

Jan. 25, 1949.

J. D. LONG ET AL
METHOD AND APPARATUS FOR COUNTER-CURRENT
CONTACTING OF LIQUIDS

2,460,019

Filed Aug. 23, 1944

3 Sheets-Sheet 1

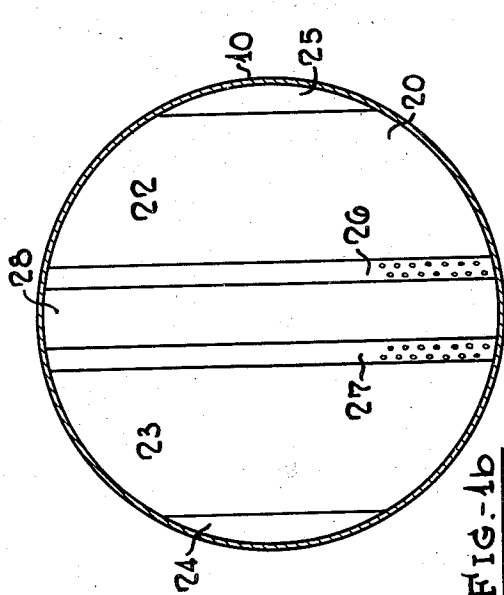


FIG. 1b

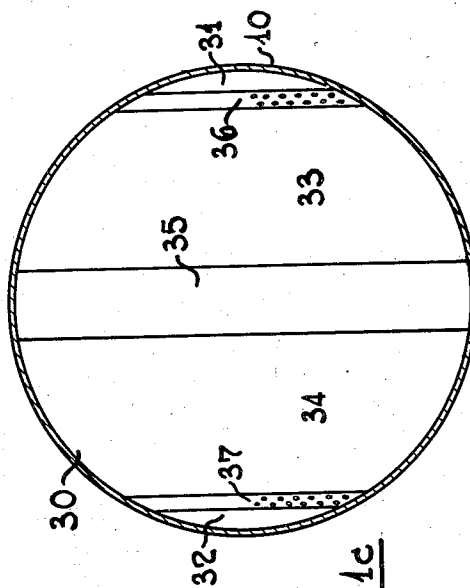


FIG. 1c

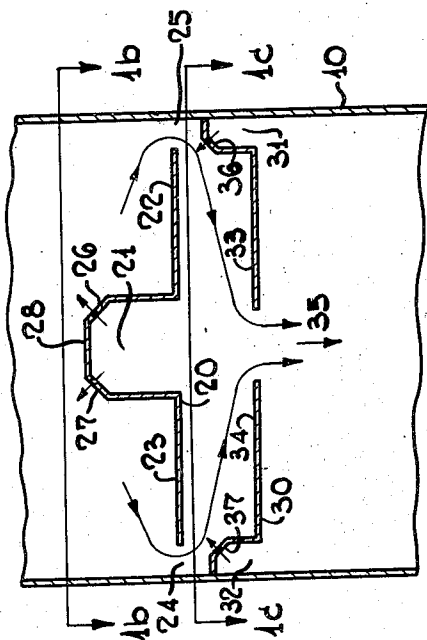


FIG. 1a

John D. Long
Cyril O. Rhys, Jr. Inventors
By P. H. Young Attorney

Jan. 25, 1949.

J. D. LONG ET AL
METHOD AND APPARATUS FOR COUNTER-CURRENT
CONTACTING OF LIQUIDS

2,460,019

Filed Aug. 23, 1944

3 Sheets-Sheet 2

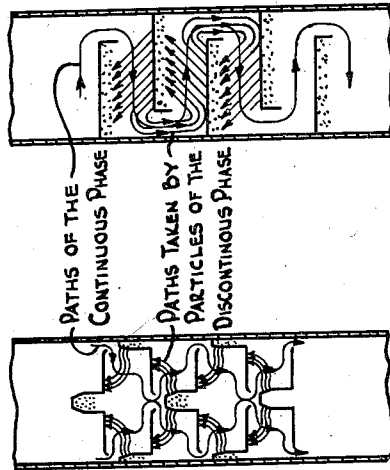


FIG.-2b

FIG.-2a

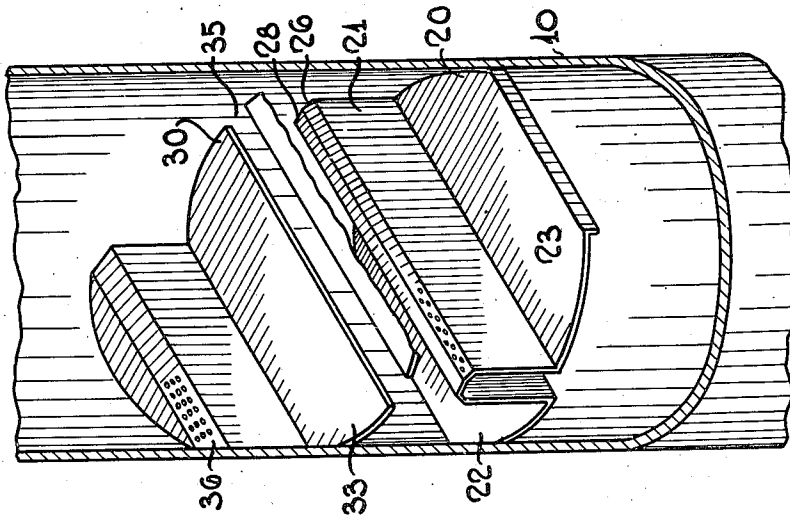


FIG.-1d.

John D. Long
Cyril O. Rhys, Jr. Inventors

By C. P. Young Attorney

Jan. 25, 1949.

J. D. LONG ET AL
METHOD AND APPARATUS FOR COUNTER-CURRENT
CONTACTING OF LIQUIDS

2,460,019

Filed Aug. 23, 1944

3 Sheets-Sheet 3

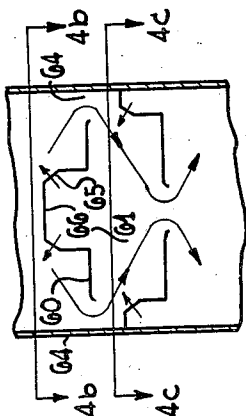


FIG.-4a

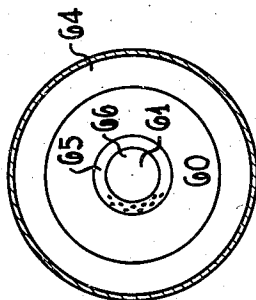


FIG.-4b

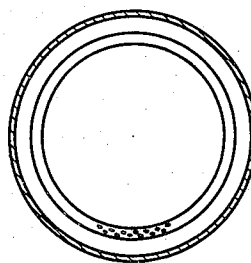


FIG.-4c

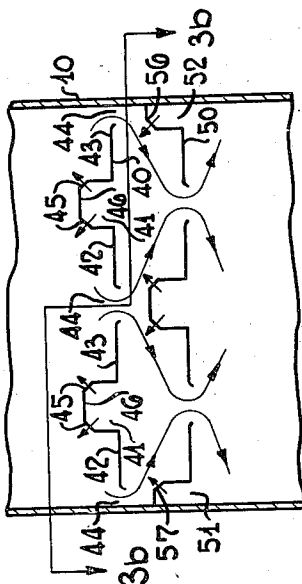


FIG.-3a

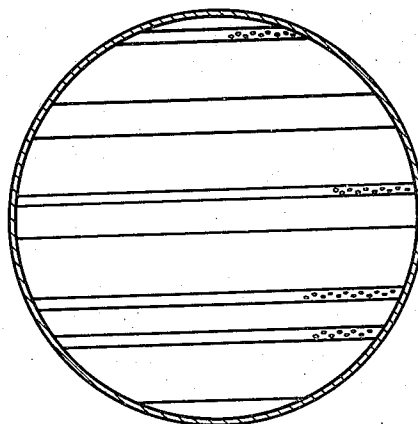


FIG.-3b

John D. Long
Cyril O. Rhys, Jr. Inventors

By P. H. Young Attorney

UNITED STATES PATENT OFFICE

2,460,019

METHOD AND APPARATUS FOR COUNTER-CURRENT CONTACTING OF LIQUIDS

John D. Long, Westfield, and Cyril O. Rhys, Jr.,
Cranford, N. J., assignors to Standard Oil De-
velopment Company, a corporation of Delaware

Application August 23, 1944, Serial No. 550,798

8 Claims. (Cl. 23—270.5)

1

This invention relates to the countercurrent contacting of fluids, and in particular to the countercurrent treatment of substantially immiscible liquids of different gravities.

In the art of intercontacting fluids for refining purposes, many processes have been devised which are dependent in some measure upon the design of the apparatus employed. It has been found, for example, more advantageous to effect large volume inter-fluid treating, especially liquid-liquid contacting, in towers rather than in mixers and settlers (or centrifuges). Processing in towers has been found more advantageous from the economic viewpoint because of the lower initial and operating costs, the lower steel requirements and the less ground space required. Considerable attention has therefore been given to liquid-liquid contacting in towers. It is desirable to achieve high capacity under good contacting conditions in the towers so that superior product quality and high processing yields may be attained. The efficiency of treating in the towers depends, therefore, upon the attainment of good phase contacting without the formation of stabilized emulsions. Of the various towers employed, those involving the use of pierced plates have been particularly advantageous when large quantities of liquids are involved.

Conventional pierced plate towers consist of a large number of horizontally disposed perforated plates extending over a large part of the tower area. The plate perforations are small orifices by means of which one of the phases is dispersed.

When the heavier liquid is passed downward through the tower as the discontinuous phase, each plate is provided with risers for the passage upward of the lighter continuous phase and which form a weir or weirs of sufficient height for the heavier phase to insure a definite head on the plate to effect, by reason of gravity differential, the dispersion of the heavier liquid into the lighter liquid. The intercontacting of liquids in pierced plate towers under the given flow conditions is thus the dispersion vertically of one phase from the plate orifices into the continuous phase which is moving in an essentially horizontal direction beneath the plate to reach the location of the nearest weir. Such treating conditions have been found generally satisfactory

2

except in regard to the development of local areas of recycling of the disperse phase especially when the tower is being used above a certain capacity, that is, near flood conditions. Processing according to the invention practically eliminates such disadvantages.

The settling rate of the disperse phase in a conventional pierced plate tower clearly decreases as the throughput and consequently as the velocity of the continuous phase increases. As a result therefore of conventional pierced plate tower operation, the disperse phase issuing from the orifices nearest to the weirs may be carried into the adjacent settling zone by means of the horizontally moving stream of the continuous phase and then recycled to the plate from which such portions of the disperse phase had previously come. Furthermore, this tendency towards recycling increases as the throughput of the continuous phase increases. Thus, as the flow of the continuous phase over such pierced plates is increased beyond a certain value, the particularly advantageous capacity of the tower, correspondingly poorer and poorer treating effects are obtained.

In an effort to modify the conventional pierced plate tower to avoid recycling, it was found that by blanking the orifices on the side of the plates nearest to the weir or weirs an improvement in contacting conditions was obtained. It was further found that by moving the weirs back to a point close to the pierced section and building the plate in the form of two steps with the weir as a riser and the spacing of the plates widely apart a further improvement could be attained. This change also involved a change in the settling conditions from that of settling from horizontally moving streams of the continuous phase to settling from substantially vertically moving countercurrent streams. From a contacting as well as from a settling viewpoint, this change was found advantageous. Thus, the plates in the tower could be spaced further apart and on the plates narrow and deeper troughs could be constructed, thereby permitting a higher head than previously of the phase to be dispersed.

According to these principles, as the basis of the present invention, an embodiment or illustration of the invention may be taken in the design of a particular treating tower. In this tower the

design involves having a series of plates containing troughs for the collection of the heavier discontinuous phase. These troughs have their bases drilled with holes through which the phase which collects therein becomes dispersed into the space below, particularly by reason of the liquid head maintained in the troughs. By adjusting the size and number of the holes in the troughs, it has been found possible to obtain various sized droplets and thus to determine the intimacy of contact between the phases in the tower. The design of the troughs in the various plates clearly determines the extent of the liquid head necessary to force the phase to be dispersed through the holes in the bases. The droplets which are sprayed from the holes in the troughs are moreover settled through a zone in which the lighter phase has to flow in an essentially vertical direction. The droplets of the heavier phase coalesce on the top of the next lower plate and then flow into the trough or troughs in that plate. The lighter phase passes upwards in the tower through the plate openings.

In a tower of such design, as the velocity of the continuous phase moving in a substantially vertical direction increases, the settling rate of the discontinuous phase therein decreases, and at a certain maximum velocity of the discontinuous phase the majority of the droplets will no longer settle but will be entrained along with the continuous phase; that is, the point of flooding of the tower. Such processing is thus in marked contrast to that which normally occurs in the conventional type pierced plate towers. Moreover, as the throughput in towers designed according to the present invention increases, the entrainment does not become progressively greater until flooding occurs provided that droplets of relatively uniform size are produced. Moreover, as the throughput in such towers increases, the amount of time available for each droplet in the settling zone increases, thus conveying improvement in treating efficiency.

In such an apparatus, any degree of contacting may be obtained by varying the size and number of the holes in the troughs, and, since the size of the droplet determines the settling rate of the discontinuous phase, the rate of settling in the discontinuous phase is also controlled. The diameter of such a tower necessarily becomes dependent upon the flow rates and degree of contacting required, while the height of the tower will be determined by the number of plates required for the extraction operation; the latter also being determined by the number of theoretical extraction stages required and the plate efficiency.

In order to understand more clearly the advantages of processing according to the invention and the improved plate efficiency also involved, the mechanism of material transfer in liquid-liquid contacting systems will be further analyzed. In the contacting of the two phases, one of the most important features in the interchange of soluble constituents is the range of distribution coefficients of the various solutes between the various solvents. Plate efficiency is dependent also upon a number of variables. These variables include the solute concentration differential between the liquid phase, the rates of diffusion of the solutes in each phase, the size of the droplets and the time available for diffusion. Of these variables, only the latter two are subject to control by tower design. The size of the droplets is mainly a function of interfacial tension, orifice size and pres-

sure drop across the orifices. The latter two can be varied at will regardless of the type of plate construction, although from practical consideration the vertical type controller is more advantageous with higher pressure drop than with the conventional pierced plate type.

As regards the time available for diffusion, settling from substantially countercurrent vertically moving streams has, as previously pointed out, distinct advantages over settling from horizontally moving streams. Also, only the largest droplets of the disperse phase can pass through horizontally moving streams of the continuous phase when the velocity is relatively high, the droplets of smaller size being subject to the carrying power of the continuous phase and thus to recycling. Thus, in the conventional pierced plate tower, as the maximum capacity is reached plate efficiency falls off sharply due to recycling and to the rapid passage of only large droplets through the continuous phase. On the other hand, in towers designed according to the present invention, as the tower approaches maximum capacity the time of contacting for all droplets increases and hence the plate efficiency also tends to approach a maximum. This is of great importance for reasons of economy since it is always desirable to operate towers at the maximum rates.

The invention may be specifically illustrated by the processing effected in an apparatus diagrammatically illustrated in Figures 1a, 1b, 1c and 1d. Figure 1a represents a section of a tower 10 according to the invention containing two plates 20 and 30, respectively. Figures 1b and 1c represent plan views of the plates shown in Figure 1a. Figure 1d represents a cutaway section of the tower showing the same two plates. Figures 2a and 2b show the flow of fluids according to processing of the invention and in a conventional pierced plate tower. Figures 3 and 4 represent other modifications of the invention.

As shown in Figures 1a and 1b, plate 20 consists of an inverted trough 21 extending across the center of the tower and two symmetrical flat horizontal plates 22 and 23 extending in both directions from the trough edges almost to the shell of the tower. The spaces 24 and 25 between the edges of the plate and the shell of the tower 10 serve as passageways for the flow of the continuous phase. The trough 21 has angle sides 26 and 27 and may be capped by the horizontal plate 28. The angle sides 26 and 27 are perforated to act as orifices for producing droplets of the phase to be dispersed. The plate 30 is positioned directly below the plate 20 as shown in Figure 1a and consists of two short inverted troughs 31 and 32 and two flat plates 33 and 34 extending from the trough edges almost to the center line of the tower as shown in Figures 1a and 1c. The space 35 thus formed acts as a passageway for the flow of the continuous phase. The troughs are provided with angle sides 36 and 37 for ejecting the phase to be dispersed.

It is apparent that by alternating plates of the type 20 with those of 30 in the tower the continuous phase will be caused to flow downward and contact droplets of the discontinuous phase flowing upward from the inverted troughs. Such processing is indicated in Figure 2a and contrasted with the contacting occurring according to processing in a conventional pierced plate tower, Figure 2b.

It should be understood that a multiplicity of inverted troughs may be used on each plate as shown in Figures 3a and 3b rather than one or

two as described above. Plates 40 and 50 bear structural relationships to plates 20 and 30 of Figure 1. The inverted troughs 41 have angle perforated sides 45 which may be capped by horizontal plates 46. The inverted troughs have at their bases flat horizontal plates 42 and 43 which in this case do not extend almost across the diameter of the shell of the tower 10 but merely portions of the amount determined by the number of troughs on the plate. Between the various trough plate assemblies are the spaces 44 serving as passageways for the flow of the continuous phase. The plate 50 is shown as being a composite of plates of the type 20 and 30 as shown in Figure 1. The portion of the plate near the shell has the inverted trough portions 51 and 52 with perforated angle sides 56 and 57 similar to the corresponding portions of the inverted troughs 31 and 32 of plate 30. Furthermore, the shape of the troughs may be varied as, for example, in Figures 4a, 4b and 4c within the concept of the invention provided that the means thereby afforded for the settling of the discontinuous phase

nomical quantity of wash water to reduce the concentration of acetone in the butane stream to about 0.05% and to have about 13% concentration of acetone in the water wash stream. Such processing has been satisfactorily effected according to the invention by employing a tower of 5 feet in diameter and 80 feet in height containing 30 plates of the type shown in Figure 1 spaced $2\frac{1}{2}$ feet apart. The troughs are of two types: relatively long inverted troughs as designated in the diagram by the numeral 21 which have 242 $\frac{1}{4}$ -inch holes, and relatively short inverted troughs such as designated by the numeral 31 having 122 $\frac{1}{4}$ -inch holes per trough or 244 holes per plate of the type designated by the numeral 30. The average head in the inverted troughs is between 10 and 12 inches. Under such conditions a 50 to 75% spray of average size droplet of $\frac{1}{16}$ of an inch is obtained. The settling zones were designed to allow adequate settling of the droplets as small as $\frac{1}{16}$ of an inch in diameter.

Data obtained by processing in such a tower are as follows:

Table I

Butane stream.....	gallons per hour..	7,395	7,165	6,515	6,880	5,950	6,900	6,900
Water supply.....	gallons per hour..	4,005	4,005	4,005	4,005	4,005	4,005	4,005
Washed hydrocarbon stream.....	gallons per hour..	6,085	4,940	4,355	5,710	5,645	5,670	5,660
Concentration of acetone in washed hydrocarbon stream	weight per cent..	0.01	0.01	0.003	0.0	0.01	0.0	0.0

in the continuous phase is through a vertical stream of the continuous phase rather than a horizontally flowing stream.

Processing according to the invention involving the use of the above type apparatus requires that the heavier of the two liquids be introduced at the top of the tower while the lighter component is introduced at the bottom. As indicated by the arrows in Figures 1, 2a, 3 and 4, the lighter phase is processed in the tower by passing through the orifices in the inverted troughs and thence by settling through the continuous, substantially vertically moving streams of the continuous phase. The lighter phase then collects on the under side of the various plates such as plate 20 and is transferred to the trough such as 21 from which the liquid again emerges as droplets to be contacted again by the continuous phase. Such operation is repeated until the lighter phase reaches the top of the tower and the heavier phase reaches the bottom of the tower. In this manner, intimate contact within the two phases is attained and adequate settling assured.

The direction of flow of the two streams is immaterial according to the invention, although in the above description it has been assumed that the continuous phase passes downward and the discontinuous phase flows upward.

As a specific illustration of processing according to the invention, the treatment with water of the hydrocarbon stream from an extractive distillation system to remove solvent soluble in water may be employed. Thus, in the extractive distillation of the C_4 and C_5 hydrocarbons with aqueous acetone as the solvent, the acetone has to be recovered in the interests of economy from the various streams. For such an example, to a tower design as illustrated in Figure 1, may be taken the removing of acetone from the predominantly butane stream from an extractive distillation unit employing aqueous acetone as the solvent. Such a stream usually contains about 10% acetone. The data from a ternary diagram for the system—water, acetone and butane—and a stage-to-stage calculation indicate that seven theoretical stages are required when using an eco-

What is claimed is:

1. A process for countercurrently contacting relatively immiscible fluids of different gravities, comprising passing a continuous stream of one fluid through a system containing a series of contact zones, each of said zones containing a plurality of alternate regions of high and low velocity, the said change of velocity being obtained by alternately constricting and enlarging the stream as it flows through the system passing the other fluid as a discontinuous phase in the opposite direction, injecting the said other fluid in a finely divided state into the high velocity regions of the stream of the said first fluid, allowing the mixed fluids to separate by gravity in the low velocity regions, passing the thus separated fluids in opposite directions for further contact in the successive contact zones, and withdrawing the separated fluids respectively from opposite sides of said system.

2. A process as in claim 1, in which both fluids are liquids.

3. A process as in claim 1, in which the respective fluids are contacted by intersection substantially at right angles in the high velocity zones.

4. A process as in claim 1, wherein the said discontinuous phase is dispersed to form fine portions by being forced through small orifices by gravity differentials.

5. A process as in claim 1, wherein the separation of the two phases by settling occurs in regions where the path of the continuous stream has a relatively large cross-section.

6. An apparatus for the countercurrent treatment of relatively immiscible fluids of different gravity, comprising an upright shell fitted with a plurality of transverse plate assemblies which subdivide the interior of said shell into a series of settling chambers, each assembly comprising at least one narrow inverted trough, adapted to collect an ascending fluid of relatively light gravity, the trough having its open end edges flanged by horizontally disposed base portions extending outwardly from the trough edges, each said base portion having at least one constricted opening for permitting the downward flow of a fluid of

7

relatively heavy gravity, the trough being fitted in its upper solid portion with small perforations adapted to disperse the ascending fluid, alternate assemblies having said troughs and said constricted openings alternately disposed, said troughs of one assembly terminating in spaced relation to said constricted openings of the next higher assembly to cause the ascending fluid to intercept the descending fluid at a point of substantially most rapid flow of said descending fluid as it passes through said constricted opening, and an inlet and an outlet in both the uppermost and the lowermost settling chamber.

7. An apparatus for countercurrent extraction of fluids comprising an upright shell, a plurality of transverse baffle assemblies dividing the interior of the shell into a series of settling chambers, each assembly formed of a horizontally disposed base containing at least one constricted slot extending transversely of the base and adapted to permit downward flow of a relatively heavy liquid in a constricted stream, a portion of said base formed into at least one inverted U-shape portion which extends upwardly from the horizontal base portion and is aligned in spaced relation to a constricted slot of the next higher assembly, such U-shape portion being fitted near its crest with perforations adapted to disperse a relatively light liquid in a finely divided state into a rapidly flowing stream of the descending liquid as it passes through said constricted slot.

8. An apparatus for the countercurrent treatment of fluids of different gravity comprising a cylindrical shell, two types of alternately arranged baffle plates $2\frac{1}{2}$ feet apart which subdivide said shell transversely into a series of settling chambers, one type of said plates consisting of a narrow inverted trough at least 10 inches deep extending across a diameter of the shell, the upper solid portion of said trough fitted with fine perforations $\frac{1}{4}$ inch in diameter adapted to dis-

8

perse a relatively light fluid, the open end of each inverted trough flanged with two symmetrical horizontal plates extending in both directions from the trough edges and terminating in spaced relation to the shell, thus forming a constricted passageway for a descending relatively heavy fluid, the alternate type of plate consisting of a horizontally disposed base having a constricted slot extending transversely of the base and aligned with the inverted trough of the next lower plate, said base terminating on opposite sides in edges spaced from the wall and having an upwardly extending flange on each said edge, the upper portion of each flange terminating in a wall which is inclined and extends to meet the shell, the inclined wall being fitted with small perforations; the shell also comprising an inlet and an outlet in both the uppermost and the lowermost settling chamber.

JOHN D. LONG.
CYRIL O. RHYS, JR.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,170,658	Mitchell	Feb. 8, 1916
1,320,396	Laird	Nov. 4, 1919
1,621,728	Jordan	Mar. 22, 1927
1,782,735	Mackenzie	Nov. 25, 1930
2,009,510	Mobley	July 30, 1935
2,144,797	Dons et al.	Jan. 24, 1939
2,153,507	Mann	Apr. 4, 1939
2,274,030	Atkins	Feb. 24, 1942

FOREIGN PATENTS

Number	Country	Date
156,490	Great Britain	Jan. 5, 1921
199,345	Germany	Dec. 17, 1907