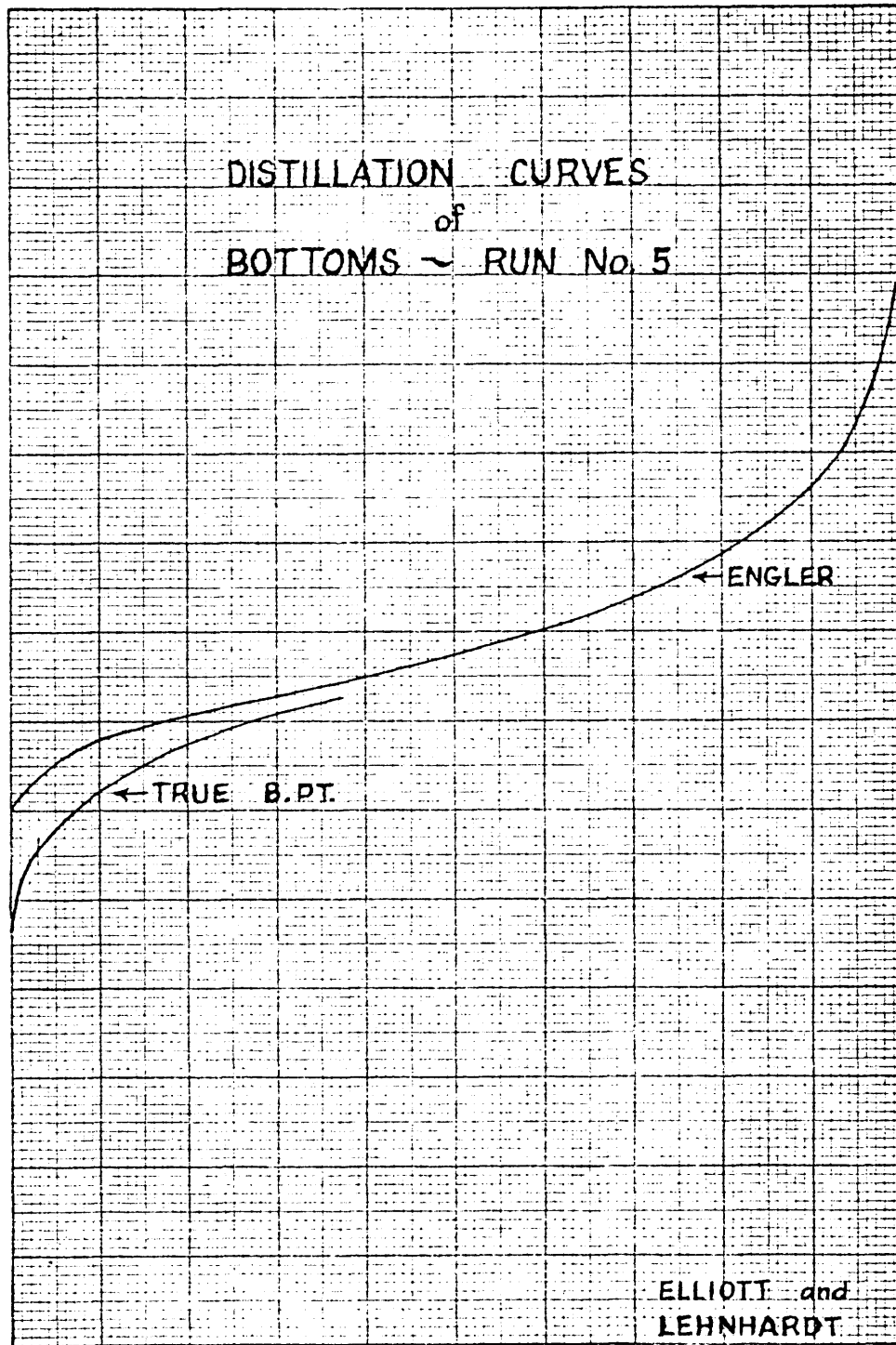
0 10 20 30 40 50 60 70 80 90 100
PER CENT OVER

DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 4



DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 5

°F
440
400
360
320
280
240
200
160
120
80
40
0

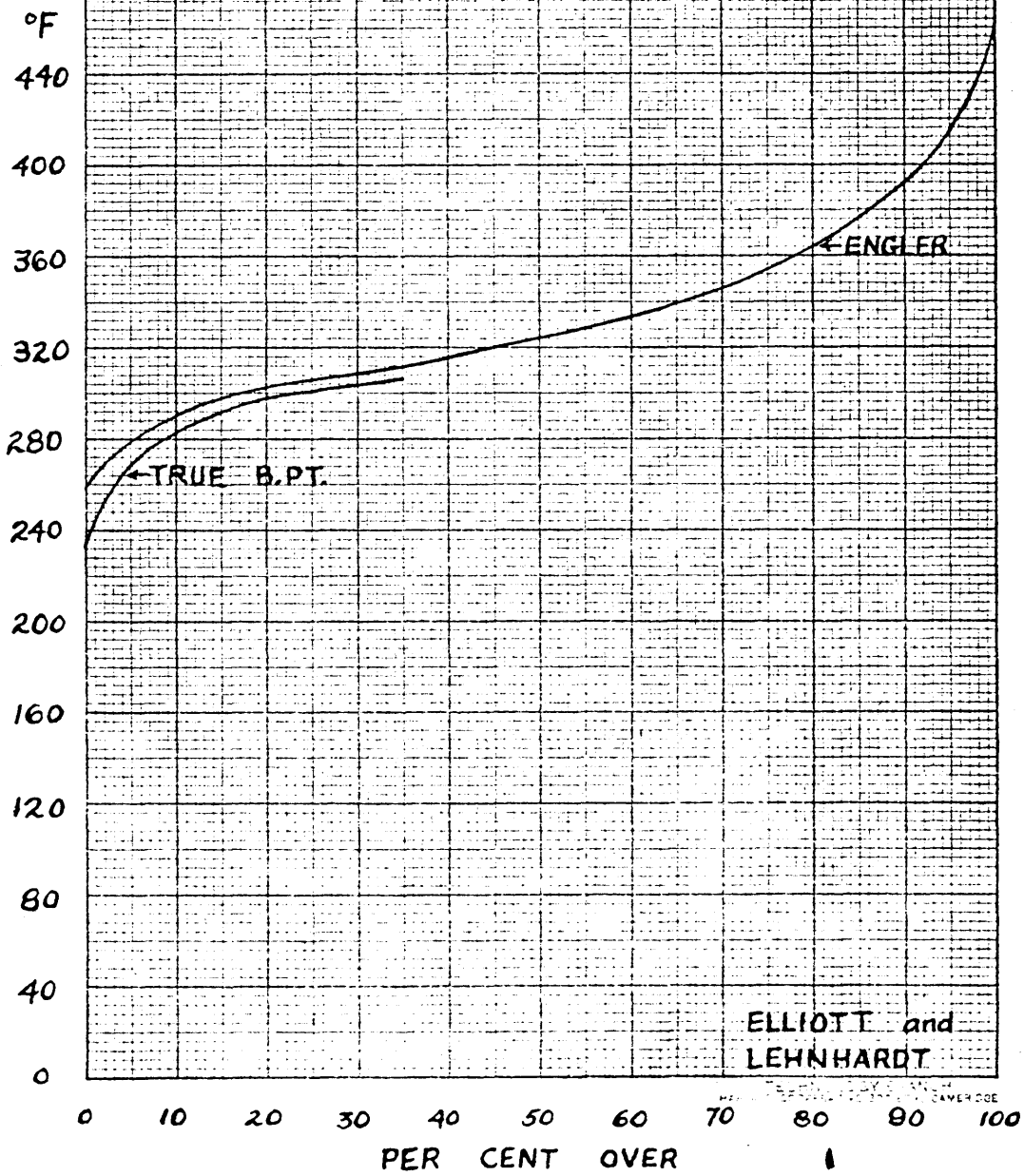


ELLIOTT and
LEHNHARDT

0 10 20 30 40 50 60 70 80 90 100
PER CENT OVER

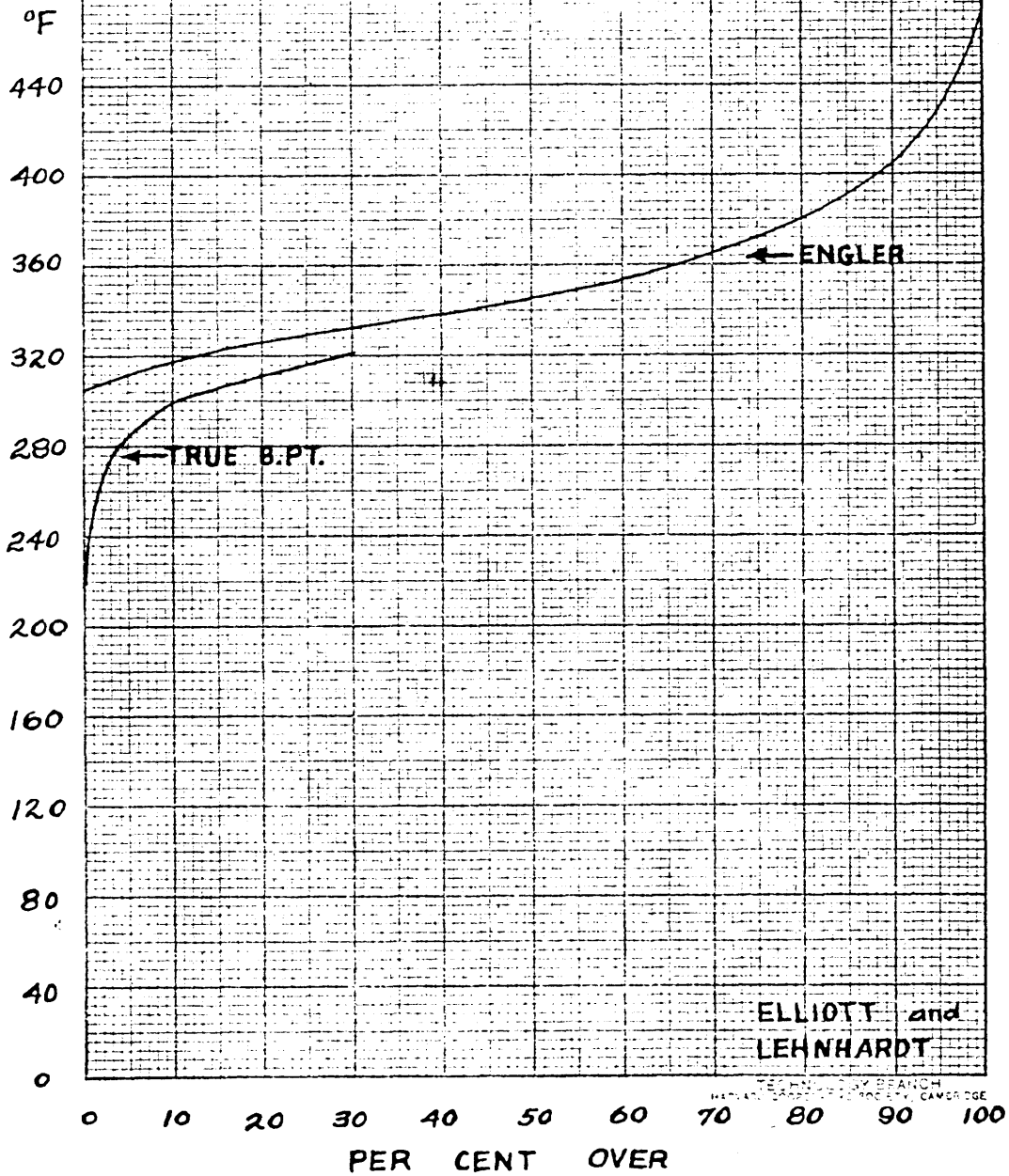
ELLIOTT and LEHNHARDT
MANUFACTURING COMPANY
100 BRIDGE STREET, CAMBRIDGE, MASS.

DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 6

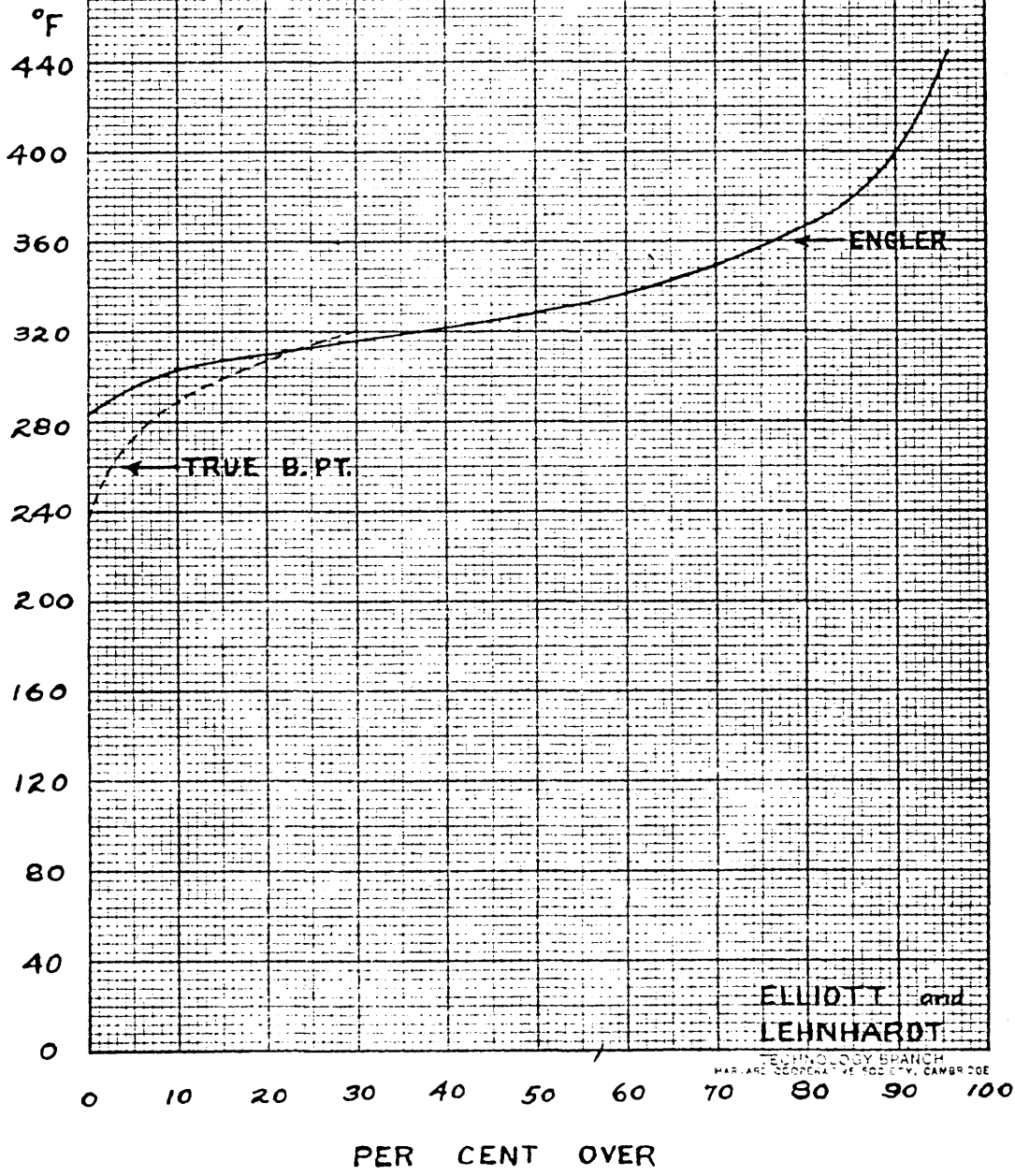


ELLIOTT and
LEHNHARDT

DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 7



DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 8



ELLIOTT and
LEHNHARDT

TECHNOLOGY BRANCH
MARINE COOPERATIVE SOCIETY, CAMBRIDGE

DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 9

°F

440

400

360

320

280

240

200

160

120

80

40

0

← ENGLER

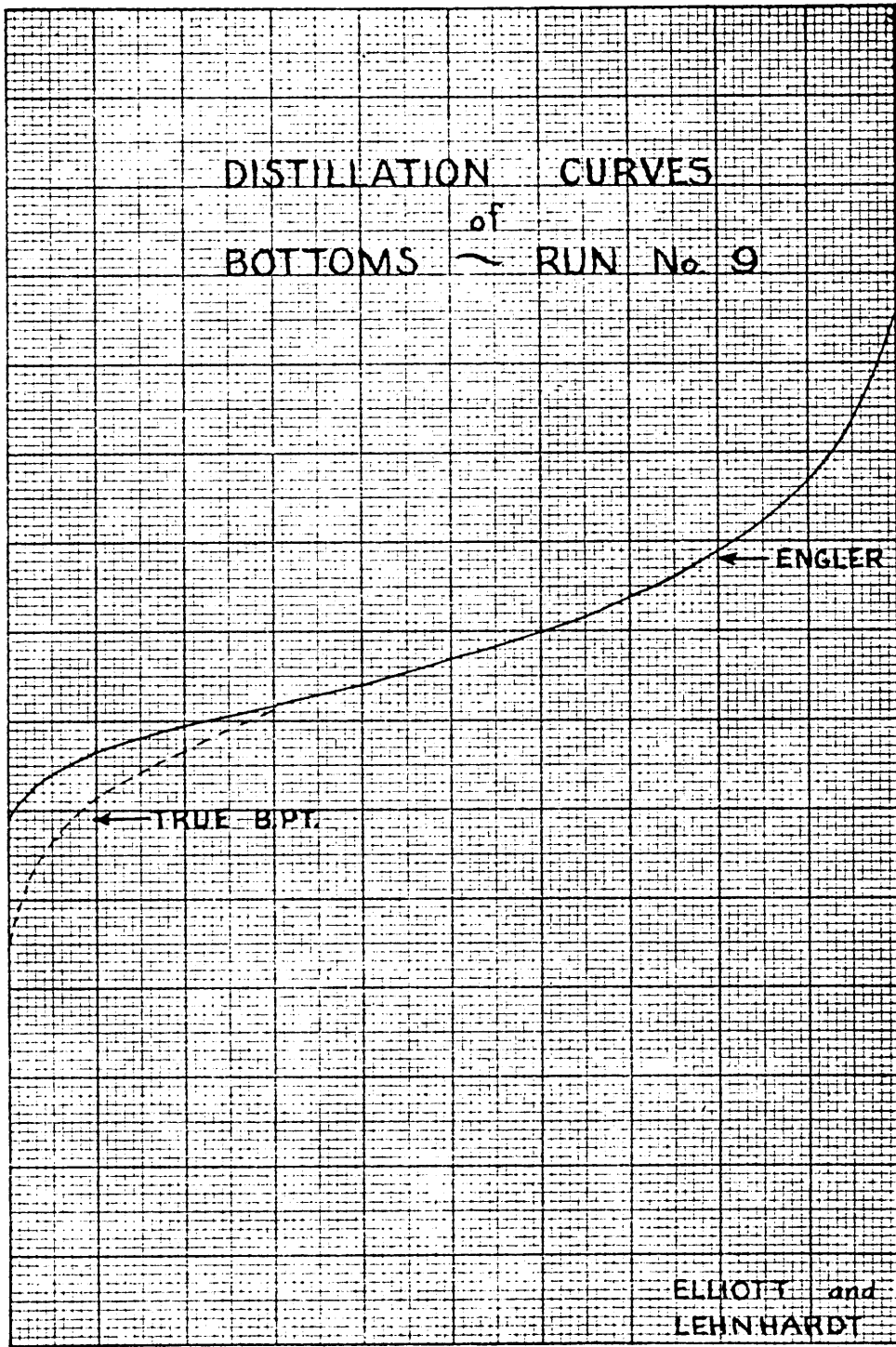
← TRUE B.P.T.

ELLIOTT and
LEHNHARDT

TECHNOLOGY BRANCH
MARKET STREET, CAMBRIDGE

0 10 20 30 40 50 60 70 80 90 100

PER CENT OVER



DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 10

°F

440

400

360

320

280

240

200

160

120

80

40

0

← ENGLER

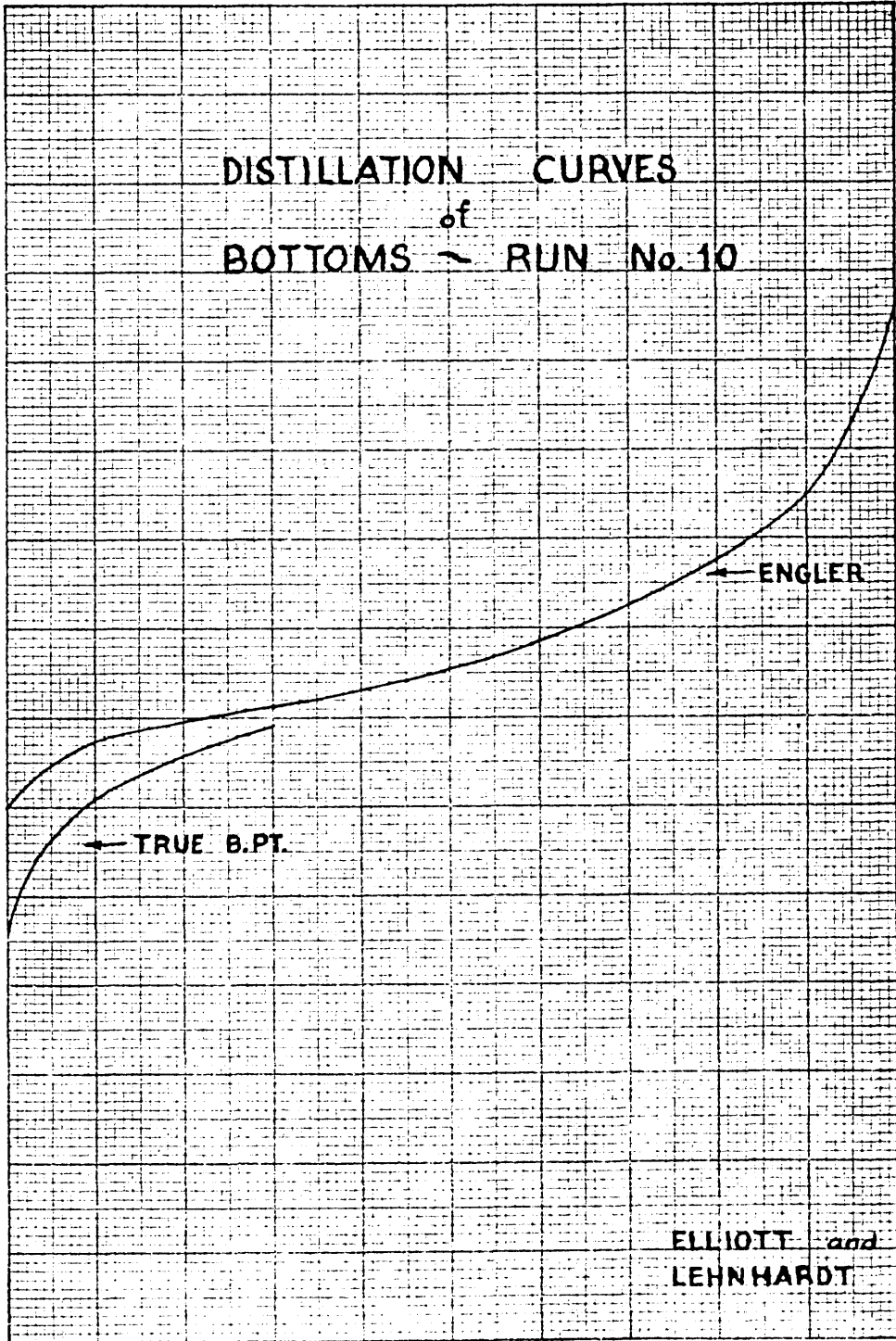
← TRUE B.P.T.

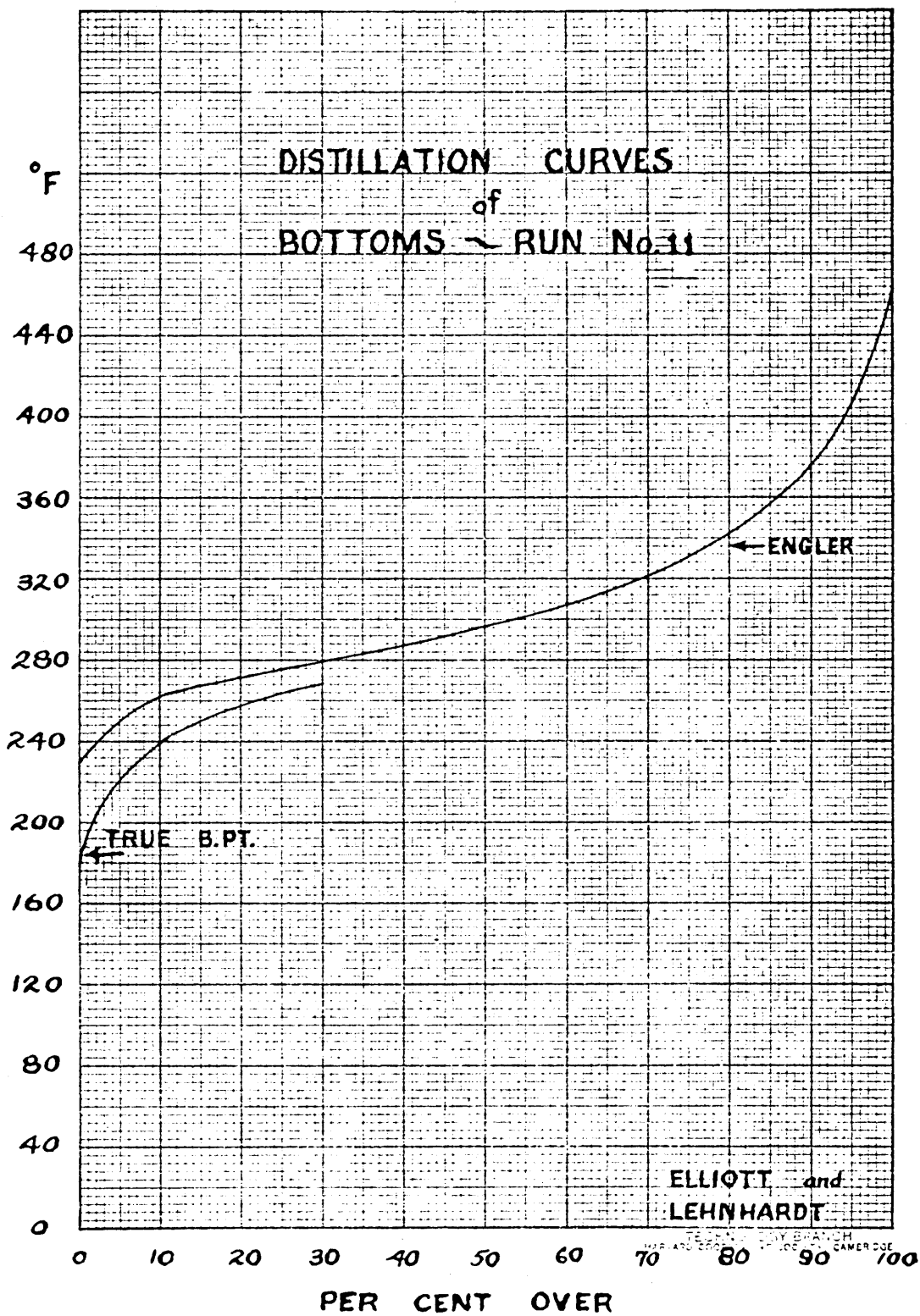
ELLIOTT and
LEHNHARDT

0 10 20 30 40 50 60 70 80 90 100

PER CENT OVER

TEMPERATURE IN °C
SCALE AND PERCENTAGE IN °C
SCALE





DISTILLATION CURVES
of
BOTTOMS ~ RUN No. 12

°F

440

400

360

320

280

240

200

160

120

80

40

0

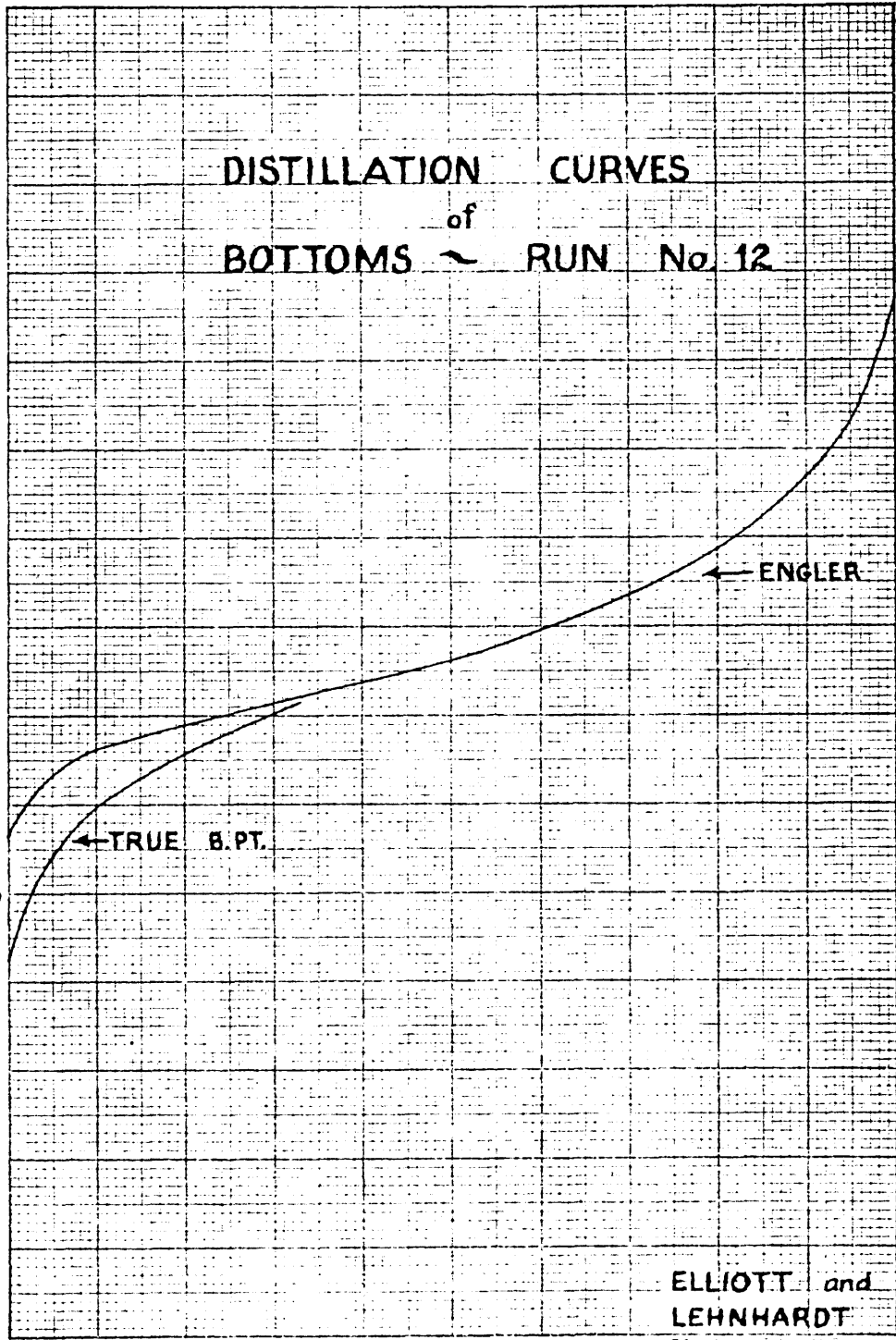
← ENGLER

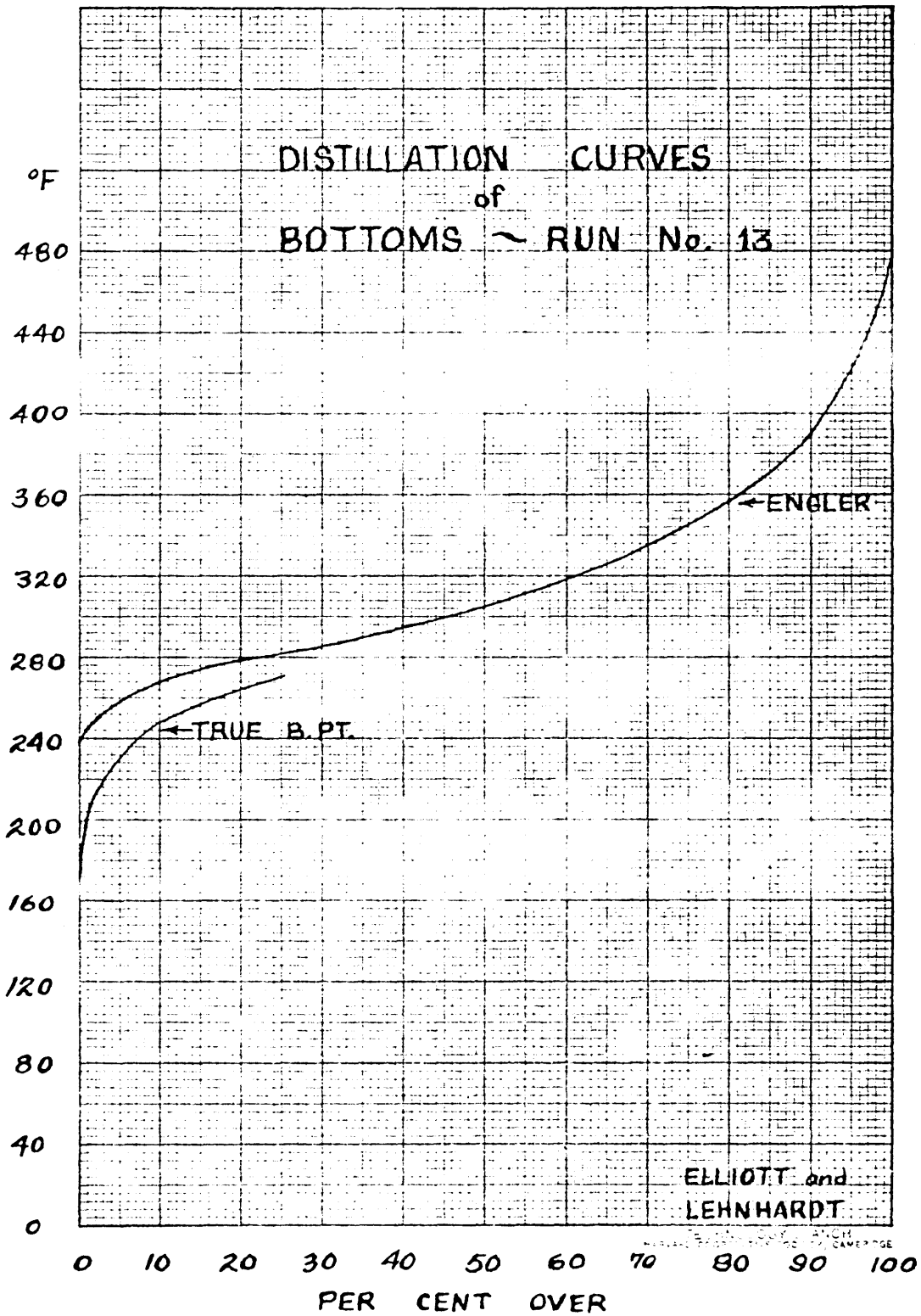
← TRUE B.P.T.

ELLIOTT and
LEHNHARDT

0 10 20 30 40 50 60 70 80 90 100

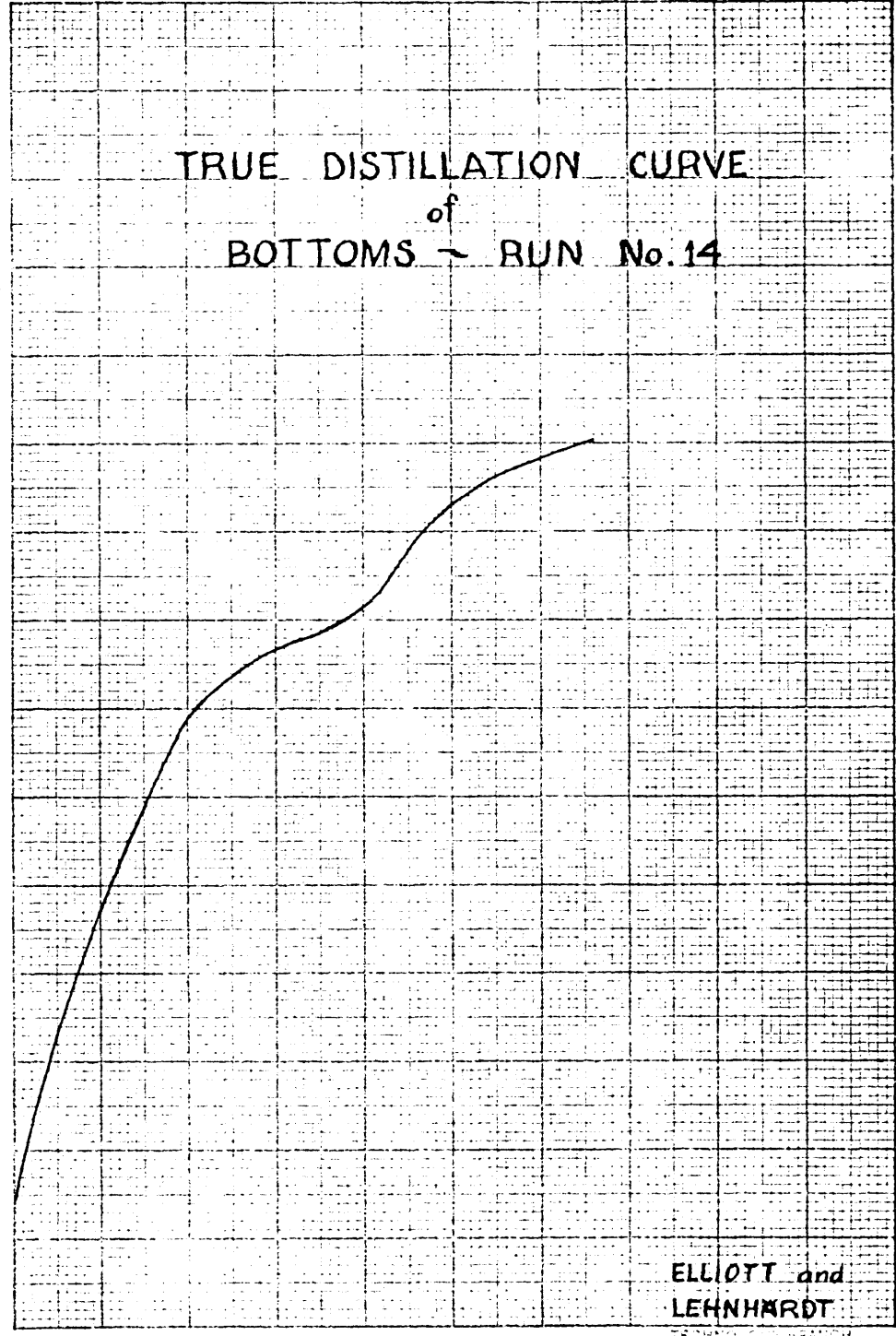
PER CENT OVER





TRUE DISTILLATION CURVE
of
BOTTOMS ~ RUN No.14

°F
280
260
240
220
200
180
160
140
120
100
80
60

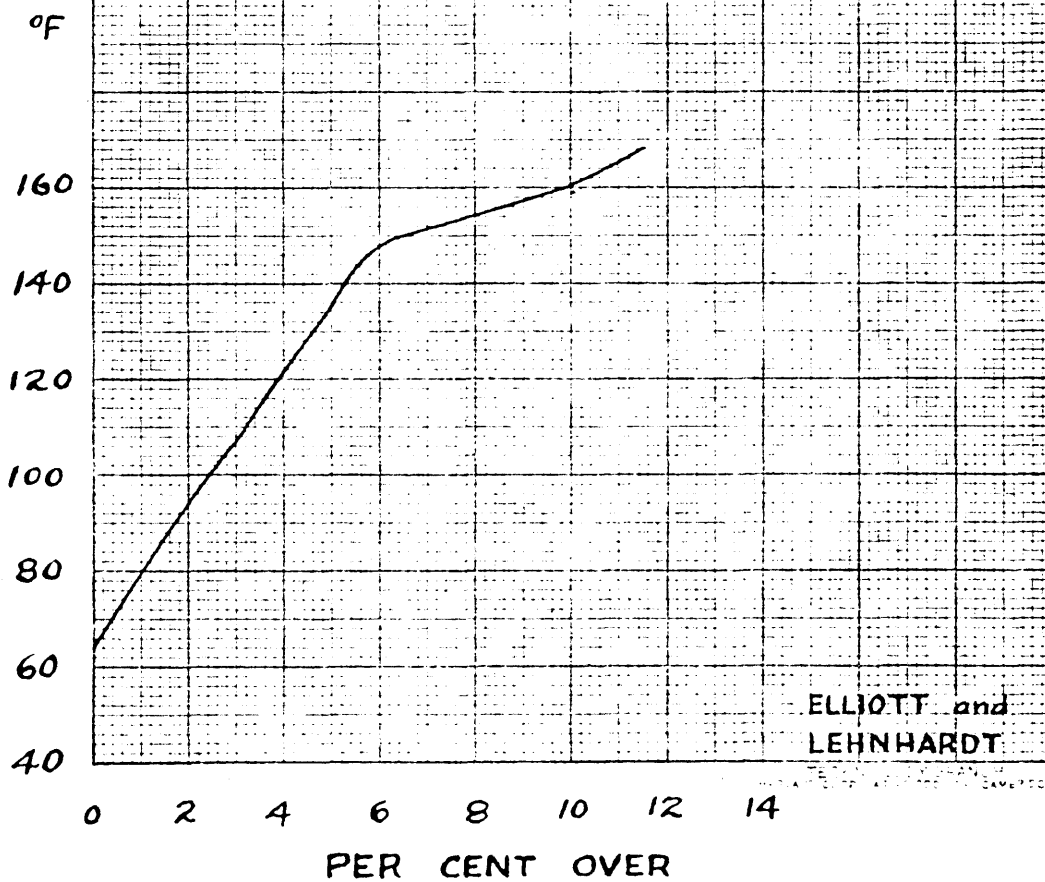


0 10 20 30 40 50
PER CENT OVER

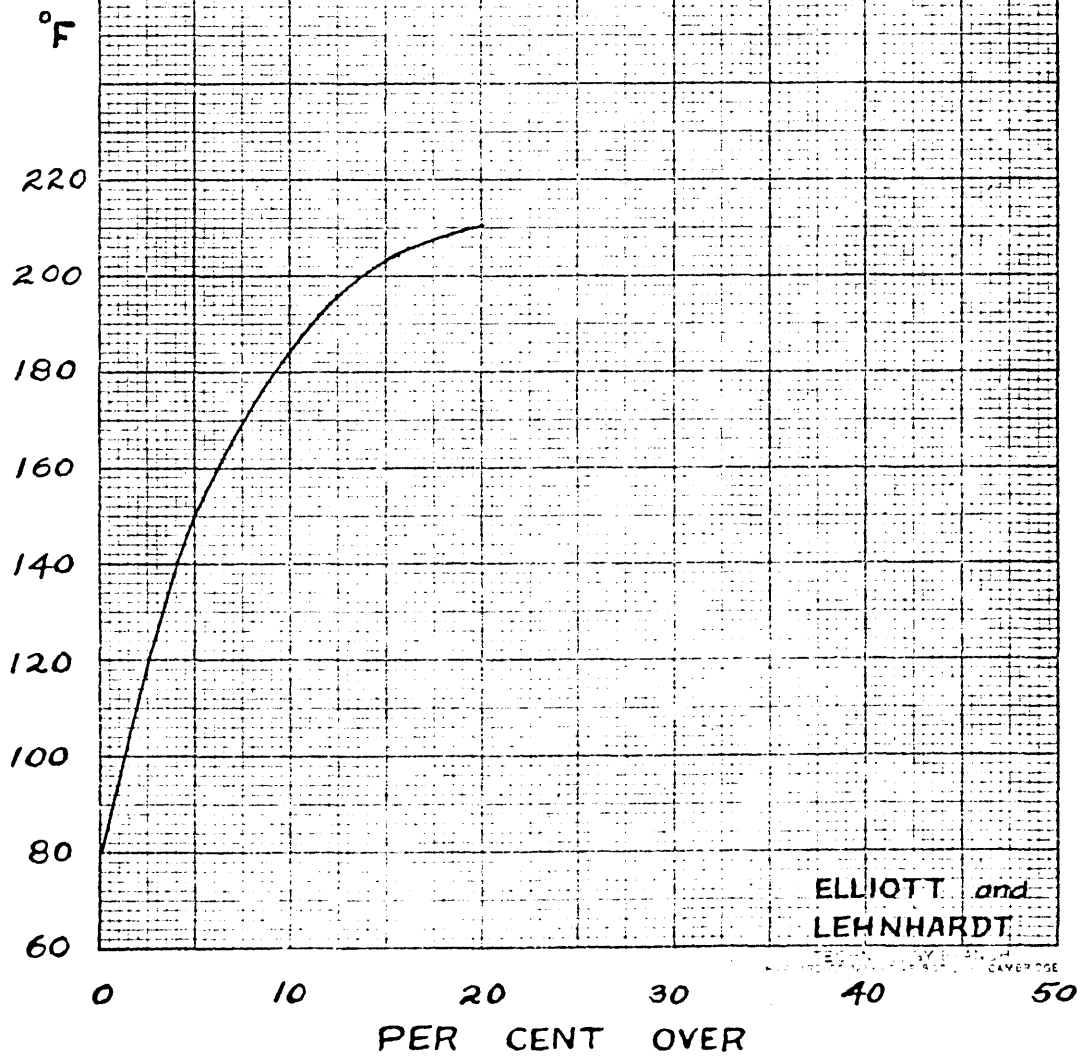
ELLIOTT and
LEHNHARDT

THE ENGINEERING EXPERIMENT STATION
MASSACHUSETTS INSTITUTE OF TECHNOLOGY, CAMBRIDGE

TRUE DISTILLATION CURVE
of
BOTTOMS - RUN No. 15



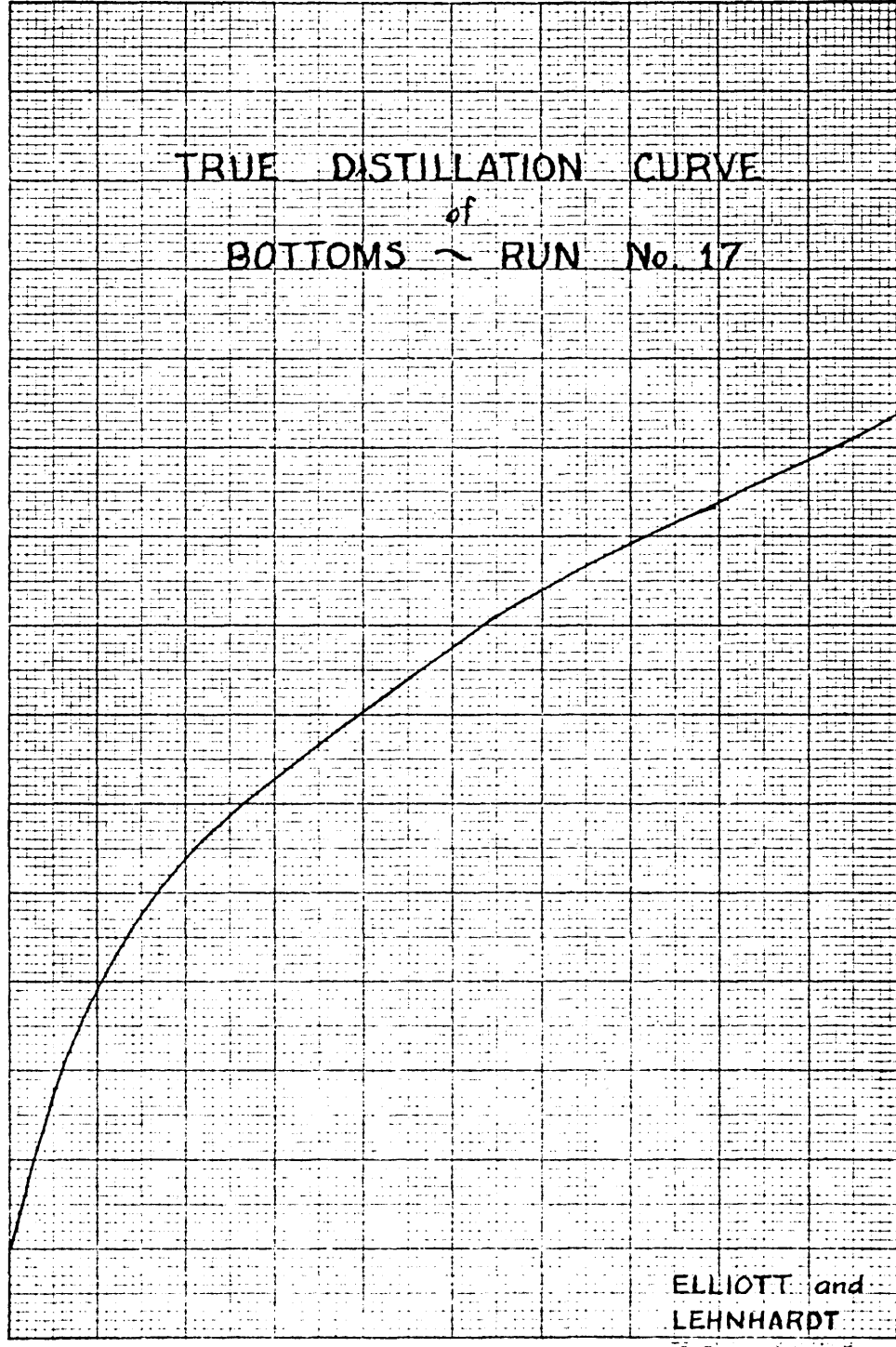
TRUE DISTILLATION CURVE
of
BOTTOMS ~ RUN No. 16



ELLIOTT and
LEHNHARDT

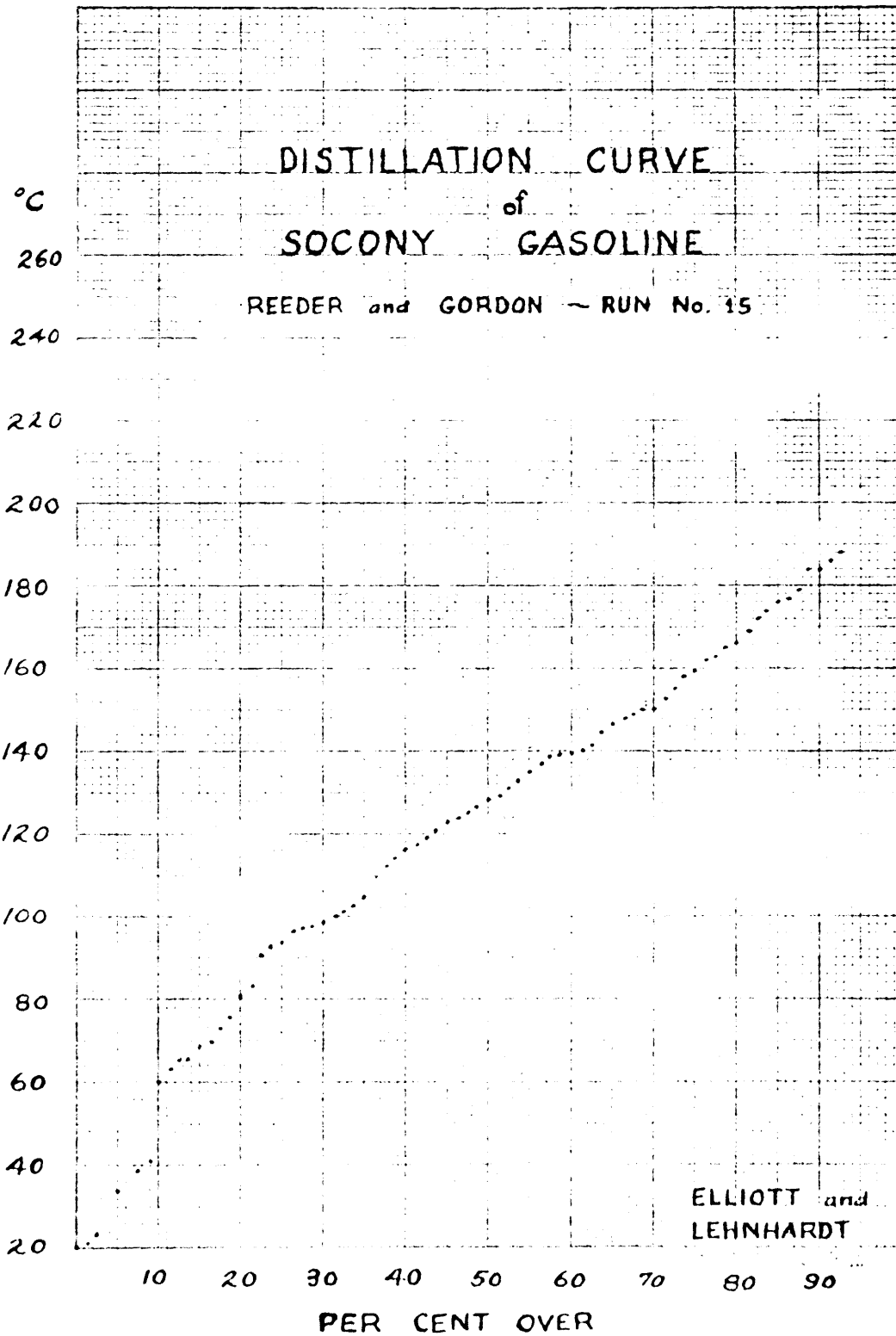
TRUE DISTILLATION CURVE
of
BOTTOMS ~ RUN No. 17

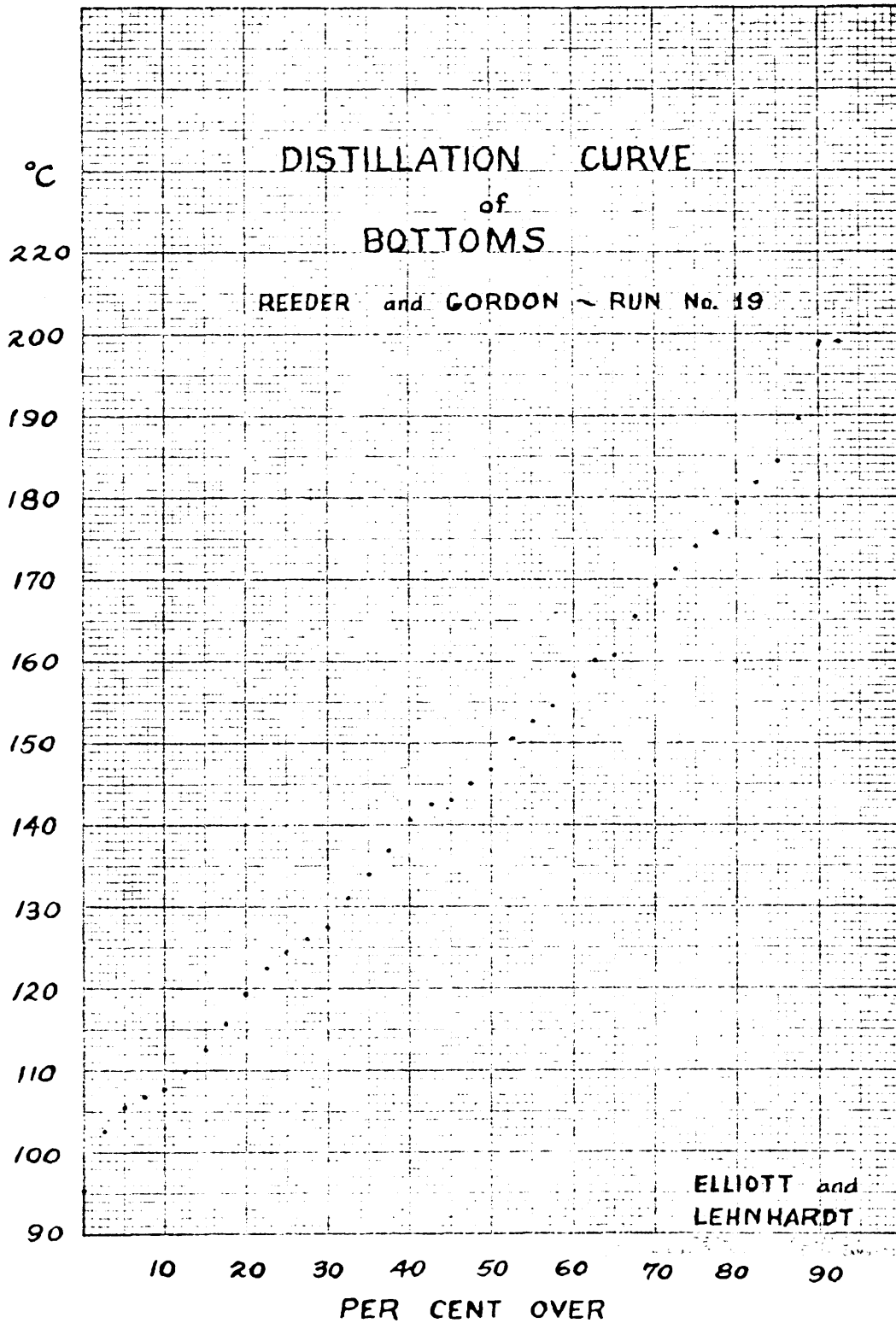
°F
320
300
280
260
240
220
200
180
160
140
120
100

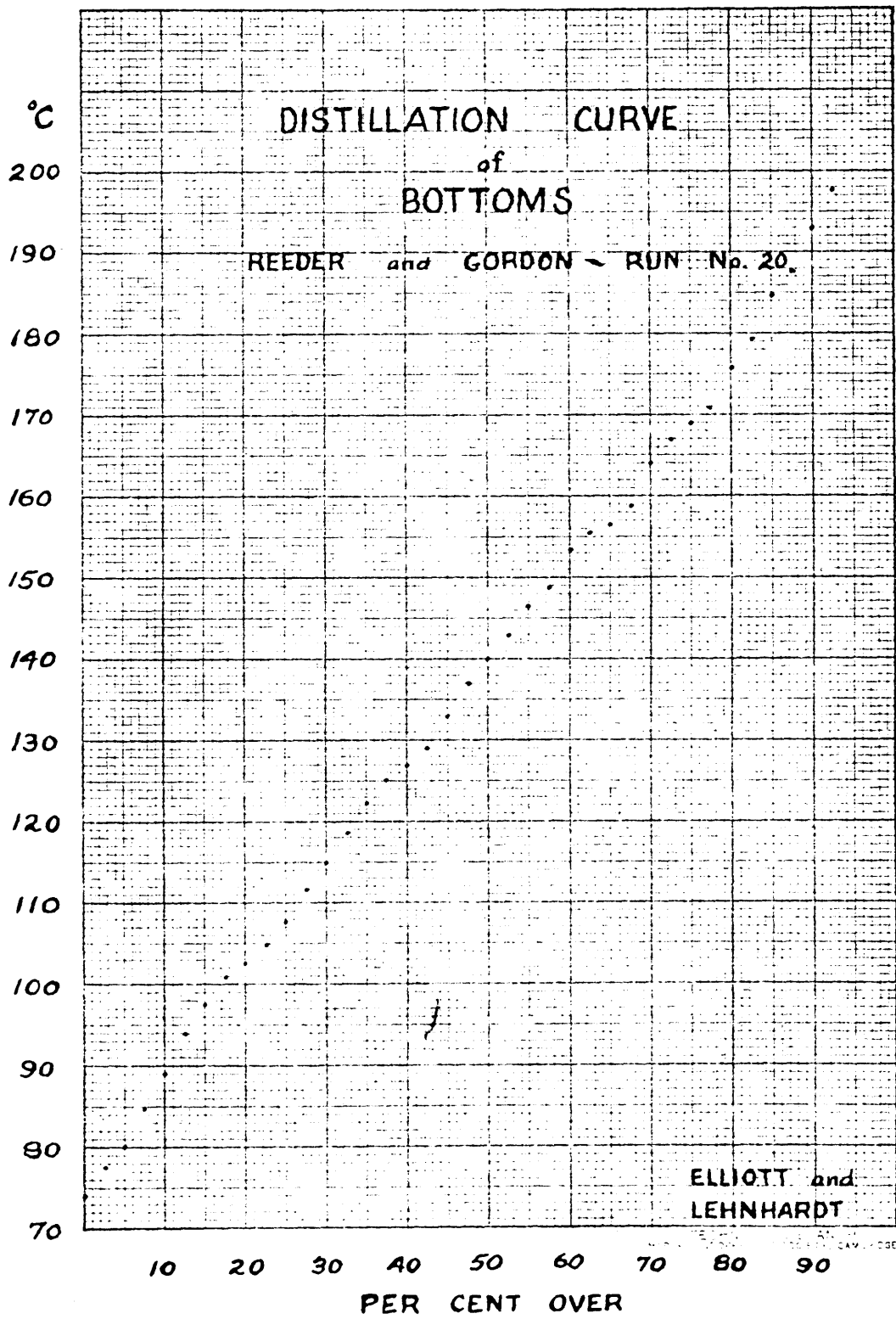


PER CENT OVER

ELLIOTT and
LEHNHARDT







RUN # 1

	Temperatures, degrees F.	Temperatures, degrees C.	Rates, c.c./min.
Column	Jacket	Inlet oil	Outlet oil
			Oil Dist.
186	189	---	---
			0
185	191		
			14.3
184	192		
			14.3
184	192		
			14.8
184	192		
			14.5
184	191		
			15
184	192		
			14.8
185	193		
			15
185	193		
			14.5
185	195		
			14.5
185	180		
			14.5
182	183		
			14.5
181	182		
			14.5
180	182		
			14
178	183		
			14.5
178	182		
			14
178	183		
			14.5
178	183		
			14
177	184		
			14.5

RUN # 1 Continued.

Temperature of feed ----- 78 F.
Temperature of still ----- 430--470 F.
Length of run ----- 69 min.
Specific gravity of distillate ----- 0.700 @ 60 F.
Specific gravity of bottoms ----- 0.780 @ 60 F.
Total volume of distillate ----- 1000c.c.@ 80 F.
Total volume of bottoms ----- 2325 c.c.@90 F.

RUN # 2

Column	Temperature, degrees F.		Temperature, degrees C.		Rate, c.c./min.	
	Jacket	Inlet oil	Outlet oil	Oil	Dist.	
174	176	---	---	0	12	
173	175				11	
171	179				11.5	
170	177				11	
169	178				11.5	
169	178				12	
168	178				12	
168	177				11	
167	177				11.5	
167	176				11	
167	175				11	
166	174				12	
166	176				12	
166	183				11	
166	170				11.5	
165	165				11	
164	170				11.5	

Temperature of feed ----- 76 F.
 Temperature of still ----- 485-510 F.
 Length of run ----- 70 min.
 Specific gravity of distillate ----- 0.685 @ 60 F.
 Specific gravity of bottoms ----- 0.780 @ 60 F.
 Total volume of distillate ----- 800 c.c.@80 F
 Total volume of bottoms ----- 2248c.c.@90F.

RUN # 3

Column	Temperature, degrees F.		Temperature, degrees C.		Rate, c.c./min.	
	Jacket	Inlet oil	Outlet oil	Oil	Dist.	
119	121	27.5	40	97	7.1	
119	122	27.5	40	97	6.5	
119	120	27.5	40	97	6.7	
118.5	119.5	26	40	87	6.7	
118.5	119.5	26	40	92	7.5	
118.5	119.5	26	39.5	92	7.5	
119	125	26	40	89	9.0	
119	120	26.5	39	92	8.0	
119	120	26.8	38	93	9.0	
119	120	26.7	38.2	92	8.0	
119	120	26.8	38.1	92	9.0	
119	119.5	26.8	38.3	92	9.0	

Temperature of feed ----- 76 F.
 Temperature of still ----- 284 F.
 Length of run ----- 62 min.
 Specific gravity of distillate ----- 0.63 @ 60 F.
 Specific gravity of bottoms ----- 0.770 @ 60 F.
 Total volume of distillate ----- 450 c.c.@72 F.
 Total volume of bottoms ----- 2588c.c.@90F.

RUN # 4

Temperatures, degrees F.		Temperatures, degrees C.		Rates, c.c./min.	
Column	Jacket	Inlet oil	Outlet oil	Oil	Dist.
148	147	56	59	167	11
147	148	55.5	58.5	----	--
147	146	56	58.5	170	10.3
145	145	56.2	58.5	160	10
144	146	57.5	59	170	10
143	143	63.5	64.5	500	--
143	143	64	64.5	800	10.3
143	144	63.5	64	752	10.4
142	141	60.8	61.4	780	10
141.5	142	55.8	57	520	10.1
142	148	53	55.5	140	--
142	140	50	54	200	--

Temperature of feed ----- 76 F.
 Temperature of still ----- 300 F.
 Length of run ----- 60 minutes
 Specific gravity of distillate ----- 0.64 @ 60 F.
 Specific gravity of bottoms ----- 0.775 @ 60 F.
 Total volume of distillate ----- 620 c.c. @ 65 F.
 Total volume of bottoms ----- 2195 c.c. @ 86 F.

RUN # 5

Temperature, degrees F.		Temperature, degrees C.		Rate, c.c./min.	
Column	Jacket	Inlet oil	Outlet oil	Oil	Dist.
174	178	68.2	70.5	320	17.5
175	172	70	72.5	340	--
177	178	69	72.2	340	18.5
178	178	69.5	72.5	350	18
178	180	69.7	72.9	350	18.5
179	178	70	73	330	15.5
178	178	70	74	320	15
179	180	70.5	74	330	15
180	180	67.6	72	330	18
183	181	65	71	320	17
182	183	65	71	330	19.5
184	184	65	72	330	20

Temperature of feed ----- 76 F.
 Temperature of still ----- 310 F.
 Length of run ----- 117 min.
 Specific gravity of distillate ----- 0.705@60 F.
 Specific gravity of bottoms ----- 0.790@60 F.
 Total volume of distillate ----- 2096c.c.@80 F.
 Total volume of bottoms ----- 3030c.c., 95F.

RUN # 6

Temperature, degrees F.		Temperature, degrees C.		Rate, c.c./min.	
Column	Wacket	Inlet oil	Outlet oil	Oil	Dist.
198	194	---	---	0	21
198	197.5				21.5
199	198				21.5
199.5	198				23.5
200	199				24
201	200				23
200	199				21
199	199				21.5
199	199				--
198	196				--
198	198				22
198	197				21.5
199	198				24
200	200				22.5
201	201				22.5
200	200				22

Temperature of feed ----- 75 F.
 Temperature of still ----- 308 F.
 Length of run ----- 120 min.
 Specific gravity of distillate ----- 0.71@60 F.
 Specific gravity of bottoms ----- 0.79@60 F.
 Total volume of distillate ----- 2570c.c.@70F.
 Total volume of bottoms ----- 2080c.c.@90F.

RUN # 7

Column	Jacket	Temperature,		Rate,	
		degrees F.	degrees C.	c.c./min.	
		Inlet oil	Outlet oil	Oil	Dist.
212	213	---	---	0	27
212	212				26.5
212	212				27
212	212				26.5
212	212				27
212	212				27
211	212				26.5
212	212				27
213	212				29
214	212				28.5
212	211				27.5
212	212				26.5
212	212				26.5
212	212				26.5
212	212				26
212	212				--
211	211				25
213	212				23.5
214	213				24
212	212				26
210	212				24

RUN No.7 continued.

Temperature of feed ----- 75 F.
Temperature of still ----- 335 F
Length of run----- 126 min.
Specific gravity of distillate ----- 0.730 at 60 F.
Specific gravity of bottoms ----- 0.770 at 60 F.
Total volume of feed -----4860 cc
Total volume of distillate ----- 2909 at 75 F.

RUN # 8

Column	Temperature, degrees F.		Temperature, degrees C.		Rate, c.c./min.	
	Jacket	Inlet oil	Outlet oil	Oil	Dist.	
202	201	---	---	0	23	
201	199				24	
201	202				23	
200	200				21.5	
200	200				21	
200	200				21.5	
200	201				21.5	
200	200				22	
199	200				22.5	
198	200				22	
196.5	199				22	
197.5	200				22	
198	200				22	
198	200				22	
198	200				21.5	
198	200				22	
198	199				22	
198.5	201				22.5	
199	199				22.5	
198.5	198				21.5	
198	198				22	
198	198				22	
199	199				22	

RUN # 8 Continued.

Column	Jacket	Temperature, degrees F.	Temperature, degrees C.	Inlet oil	Outlet oil	Rate, c.c./min.	Oil	Dist.
199	199			---	---	0		23
200	199							22.5
199.5	199							22.5
199	200							22
198.5	198							20.5
198	198							22
199	201							22
199	201							22
199	201							22
199	198							22
199	200							22
198	198							22

Temperature of feed----- 72 F.
 Temperature of still ----- 330 F.
 Length of run ----- 176 min.
 Specific gravity of distillate ---- 0.700 @ 60 F.
 Specific gravity of bottoms ----- 0.770 @60 F.
 Total volume of distillate ----- 3800 c.c.
 Total volume of feed---- -----6562 c.c.

RUN # 9

Column	Jacket	Temperature, degrees F.	Temperature, degrees C.	Inlet oil	Outlet oil	Oil	Rate, c.c./min.	Dist.
167	168			---	----	0		15
166	167							15.7
165	168							16.5
165	168							17
166	169							17
167	167							17
168	167							17
168	166							17
169	166							17
169	167							17
169	169							17
170	170							17
170	169							17
170	170							17
170	169							17
170	169							17
170	168							16.5
170	172							17
170	171							17
169	169							17

RUN # 9, Continued.

Temperature of feed ----- 75 F.
Temperature of still ----- 285 F.
Length of run ----- 97 minutes
Specific gravity of distillate ----0.700 @ 60 F.
Specific gravity of bottoms -----0.780 @ 60 F.
Total volume of distillate -----1650 c.c. @ 70 F.
Total volume of feed -- -----4200 c.c. .

RUN # 10

Column	Temperature, degrees F.		Temperature, degrees C.		Rate c.c./min.	
	Jacket	Inlet oil	Outlet oil	Oil	Dist.	
167	171	40	54.5	270	18	
167	170	39	51	260	17	
167	169	39	53	330	17	
167.5	169	39	53	320	17.5	
168	169	39	53	300	18	
168	169	39	53	290	18	
168	169	39	53	300	18.5	
169	169	39	53.5	290	18.5	
169	170	39	53.5	285	19	
169	171	40	54	340	19	
169	170	40	54	310	18.5	
169	169	40	53.5	300	19	
168.5	169	40.5	54	340	19	
168	168	41	53.5	340	19	
167	168	41	54	330	18	
166	167	40.5	56.5	320	18	
167	167	40.5	54.5	320	19	
168	167	40.5	54.5	340	19	
168	167	40.5	54.5	310	19	
169	169	41	55	340	19	
169	170	41	54.5	320	19	
170	169	41	55	310	18	

RUN # 10, Continued.

Temperature of feed -----	76 F.
Temperature of still -----	310 F.
Length of run -----	105.5 min.
Specific gravity of distillate -----	0.685 @ 60 F.
Specific gravity of bottoms -----	0.780 @ 60 F.
Total volume of distillate -----	1800c.c.@ 70F.
Total volume of feed -----	4530 c.c.

RUN # 11

Column	Temperature, degrees F.		Temperature, degrees C.		Rate, c.c./min.	
	Jacket	Inlet oil	Outlet oil	Oil	Dist.	
170	172	50.2	54	2200	17	
170	172	50.3	53.8	2040	17.5	
170	169	50.8	54	2050	17	
168	167	50.8	54	2000	17	
167	166	51	54.2	1800	17	
166	166	51	54	1900	17	
166	172	50.9	54	2200	17	
166	166	51	54	2000	18	
165	165	50.9	53.8	2000	18	
163	165	50.2	53.2	2130	17.5	
163.5	165	50.2	53	2200	17.5	
164	165	50	53.3	2000	17.5	
164	164	50	53.2	1950	17	
164	165	50	53	1950	17	
164	165	50.2	53.1	2100	17	
164	165	50.2	53.2	1900	17	
164	165	50.5	53.5	2080	17	
163	164	50.2	53.2	2000	17	
164	167	50.4	53.5	2000	17.5	
164	162	50.2	53.6	1800	17.5	
164	165	50.2	53.2	2000	17	

RUN # 11, Continued.

Temperature of feed -----	77 F.
Temperature of still -----	340 F.
Length of run -----	104 minutes
Specific gravity of distillate -----	0.685 @ 60 F.
Specific gravity of bottoms -----	0.775 @ 60 F.
Total volume of distillate -----	1700c.c. @70F.
Total volume of feed -----	4460 c.c.

RUN # 12

Column	Jacket	Temperature,		Rate,	
		degrees F.	degrees C.	Inlet oil	Outlet oil
164	160	---	----	0	18
164	160				17.5
164	161				17.5
164	161				17.5
165	164				17.5
166	166				17.5
166	163				17
166	162				17
166	162				17
167	167				17
167	163				17.5
166	165				17
165	164				17
165	165				17
165	165				17
166	165				17
166	163				17.5
166	164				17
166	164				17
165	163				17
165	165				17

RUN # 12, Continued.

Temperature of feed -----	73 F.
Temperature of still -----	290 F.
Specific gravity of distillate -----	0.685@60 F.
Specific gravity of bottoms -----	0.775@60 F.
Length of run -----	99 minutes
Total volume of distillate -----	1650c.c.@70F.
Total volume of feed -----	4260 c.c.

RUN #13

Column	Jacket	Temperature,		Rate,	
		degrees F.	degrees C.	c.c./min.	
		Inlet oil	Outlet oil	Oil	Dist.
167	166	49.4	66.2	125	18
167	166	49.5	65.5	125	18
167.5	167	49.5	65.5	125	18
167	166	49.5	65.5	130	18
167.5	168	49.5	65.5	130	18
168	167	50	65.5	130	18
168	168	49.8	65.4	132	18
168	167	49.2	64.5	135	18
167.5	167	48.8	64.0	140	18
166	167	47.5	62.5	140	18
166	166	47	63.0	140	18
165.5	165	47	62.4	140	17.5
165	165	47	62.5	140	17
164.5	165	48.3	62.8	140	17
165	166.5	49	62.8	145	17
165	166	49.5	63.5	145	17.5
167	167	50.2	64.5	150	18
167	167	50.4	64.8	147	17.5
167	167	50.4	64.5	147	18
168	168	51	65.0	150	18
169	169	50.5	66.1	137	18
169	167	50.2	65.6	140	17.5
169	167	50.2	64.8	143	18

RUN # 13, Continued.

Temperature of feed ----- 73 F.
Temperature of still ----- 305 F.
Specific gravity of distillate--- 0.690 @ 60 F.
Specific gravity of bottoms ----- 0.775 @ 60 F.
Length of run ----- 107 minutes.
Total volume of distillate ----- 11730c.c. @ 70 F.
Total volume of feed ----- 4580 c.c.

RUN #14 -----NO REFLUX

Temp.at top of flask F.	Dist.rate, c.c./min.	Temp.at top of flask F.	Dist.rate, c.c./min.
269	16	268	16
266	17	266	17.5
265	16.5	266	18
267	16	269	17
269	16.5	269	16
268	17	266	16
270	17	267	17
269	17		
269	17		
268	17		
272	17		
272	17		
265	17		
265	17		

Length of run ----- 106 minutes
 Total volume of feed ----- 4028 c.c. @ 73 F.
 Total volume of distillate ---- 1812 c.c. @ 80 F.
 Specific gravity of feed ----- 0.75 @ 60 F.
 Specific gravity of distillate--0.71 @ 60 F.
 Specific gravity of bottoms --- 0.77 @ 60 F.

RUN # 15 ----- NO REFLUX

Temperature at top of flask. F.	Distillate rate, c.c./minute.
236	10
236	12
234	11
234	12
235	10
236	10

Length of run ----- 27 minutes

Total volume of feed ----- 2700 c.c. @ 78 F.

Total volume of distillate---- 290 c.c. @ 88 F.

Specific gravity of feed ----- 0.75 @ 60 F.

Specific gravity of distillate 0.685 @ 60 F.

Specific gravity of bottoms -- 0.755 @ 60 F.

RUN # 16 ----- NO REFLUX

Temperature at top of flask. F.	Distillate rate, c.c./minute.
258	26
258	27
256	26
254	27
254	26
253	27
252	27
254	27
254	27
255	27
259	28
260	28

Length of run ----- 30 minutes
 Total volume of feed ----- 3000 c.c. @ 80 F.
 Total volume of distillate ----- 836 c.c. @ 88 F.
 Specific gravity of feed ----- 0.75 @ 60 F.
 Specific gravity of distillate - 0.695 @ 60 F.
 Specific gravity of bottoms 0.760 @ 60 F.

RUN # 17 -----NO REFLUX

Temp.at top of flask. F.	Dist.rate, c.c./min.	Temp.at top of flask. F.	Dist.rate, c.c./min.
2			
281	25	277	25.5
279	25	279	25.5
280	25	278	25.5
282	25	280	25
284	25.5	278	24.5
283	25.5	278	25
281	26	282	25
283	25	279	25
281	25	278	25.5
280	25.5	280	26
281	25	281	25
		279	25

Length of run ----- 135 min.

Total volume of feed ----- 5400 c.c. @ 80 F.

Total volume of distillate ----- 3412 c.c. @ 80 F.

Specific gravity of feed ----- 0.75 @ 60 F.

Specific gravity of distillate --- 0.725 @ 60 F.

Specific gravity of bottoms ----- 0.775 @ 60 F.

DATA FROM REEDER and GORDON, (M.I.T. THESIS 1922)

RUN # 19

Continuous Run on Socony Gasoline Using Plant Still

Final Temp*, degrees C.	Still Temp., degrees C.	Product, c.c.	Time, Min.
69.0	135.5	0	0
68.0	135.0	500	
67.0	134.0	1000	24
67.5	135.0	1500	
66.0	134.0	1800	40
		2000	

87 pounds of water in 16 min. enters reflux condenser at 12.5 C.

Average temp. of exit water ----40 C.

Total feed ----- 7000 c.c.

Total distillate ----- 2000 c.c.

Total bottoms ----- 4500 c.c.

Calculated reflux----- 19:1

* This temperature corresponds to the column temperature in our runs.

The distillation curves for these bottoms will be found with the other distillation curves in this report.

DATA FROM REEDER AND GORDON (M.I.T. THESIS, 1922)

RUN # 20

CONTINUOUS STILL USING SOCONY GASOLINE.

Final Temp*, degrees C.	Still Temp., degrees C.	Product, c.c.	Time, Min.
47	120.0	100	0
46.5	121.0	200	
46.5	120.1	600	
47.5	119.7	1000	282
48.5	118.0	1200	36
		1400	
		1700	

Rate of flow of water through reflux condenser between 100c.c. of product and 700c.c. over was 79.5 pounds in 17 minutes.

Calculated reflux ratio is 16.5:1

Eight liters of feed was run in continuously to the still. 1700c.c. was obtained as distillate. 6000c.c. was drawn out of the still as bottoms.

Average exit water temp.--32.6C. Inlet temp. not given.

* This temperature corresponds with the column temperature in our runs.

TRUE DISTILLATION OF SOCONY GASOLINE

2000cc charge		Temperature--degrees Centigrade	
Temp.	cc. over	Temp.	cc. over
31	10	79.5	296.5
33	25	83	315
34	35.5	84	328
35	41.5	85	343.5
36	47	87	359.5
37	52	86	363.5
38	59	87	367.5
39	65.5	88	371.5
39	72	89.5	379
40	76.5	89.5	387.5
40	81.5	91.5	398.5
42	88	92.5	411.5
44	94	93	423
48	101	93	426
58	113	92	430
65	140	94	439
67.5	169.5	94	444
69.5	195	96.5	458
70.5	219	97.5	483
71.5	227	99	506
73.5	244.5	99	517
75.5	266	101	537
76.5	274.5	102	561
77.5	282.5	102	571.5

Continued on following page

TRUE DISTILLATION OF SOCONY GASOLINE, continued.

Temp.	cc. over	Temp.	cc. over
102.5	582	136.5	1111.5
103.5	597	137.5	1130
105.5	615	139.5	1156
106.5	634.5	140.5	1172
109	680	141	1185
114	706	142	1199
115	725	142.5	1212
115.5	737	145	1239
116.5	750	147	1258
117	761.5	151	1303
118.5	775	152	1328.5
120	797	153	1350
121.5	814	158.5	1400
122.5	842	158	1435
123	863.5	161	1450
124	873	162	1472
125	890	162.5	1484
126	906	164.5	1492
127	925	167	1507
130	991	168.5	1519
130	1025	170	1527
134	1056	170.5	1544
135.5	1087	172	1564

Continued on following page

TRUE DISTILLATION OF SOCONY GASOLINE, continued

Temp.	cc. over	Temp.	cc. over
173	1581	180	1664
174	1597	184	1695
176	1610	184	1722
179	1635	186	1733

Engler Distillation of Remaining Fraction

Temp. F	Percent over
375	0
396	10
401	20
405	30
408	40
412	50
417	60
423	70
432	80
450	90
490	96.5

TRUE DISTILLATION OF BOTTOMS-- RUN #1

2000 c.c. charge

Temperature---degrees C.

Temp.	c.c. over	Temp.	c.c. over
52	0	126.2	430
90	26	126	451
97.5	64	127	472
98	85	128	490
99	102	129	507
101	126	130	521
103	147	131	539
105	159	132	571
106	175	133	590
105.5	181	134	611
106.5	199	134.5	621
109	225	135	640
111	244	137	665
111.5	260	137.5	681
112	267	139	713
114.5	287	139	728
116.5	305	139.5	737
118	324		
120	342		
120	355.5		
122	375		
124.5	407		

TRUE DISTILLATION OF BOTTOMS, --- RUN #2

2000 c.c. charge

Temperature ---degrees C.

Temp.	c.c. over	Temp.	c.c. over
27	0	116	398
57	8	120.5	442
85	27	123	480
92.5	60	123.5	508
96.5	104	124	526
96.5	124	124	542
98.5	147	124	553
99.5	169	125.5	571
100.5	189	127	593
101	211	128	616
101.5	222	127	624
102	235	129	637
102	245	131	663
102.5	253		
105	274		
107	294		
108.5	312		
110	325		
111.5	341		
113	357		
113.5	374		
114	382		

TRUE DISTILLATION OF BOTTOMS, ---- RUN # 3

2000 cc. charge

Temperature, --degrees C.

Temp.	c.c. over	Temp.	c.c. over
69	36	91	214
70	52	92	226
71	74	93	235
71.5	91	94.5	244
79.5	129	95	261
83	138	96	275
84	150	96	281
85.5	164	97	300
86.5	181	100	327
89.5	192	99	340
90	202		

TRUE DISTILLATION OF BOTTOMS, RUN #4

2000 c.c. charge

Temperature, ---degrees C.

Temp.	c.c. over
71	5
82.	38
92	77
93	100
94	123
95	140
95	150
95.5	160
96	169
96.5	178
97.5	185
98	200
100	228
100.5	250
101.5	273
102	285
103	300
106	336
107	350

TRUE DISTILLATION OF BOTTOMS, RUN #5

2000 c.c. charge

Temperature, ---degrees C

Temp.	c.c. over	Temp.	c.c. over
105	25	127.5	292.5
112	85	128	307
113	92	128.5	319
112	105	128.5	329
113	118	130	345
114	128	131.5	385
115.5	139	133	419
116	150	134	441
117	167	137	475
119	181	139	535
121	197	139.5	567
122	218	140	608
123	227	140.5	634
124	244	141	650
126	265	142	689
127	277	143	709

TRUE DISTILLATION OF BOTTOMS, RUN #6

2000 c.c. charge

Temperature, --degrees C.

Temp.	c.c. over	Temp.	c.c. over
118.5	15	148	409
128	50	148.5	428
131	86	148	438
132	103	148.5	452
133	119	148	467
134.5	140	149	478
136	157	149.5	500
137.5	177	148.5	512
138.5	192	148.5	531
139	204	150.5	560
139.5	215	150	587
141	228	150.5	592
143	250	151	615
144.5	281	151	630
146	311	151	641
147	344	151	673
148	377	152	690
148	400		

TRUE DISTILLATION OF BOTTOMS ----RUN No.7

1290 c.c. charge

Temperature----degrees C.

Temp.	c.c. over	Temp.	c.c. over
129	20	152	188
133	30	152.5	200
	4		
136	40	153.5	220
137.5	50	155.5	250
140	63	156	271
140.5	75	157	292
143	88	157.5	300
145.5	100	158.5	321
147.5	115	159	335
149	128	159.5	350
150.5	150	160.5	370
151	165	161	385

TRUE DISTILLATION OF BOTTOMS, RUN #10

2000 c.c. Charge

Temperature, ---degrees C.

Temp.	c.c. over	Temp.	c.c. over
93	28	122.5	264
94	31	123	283
96	34	123.5	300
100	38	124	317
105	62	125	343
107.5	98	125.5	352
107.5	99	126	373
106	102	128	400
106	103	129	420
110	117	130	441
111.5	129	130.5	464
113	141	131	482
115.5	155	131.5	500
117	176	132.5	521
117	183	133	532
118	207	133	545
119	219	134	557
120	233	135	576
121.5	250	137	600

TRUE DISTILLATION OF BOTTOMS, RUN #11

2000 c.c. charge Temperature, ----degrees C.

Temp.	c.c. over	Temp.	c.c. over
85	10	119	269
93	19	120.5	283
99.5	35	122	300
102.5	50	122	323
105	70	123	348
107	94	124.5	388
108.5	120	125.5	403
108.5	130	125.5	431
108	140	127.5	459
108	143	128.5	489
109	150	129	518
110.5	167	130	541
113	185	130	558
115.5	200	131	575
117.5	232	131	600
118.5	250		

TRUE DISTILLATION OF BOTTOMS, RUN #12

2000 c.c. charge Temperatures, ----degrees C.

Temp.	c.c. over	Temp.	c.c. over
97	45	121	290
98	55	121.5	297
98.5	85	123	300
101	95	124	330
103.5	102	124.5	335
107	118	125.5	359
107	128	125	393
105	135	129	402
106.5	139	129.5	421
108.5	145	132	455
109.5	150	132	479
113.5	165	131	492
114.5	182	137	530
115.5	197	137	557
118	228	137.5	585
119	240	137.5	600
120.5	259	139	616
120	272	139.5	627
120	281	141	650

TRUE DISTILLATION OF BOTTOMS, --RUN #13

2000 c.c. charge Temperature, --degrees C.

Temp.	c.c. over	Temp.	c.c. over
97	33	122	243
102	50	122	255
103	62	124	280
106	69	124.5	293
108	84	125.5	311
108	90	124	327
109	97	124.5	335
111	109	125	344
112.5	136	126	350
116	150	128	385
117	171	130	450
117.5	187	130	466
119	200	130.5	482
120.5	223	132.5	500
121	236	132	513

TRUE DISTILLATION OF BOTTOMS, RUN #14

2000 c.c. charge

Temperature, ---degrees C.

Temp.	c.c. over	Temp.	c.c. over
32	0	102.5	322
40	21	103	333
45	34	101.5	342
54	50	103.5	363
57	64	104	373
61	81	105.5	385
64	90	105.5	392
69	105	106.5	408
72	118	108.5	415
77	137	110.5	429
79	145	114	450
82	157	117	474
86	171	118	482
89	182	118.5	494
91	193	119.5	508
93.5	208	120	521
95	225	121.5	530
95	240	122.5	550
98	258	123	564
100	280	124	577
100	293	124.5	587
100.5	305	125	600

TRUE DISTILLATION OF BOTTOMS, RUN # 14 Cont.

Temp.	c.c. over
126	622
127	634
127	645
127.5	650
127	662
129	690
131.5	717
132.5	732
133.5	750
135	768
135.5	778

TRUE DISTILLATION OF BOTTOMS, --RUN # 15

2000c.c. charge.

Temperatures, ----degrees C.

Temp.	c.c. over	Temp.	c.c. over
31	15	65.5	130
36	25	66	135
38	39	66.5	140
39.5	50	67.5	150
44	68	68.5	160
47	75	70	175
51	88	71	195
56.5	96	72	200
60	104	73.5	212
62	110	74	215
63.5	115	74	220
64.5	120	75	225
65	125	76	230

TRUE DISTILLATION OF BOTTOMS, --RUN # 16.

20006.c.charge		Temperatures, ----degrees C.	
Temp.	c.c.over	Temp.	c.c.over
36	20	91	250
41	36	93	270
43	45	94	285
50	55	95	300
64	78	96.5	325
68.5	102	97.5	350
71	125	98	365
74.5	150	98.5	375
76	170	98.5	380
84	196	99	385
89	228	99.5	390
90	238	100	400

TRUE DISTILLATION OF BOTTOMS, ---RUN # 17.

2000c.c.charge Temperatures, --degrees C.

Temp.	c.c.over	Temp.	c.c.over
52	10	133	650
69	50	137	700
79	100	140.5	750
94.5	150	143.5	800
98.5	200	145	825
101.5	250	146	875
105.5	300	147	890
111.5	350	147	900
116	400	148	918
118.5	450	149	937
124	500	150	950
128	550	153	992
130.5	600	153.5	1000

Engler Distillation of Bottoms

Percent over	Run #	Temperature				
		1	#2	#3	#4	#5
0		221	207	188	208	240
10		256	244	218	240	273
20		265	255	231	251	283
30		286	265	243	261	291
40		286	276	257	273	300
50		299	289	271	285	310
60		213	304	288	301	321
70		330	320	307	319	335
80		350	342	328	340	355
90		381	376	364	375	386
97	end	---	425	---	428	434
97.5	point	432	---	418	---	---

Engler Distillation of Bottoms

Percent over	Temperature			
	Run #6	#7	#8	#9
0	260	296	284	236
10	290	318	304	270
20	301	326	310	278
30	308	332	315	286
40	312	338	323	297
50	325	346	330	308
60	334	354	338	319
70	346	368	350	336
80	364	381	367	357
90	390	406	395	388
97.5	433	446	441	434

ENGLER DISTILLATION OF BOTTOMS

Percent over	Temperature F.			
	Run, #10	#11	#12	#13
0	241	230	227	238
10	270	262	266	268
20	278	270	275	278
30	285	278	283	285
40	293	287	294	295
50	302	296	304	305
60	316	307	318	318
70	330	322	334	335
80	349	341	354	356
90	381	375	388	390
97.5	437	433	436	446

Engler Distillation of Socony Gasoline

Percent over	Temperature F.	
	#1	#2
0	123	120
10	173	170
20	200	198
30	222	222
40	240	242
50	260	259
60	280	279
70	300	301
80	325	326
90	362	364
97.5	418	421

IX B I B L I O G R A P H Y.

1. Walker, Lewis, and Mc Adams---"Principles of Chemical Engineering".
2. W. K. Lewis----J.I.E.C. , 1 , 522-33
3. W. K. Lewis----J.I.E.C. , 14 , 6 , June 1922.
4. Peters----J.I.E.C. , 14 , 467-79 , (1922)
5. C. S. Robinson---"Elements of Fractional Distillation".
6. Sidney Young---"Distillation Principles and Processes".
7. Leslie---"Motor Fuels".
8. Bacon and Hamor---"Petroleum".
9. S. F. Dufton--- "The Limits of Separation By Partial Distillation" , J. Soc. Chem. Ind., 38 , pp. 45-6 (1919).
10. Reeder and Gordon---M. I. T. Thesis, 1922.
11. Mc Farland and Mc Grath---M. I. T. Thesis, 1924.
12. Davis---"True Boiling Point Curve", Research Lab. Appd. Chemistry, M. I. T.
13. Huggins---M. I. T. Thesis, 1922.
14. Mackie---M. I. T. Thesis, 1924.
15. R. E. Wilson, W. H. Bahlke--"The Physical Properties of the Paraffin Hydrocarbons", J.I.E.C., Feb. 1924.
16. R. E. Wilson, D. P. Barnard IV--"Sensible Heats of Motor Fuels", J.I.E.C., 13 , 912-5 (1921).
17. Dean et al.---"The Analytical Distillation of Petroleum and Its Products" , U. S. Bul. Mines No. 207, (1922).